# **ISFRS** 2015

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structure

## **ABSTRACT BOOK OF THE**

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INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

JUNE 7 - 11 2015 ZURICH SWITZERLAND

### EDITORS: PETER FISCHER ERICH J. WINDHAB



Eidgenössische Technische Hochschule Zürich Swiss Federal Institute of Technology Zurich Laboratory of Food Process Engineering Institute of Food, Nutrition and Health

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#### AUTHOR INDEX

Akhtar, Mahmood	206, 207	Benjamins, Jan-Willem	210
Aktar, Tugba	212	Benyahia, Lazhar	5
Al-Habahbeh, Abdallah	121	Bernard, Jörg	33
Alba, Katerina	86	Berta, Marco	26
Almeida, Giana	224	Berton-Carabin, Claire	94,95
Alokaily, Samer	121	Bibiloni, Rodrigo	141
Altskär, Annika	27	Bindrich, Ute	57, 59
Altuntas, Fatma	192	Binois, Damien	167
Amani, Georges N'Guessan	162	Birbaum, Fabian	185
Ambebila, Emmanuel Neba	150	Bistac-Brogly, Sophie	44
Amigo Rubio, Jose Manuel	141	Bjerrum, Morten Jannik	23
Amin, Samiul	28	Blake, Steven	28
Andersson, Johan	210	Boiani, Mattia	61
Anema, Skelte	6	Bonazzi, Catherine	39
Archer, Richard	103	Boom, Remko M.	107
Ardö, Ylva	170	Bosc, Véronique	44, 74, 167
Arleth, Lise	23	Bourlieu, Claire	104
Arranz, E.	106	Bousquières, Josselin	39
Asensio-Barrowclough, Richard	219	Boyd, Ben	111
Aslan, Duygu	192, 193, 208, 212, 222	Brambilla, Giovanni	69,134
Assegehegn, Getachew	213, 214	Braun, André	45, 176, 223
Auty, Mark A.E.	127, 178	Braun, Peter	173
Ayunta, Anabel	155	Breau, Sophie	49
Azon, Carmen González	150, 204	Briard-Bion, Valérie	17
Badrinarayanan, Prashanth	31	Briesen, Heiko	45, 102, 176
Bae, Woosung	132	Brito-de La Fuente, Edmundo	213, 214
Bahtz, Jana	87	Brodkorb, Andre	180,226
Bak Jensen, Hanne	141	Bronze, Maria do Rosário	143
Baldino, Noemi	21, 48, 203, 242	Brun, Yefim	31
Banc, Amélie	20,71	Brütsch, Linda	160
Baniabedalruhman, Ahmad	119	Budelli, Andrea	62
Barbut, Shai	29, 139, 234	Buetikofer, Ueli	239
Baumann, Andreas	160	Buggy, Aoife	180
Bazin, Maxime	69,184	Buldo, Patrizia	141
Beccard, Steffen	33	Bulut-Solak, Birsen	228
Benfeldt, Connie	141	Burdikova, Zuzana	127

Butt, Saba         110         Dellemann, Jean-Philippe         236           Böcker, Lukas         218         Dersert, Nicolas         74           Bön, Lukas         218         Derser, Izvetelin T.         55           Bülow, Margareta         26         Dewaest, Marine         80           Campanella, Osvaldo         126         Dimitrakopoulou, Eleni Alkmini         147           Cardinaels, Ruth         43         Dirschert, Jennifer         176           Carnevale, Ilaria         48         Divoux, Thibaut         30           Carrington, Stephen         28         Dogsa, Iztok         72           Case, William         118         Duarte, Catarina         143           Chan, Christopher David         31         Duffy, John         28           Chasenieux, Christophe         5         Duri, Agnès         20, 71           Chasenieux, Christophe         5         Duri, Agnès         20, 71           Chaudemanche, Cyril         74         Duvarci, Oztem         225           Chen, Min         89         Ekberg, Olle         26           Christaki, Maria         161         Ermadzach, Bahareh         145, 152, 153           Christaki, Maria         611         Ermadzach, Bahareh	Bustos, Ana Yanina	163	Dekkers, Birgit	64
Böni, Lukas         218         Dessev, Tzvetelin T.         55           Bülow, Margareta         26         Dewaest, Marine         80           Campanella, Osvaldo         126         Dimitrakopoulou, Eleni Alkmini         147           Cardinaels, Ruth         43         Dirscherl, Jennifer         176           Carnevale, Ilaria         48         Divoux, Thibaut         30           Carriere, Frederic         105         Dogan, Mahmut         192, 193, 208, 212, 222, 227           Carrington, Stephen         28         Dogas, Jatok         72           Case, William         118         Duarte, Catarina         143           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnés         20, 71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgetj, Dana         46           Christodouldou, Christalli-Christina         166         Ermambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60,161	Butt, Saba	110		236
Bülow, Margareta26Dewaest, Marine80Campanella, Osvaldo126Dimitrakopoulou, Eleni Alkmini147Cardinaels, Ruth43Dirscherl, Jennifer176Carnevale, Ilaria48Divoux, Thibaut30Carriere, Frederic105Dogan, Mahmut192, 193, 208, 212, 222, 227Carriington, Stephen28Dogas, Iztok72Case, William118Duarte, Catarina143Char, Christopher David31Duffy, John28Charalambides, Maria110Dupont, Didier104Chassenieux, Christophe5Duri, Agnès20, 71Chaudemanche, Cyril74Duvarci, Oztem225Chen, Min89Ekberg, Olle26Chever, Sophie104Elgeti, Dana46Christophe104Elgeti, Dana46Christopha161Erndzadeh, Bahareh145, 152, 153Christonkou, Christalli-Christina166Ernambux, Mohammad Naushad38Chung, Cheryl68Ercili-Cura, Dilek209Claes, Johan60, 161Erickson, Daniel126Clifton, Peter9Ersch, Carsten147Cox, Philip37, 92Favaro, Jennifer40Curz Ortiz, Gonzalo177Feigk, Katheen118, 119, 121Courtiz, Gonzalo177Feigk, Katheen118, 119, 121Cuvelic, Gérard167, 224Feneton, Mark A171, 178, 179, 180, 226Dabestani, Mohsen20, 71Fischer, P	Böcker, Lukas	218	Demetri, Nicolas	74
Campanella, Osvaldo         126         Dimitrakopoulou, Eleni Alkmini         147           Cardinaels, Ruth         43         Dirschert, Jennifer         176           Carnevale, Ilaria         48         Divoux, Thibaut         30           Carriere, Frederic         105         Dogan, Malmut         192, 193, 208, 212, 222, 227           Carnington, Stephen         28         Dogas, Iztok         72           Case, William         118         Duarte, Catarina         143           Chan, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Cher, Min         89         Ekberg, Olle         26           Christaki, Maria         161         Emazadeh, Bahareh         145, 152, 153           Christopheryl         68         Ercili-Cura, Dilek         209           Christopheryl         68         Ercili-Cura, Dilek         209           Clase, Johan         60,161         Erickson, Daniel         112           Clifton, Peter         9         Ersch	Böni, Lukas	218	Dessev, Tzvetelin T.	55
Cardinaels, Ruth         43         Dirscherl, Jennifer         176           Carnevale, Ilaria         48         Divoux, Thibaut         30           Carriere, Frederic         105         Dogan, Mahmut         192, 193, 208, 212, 222, 227           Carrington, Stephen         28         Dogsa, Iztok         72           Case, William         118         Duarte, Catarina         143           Chara, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Ermazbux, Mohammad Naushad         38           Christodouldou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60,161         Erickson, Daniel         126           Clifton, Peter         9 <td< td=""><td>Bülow, Margareta</td><td>26</td><td>Dewaest, Marine</td><td>80</td></td<>	Bülow, Margareta	26	Dewaest, Marine	80
Carnevale, Ilaria         48         Divoux, Thibaut         30           Carriere, Frederic         105         Dogan, Mahmut         192, 193, 208, 212, 222, 227           Carrington, Stephen         28         Dogsa, Iztok         72           Case, William         118         Duarte, Catarina         143           Char, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Christaki, Maria         161         Ermadzadeh, Bahareh         145, 152, 153           Christaki, Maria         161         Ermadzadeh, Bahareh         145, 152, 153           Christodoulidou, Christalli-Christina         136         Ermambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Ditek         209           Claes, Johan         60,161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.	Campanella, Osvaldo	126	Dimitrakopoulou, Eleni Al	kmini 147
Carriere, Frederic         105         Dogan, Mahmut         192, 193, 208, 212, 222, 227           Carrington, Stephen         28         Dogsa, Iztok         72           Case, William         118         Duarte, Catarina         143           Chan, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chasenieux, Christophe         5         Duri, Agnès         20, 71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christong, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clinfor, Peter         9         Ersch, Carsten         14           Christina         16         Famelart, Ma	Cardinaels, Ruth	43	Dirscherl, Jennifer	176
Carrington, Stephen         28         Dogsa, Iztok         72           Case, William         118         Duarte, Catarina         143           Chan, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chasenieux, Christophe         5         Duri, Agnès         20, 71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christosen, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209         20           Claes, Johan         60, 161         Erickson, Daniel         126         26           Christon, Peter         9         Ersch, Carsten         14         21, 200, 201         20           Coker, Christina         106         Fang, Xixi         172	Carnevale, Ilaria	48	Divoux, Thibaut	30
Case, William         118         Durte, Catarina         143           Chan, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145,152,153           Christodoulidou, Christalli-Christina         136         Emmanbux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60,161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fags	Carriere, Frederic	105	Dogan, Mahmut	192, 193, 208, 212, 222, 227
Chan, Christopher David         31         Duffy, John         28           Charalambides, Maria         110         Dupont, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145,152,153           Christonoulidou, Christalli-Christina         136         Emmanbux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60,161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         171           Courtin, Christophe M.         43         F	Carrington, Stephen	28	Dogsa, Iztok	72
Charalambides, Maria         110         Dupnt, Didier         104           Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         144           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Courtin, Christophe M.         43         Fasseas, Costas         147           Courtin, Christophe M.         43         Fasseas, Costas         147           Cuvelier, Gérard         167, 224         Fenelon, Mark A.         171, 178, 179, 180, 226           Dabestani, M	Case, William	118	Duarte, Catarina	143
Chassenieux, Christophe         5         Duri, Agnès         20,71           Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christensen, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         172           Courtin, Christophe M.         43         Fasseas, Costas         147           Cox, Philip         37, 92         Favaro	Chan, Christopher David	31	Duffy, John	28
Chaudemanche, Cyril         74         Duvarci, Ozlem         225           Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christensen, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         172           Courtin, Christophe M.         43         Fasseas, Costas         147           Cox, Philip         37, 92         Favaro, Jennifer         40           Cruz Ortiz, Gonzalo         177         Feigl,	Charalambides, Maria	110	Dupont, Didier	104
Chen, Min         89         Ekberg, Olle         26           Chever, Sophie         104         Elgeti, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christensen, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         172           Courtin, Christophe M.         43         Fasseas, Costas         147           Cox, Philip         37, 92         Favaro, Jennifer         40           Cruz Ortiz, Gonzalo         177         Feigl, Kathleen         118, 119, 121           Cuvelier, Gérard         167, 224	Chassenieux, Christophe	5	Duri, Agnès	20,71
Chever, Sophie         104         Elger, Dana         46           Christaki, Maria         161         Emadzadeh, Bahareh         145, 152, 153           Christensen, Niels         41         Emin, M. Azad         58           Christodoulidou, Christalli-Christina         136         Emmambux, Mohammad Naushad         38           Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         141           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         172           Courtin, Christophe M.         43         Fasseas, Costas         147           Cox, Philip         37, 92         Favaro, Jennifer         40           Cruz Ortiz, Gonzalo         177         Feigl, Kathleen         118, 119, 121           Cuvelier, Gérard         167, 224         Fenelon, Mark A.         171, 178, 179, 180, 226           Dabestani, Moh	Chaudemanche, Cyril	74	Duvarci, Ozlem	225
Christaki, Maria       161       Emadzadeh, Bahareh       145, 152, 153         Christensen, Niels       41       Emin, M. Azad       58         Christodoulidou, Christalli-Christina       136       Emmambux, Mohammad Naushad       38         Chung, Cheryl       68       Ercili-Cura, Dilek       209         Claes, Johan       60, 161       Erickson, Daniel       126         Clifton, Peter       9       Ersch, Carsten       14         Clinger, Laura C.       31       Evageliou, Vasiliki       136, 199, 200, 201         Co, Edmund       54       Faber, Timo       117, 232         Coker, Christina       6       Famelart, Marie-Hélène       17, 104         Corredig, Milena       106       Fang, Xixi       172         Courtin, Christophe M.       43       Fasseas, Costas       147         Cox, Philip       37,92       Favaro, Jennifer       40         Cruz Ortiz, Gonzalo       177       Feigl, Kathleen       118, 119, 121         Cuvelier, Gérard       167, 224       Fenelon, Mark A.       171, 178, 179, 180, 226         Dabestani, Mohsen       20, 71       Fischer, Peter       21, 96, 105, 185, 218         Darwish, Tamim       111       Fiszman, Susana       168, 216 </td <td>Chen, Min</td> <td>89</td> <td>Ekberg, Olle</td> <td>26</td>	Chen, Min	89	Ekberg, Olle	26
Christensen, Niels       41       Emin, M. Azad       58         Christodoulidou, Christalli-Christina       136       Emmambux, Mohammad Naushad       38         Chung, Cheryl       68       Ercili-Cura, Dilek       209         Claes, Johan       60, 161       Erickson, Daniel       126         Clifton, Peter       9       Ersch, Carsten       14         Clinger, Laura C.       31       Evageliou, Vasiliki       136, 199, 200, 201         Co, Edmund       54       Faber, Timo       117, 232         Coker, Christina       6       Famelart, Marie-Hélène       17, 104         Corredig, Milena       106       Fang, Xixi       172         Courtin, Christophe M.       43       Fasseas, Costas       147         Cox, Philip       37, 92       Favaro, Jennifer       40         Cruz Ortiz, Gonzalo       177       Feigl, Kathleen       118, 119, 121         Cuvelier, Gérard       167, 224       Fenelon, Mark A.       171, 178, 179, 180, 226         Dabestani, Mohsen       20, 71       Fischer, Peter       21, 96, 105, 185, 218         Darwish, Tamim       111       Fiszman, Susana       168, 216         Davidovich-Pinhas, Maya       29, 139       FitzGerald, Richard J.       61	Chever, Sophie	104	Elgeti, Dana	46
Christodoulidou, Christalli-Christina136Emmambux, Mohammad Naushad38Chung, Cheryl68Ercili-Cura, Dilek209Claes, Johan60, 161Erickson, Daniel126Clifton, Peter9Ersch, Carsten14Clinger, Laura C.31Evageliou, Vasiliki136, 199, 200, 201Co, Edmund54Faber, Timo117, 232Coker, Christina6Famelart, Marie-Hélène17, 104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Christaki, Maria	161	Emadzadeh, Bahareh	145, 152, 153
Chung, Cheryl         68         Ercili-Cura, Dilek         209           Claes, Johan         60, 161         Erickson, Daniel         126           Clifton, Peter         9         Ersch, Carsten         14           Clinger, Laura C.         31         Evageliou, Vasiliki         136, 199, 200, 201           Co, Edmund         54         Faber, Timo         117, 232           Coker, Christina         6         Famelart, Marie-Hélène         17, 104           Corredig, Milena         106         Fang, Xixi         172           Courtin, Christophe M.         43         Fasseas, Costas         147           Cox, Philip         37, 92         Favaro, Jennifer         40           Cruz Ortiz, Gonzalo         177         Feigl, Kathleen         118, 119, 121           Cuvelier, Gérard         167, 224         Fenelon, Mark A.         171, 178, 179, 180, 226           Dabestani, Mohsen         20, 71         Fischer, Peter         21, 96, 105, 185, 218           Darwish, Tamim         111         Fiszman, Susana         168, 216           Davidovich-Pinhas, Maya         29, 139         FitzGerald, Richard J.         61           Day, Li         9, 40         Fleury, Mathias         69, 134, 184           De Krui	Christensen, Niels	41	Emin, M. Azad	58
Claes, Johan60, 161Erickson, Daniel126Clifton, Peter9Ersch, Carsten14Clinger, Laura C.31Evageliou, Vasiliki136, 199, 200, 201Co, Edmund54Faber, Timo117, 232Coker, Christina6Famelart, Marie-Hélène17, 104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Christodoulidou, Christalli-Christina	136	Emmambux, Mohammad	Naushad 38
Clifton, Peter9Ersch, Carsten14Clinger, Laura C.31Evageliou, Vasiliki136, 199, 200, 201Co, Edmund54Faber, Timo117, 232Coker, Christina6Famelart, Marie-Hélène17, 104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Chung, Cheryl	68	Ercili-Cura, Dilek	209
Clinger, Laura C.31Evageliou, Vasiliki136, 199, 200, 201Co, Edmund54Faber, Timo117, 232Coker, Christina6Famelart, Marie-Hélène17, 104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Claes, Johan	60,161	Erickson, Daniel	126
Co, Edmund54Faber, Timo117, 232Coker, Christina6Famelart, Marie-Hélène17, 104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Clifton, Peter	9	Ersch, Carsten	14
Coker, Christina6Famelart, Marie-Hélène17,104Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37,92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118,119,121Cuvelier, Gérard167,224Fenelon, Mark A.171,178,179,180,226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20,71Fischer, Peter21,96,105,185,218Darwish, Tamim111Fiszman, Susana168,216Davidovich-Pinhas, Maya29,139FitzGerald, Richard J.61Day, Li9,40Fleury, Mathias69,134,184De Kruif, Kees6Folkenberg, Ditte Marie63,141De Vito, Francesca81,188Font De Valdez, Graciela163	Clinger, Laura C.	31	Evageliou, Vasiliki	136, 199, 200, 201
Corredig, Milena106Fang, Xixi172Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37, 92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Co, Edmund	54	Faber, Timo	117, 232
Courtin, Christophe M.43Fasseas, Costas147Cox, Philip37,92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Coker, Christina	6	Famelart, Marie-Hélène	17, 104
Cox, Philip37,92Favaro, Jennifer40Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Corredig, Milena	106	Fang, Xixi	172
Cruz Ortiz, Gonzalo177Feigl, Kathleen118, 119, 121Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Courtin, Christophe M.	43	Fasseas, Costas	147
Cuvelier, Gérard167, 224Fenelon, Mark A.171, 178, 179, 180, 226Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Cox, Philip	37, 92	Favaro, Jennifer	40
Dabestani, Mohsen233Ferrero, Cristina177Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Cruz Ortiz, Gonzalo	177	Feigl, Kathleen	118, 119, 121
Dahesh, Mohsen20, 71Fischer, Peter21, 96, 105, 185, 218Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Cuvelier, Gérard	167, 224	Fenelon, Mark A.	171, 178, 179, 180, 226
Darwish, Tamim111Fiszman, Susana168, 216Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Dabestani, Mohsen	233	Ferrero, Cristina	177
Davidovich-Pinhas, Maya29, 139FitzGerald, Richard J.61Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Dahesh, Mohsen	20,71	Fischer, Peter	21, 96, 105, 185, 218
Day, Li9, 40Fleury, Mathias69, 134, 184De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Darwish, Tamim	111	Fiszman, Susana	168,216
De Kruif, Kees6Folkenberg, Ditte Marie63, 141De Vito, Francesca81, 188Font De Valdez, Graciela163	Davidovich-Pinhas, Maya	29,139	FitzGerald, Richard J.	61
De Vito, Francesca81, 188Font De Valdez, Graciela163	Day, Li	9,40	Fleury, Mathias	69, 134, 184
	De Kruif, Kees	6	Folkenberg, Ditte Marie	63, 141
De Wouters, Tomas 218 Forssell, Pirkko 209	De Vito, Francesca	81,188	Font De Valdez, Graciela	163
	De Wouters, Tomas	218	Forssell, Pirkko	209

Franke, Knut	57, 59	Gurmeric, Vildan	192, 193, 222, 227
Frantzeskaki, Despoina	136	Gutierrez, Jose Maria	150, 204
Gabriele, Domenico	21, 48, 203, 242	Haavisto, Sanna	185
Gagnaire, Valérie	17	Hagman, Joel	15
Gallegos Montes, Críspulo	213, 214	Hallmark, B.	175
Gallo, Marianna	62	Hamaker, Bruce	126
Gaspard, Sophie	226	Hamedi, Naser	120
Gates, Fred	37	Hamilton, Ian	24
Gebhardt, Ronald	56	Hanna, Charles	113
Gehrich, Karin	33	Hansen, Inger	170
Gehringer, Julia	186	Hansen, Janne Riis	144
Geng, Xiaolu	23	Havea, Palatasa	100
Germann, Natalie	116, 146, 187	Hawley, Adrian	111
Geue, Thomas	105	Hecker, Florian	159
Ghorbani Gorji, Elham	197	Heinrich, Stefan	129
Giampaoli, Pierre	44	Hermansson, Anne-Marie	15, 27, 149
Gibson, Douglas	175	Hernando, Isabel	168
Gilbert, Elliot	111	Heyd, Bertrand	44
Gillies, Graeme	100	Hickey, Cian	127
Gladkowska Balewicz, Izabela	24	Hill, Adrian	68
Goh, Kelvin	103	Hindmarsh, Jason	103
Goksel, Meryem	212	Hinrichs, Jörg	19,52
Golding, Matt	9, 100, 103	Hitzmann, Bernd	159,186
Golematis, Anne	31	Hogan, Sean	128, 180, 220
Goodbread, Joe	82	Hooshyar, Soroush	116
Goodison, Alan	179	Hosseini, Noushin	133,174
Goos, Peter	161	Hougaard, Anni	170
Gordon, Tyler	113	Howes, Tony	65
Goudoulas, Thomas	146,187	Huault, Lucie	44
Grabowska, Kasia	75	Hubbes, Stephen-Sven	223
Gracia Fernandez, Carlos A.	97, 190, 191	Huc, Delphine	74,167
Gravelle, Andrew	29, 234	Innings, Fredrik	120
Greco, Valeria	21	lpsen, Richard	23, 63, 138, 141, 170
Green, Ali	92	Iskakova, Janyl	183
Greiner, Maximilian	102	Iturriaga, Laura	154, 155, 163, 177
Greve, Marie	138	Iwasaki, Sayuri	13
Grizzuti, Ninoi	62	Jaishankar, Aditya	232
Guggisberg, Dominik	239	Jang, Areum	166
Gunes, Zeynel Deniz	87	Jans, Christoph	218

Janssen, Anja E.M.	107	Labrie, Steve	172
Jardin, Julien	17	Lacou, Lélia	17
Jasutiene, Ina	205	Lad, Mita	100
Javanmard, Marjan	65	Laguna, Laura	78,219
Jesinghausen, Steffen	83	Lammers, Volker	202
Jose, Jissy	135,137	Larsson, Anette	149
Joyner, Helen	243	Lazou, Andriana	240
Jurgens, Albert	126	Le, Xuan Thang	22
Kadkhodaee, Rassoul	145, 152, 153, 189	Lee, Suyong	132,166
Kalitekin, Sidika	193	Lenaerts, Sanne	60
Kalnin, Daniel	49	Leocmach, Mathieu	30
Kampuse, Solvita	217	Leskauskaite, Daiva	205
Kaneda, Isamu	13	Leverrier, Cassandre	224
Kara, Theodora	241	Lewis, E. Neil	28
Karathanos, Vaios T.	240	Lexis, Meike	93
Karazhiyan, Hojjat	230	Li, Chaoxu	25
Kelly, Alan	179,226	Li, Y.	106
Kelly, Phil	61, 128, 171, 220	Lille, Martina	209
Kennedy, Deirdre	171, 178, 179	Lim, Wensheng	103
Kenyon, Stacy	28	Litvinova, Jekaterina	217
Kersiene, Milda	205	Liu, Guanchen	138
Kidder, Linda	28	Liu, Kun	125
Kim, Yong-Ro	142	Londona, Juan David	31
Kindlein, Moritz	102	Lorén, Niklas	15,27
Kirby, Nigel	111	Luo, Qi	107
Kirkensgaard, Jacob Judas Kain	23	Lupi, Francesca Romana	21, 48, 203, 242
Kirse, Christoph	45	Luzzi, Stefania	203
Klava, Dace	217	Läuger, Jörg	77
Ko, Sanghoon	132,166	Lê, Sébastien	17
Kokini, Jozef	225	Macakova, Lubica	210
Konan, Amoin Georgette	244	Macdonald, Adam	113
Konrade, Daiga	217	Macias-Rodriguez, Braulio	47
Kontogiorgos, Vassilis	36,86	Madsen, Finn	79
Koos, Erin	101	Maia, Catarina	143
Koponen, Antti	185	Maksimenko, Anton	40
Koutinas, Apostolis	91, 147, 199, 200, 201	Malinauskyte, Ernesta	205
Kragh, Henrik	79	Mandala, Ioanna 42, 91, 147, 199,	200, 201, 240, 241
Krämer, Anna	159	Manneville, Sébastien	30
Kulmyrzaev, Asylbek	183,238	Mao, Like	61

Marangoni, Alejandro	29, 47, 54, 112, 113, 139, 234	Myllärinen, Päivi	209
Marcano, Johanna	216	Ménard, Olivia	104
Martin, Anneke	14, 135, 137	Møller, Stine	79
Martínez, Diego Mínguez	150	Nagazi, Med Yassine	184
Mayo, Sherry	40	Nettesheim, Florian	31
Mazaheri, Amir Farokh	174	Netto, Flavia Maria	206, 207
Mc Manus, Jennifer J.	180	Nguyen, Bach	5
McCann, Thu	40	Nicholson, Reed	29
McCarthy, Noel	171	Nicolai, Taco	5,12
McClements, David Julian	68	Nicolaï, Bart	161
McGrath, Kathryn	96	Nielsen, Heidi Liva	144
McKinley, Gareth	232	Nigro, Roberto	62
Meerts, Mathieu	43	Nikolidaki, Eirini K.	240, 245
Meijer, Han	117	Nindjin, Charlemagne	162,244
Meinders, Marcel	88,89	Norton, lan	16, 24, 53
Meldrum, Alexander	243	Nyström, Magda	26
Menéndez-Aguirre, Orquíd	ea 19	O'Sullivan, Jonathan	16
Meshkani, Seyed Mohamm	ad 158	O'Loughlin, Ian	128
Meunier, Gérard	69,134	O'Mahony, James A.	179, 226, 228
Meyer, Fabian	81,188	Oladzad, Nazila	182
Mezzenga, Raffaele	25, 32	Ong, Teck Ann	103
Michon, Camille	39,74	Oosterlinck, Filip	43
Middendorf, Dana	57	Otte, Jeanette	23
Mileti, Olga	242	Ozbekova, Zhyldyzai	238
Mischnick, Petra	57	Palzer, Stefan	129
Mishima, Beatriz	154	Panagopoulou, Elli	199, 200, 201
Moakes, Richard	53	Papadakis, Spyros E.	240, 245
Moldenaers, Paula	43	Papadopoulou, Evangelia	
Morant, Angelika	202	Papastamopoulou, Konstantina	136
Morel, Marie-Hélène	20,71	Park, Shinjae	
Morell, Pere	168	Partanen, Riitta	209
Morren, Sofie	60	Pasquino, Rossana	62
Mortazavi, Seyed Ali	158	Paximada, Paraskevi	91, 147, 201
Mostafavi, Fatemeh Sada	t 189	Pedersen, Sara	144
Mousavi, Seyed Mohamma	d 151	Peng, Jinfeng	140
Muijlwijk, Kelly	94	Perge, Christophe	30
Mun, Sehun	142	Peyronel, Fernanda	47, 112
Munro, Peter A.	55	Pezennec, Stéphane	17
Murray, Brent S.	206, 207	Phan, Stephanie	111

Pinheiro, Lídia	143	Sadeghy, Keyvan	235
Pink, David	112, 113	Sagis, Leonard 89, 9	
Pinto, Marialuisa	62	Saguez, Philippe	
Poberaj, Igor	72	Sala, Guido	
Pourjafar, Mohammad	235	Saldanha do Carmo, Cátia	143
Pouvreau, Laurice	135,137	Salentinig, Stefan	111
Powell, Hugh	110	Salvador, Ana	196, 215
Protonotariou, Styliani V.	42, 147, 240	Sanz, Teresa	196, 215
Protte, Kristin	19	Sargent, Michael	70,231
Puppo, Cecilia	155	Sarkar, Anwesha	78,219
Qazi, Waqas	26	Schefer, Larissa	32
Quinn, Bonnie	113	Scheuble, Nathalie	96, 105
Quinzio, Claudia	154	Schmid, Hans-Joachim	83
Radenkovs, Vitalijs	217	Schmidt, Christian	52
Rafe, Ali	148,233	Scholten, Elke	91
Rakcejeva, Tatjana	217	Schopferer, Michael	190, 191
Ramos, Laurence	20,71	Schroën, Karin	94,95
Ramsch, Roland	69,134	Schröder, Anja	95
Rasmussen, Christopher J.	31	Schuchmann, Heike Petra	58
Razavi, Seyed Mohammad Ali	148	Schuster, Erich	15, 27, 149
Reinke, Svenja	129	Schäfer, Johannes	52
Rejman, Lucie	173	Secouard, Sebastien	213, 214
Renzetti, Stefano	126	Sharma, Prateek	55
Reufer, Mathias	73	Sheehan, Diarmuid JJ	127
Revstedt, Johan	120	Sieffermann, Jean-Marc	74
Rioux, Laurie-Eve	172	Siegert, Nadja	213, 214
Ritzoulis, Christos	241	Sieuwerts, Sander	141
Robbins, Phillip	37	Sindic, Marianne	162
Rodrigues, Maria Isabel	206, 207	Skoumpi, Georgia S.	245
Rolfe, Philip	68	Slettengren, Katarina	169
Rosell, Cristina M.	42	Smanalieva, Jamila	183
Roussel, Nicolas	8	Sobolewski, Franck	49
Rovalino, Ana	95	Sonne, Alina	19
Rovers, Tijs	88	Sott, Kristin	27, 149
Rudemo, Mats	15	Speirs, Charles	37
Russ, Natalie	18	Sretenovic, Simon	72
Rühs, Patrick	218	Stading, Mats	26,76
Sadahira, Mitie Sonia	206, 207	Steingötter, Andreas	9
Sadeghi, Farzad	145, 152, 153	Stieger, Markus	125

	55
Wilkinson, Martin G	127
Willenbacher, Norbert	93,101
Williams, Gordon	110
Wilson, D.I.	175
Windhab, Erich J.	10, 87, 105, 118, 119, 121, 160,
	169, 173, 185, 202
Winkler, Hans	239
Wium, Helle	41
Wolf, Bettina	4
Wollgarten, Susanne	101
Wong, Edeline	65
Wooster, Tim	9
Wouters, Rudy	33
Yuri, A.	106
Zacharias, Jörg	176
Zare, Davoud	96
Zettel, Viktoria	159, 186
Zhang, Lanjun	63
Zhao, Jianguo	25
Zielbauer, Birgitta	18
Zogzas, Nikolaos P.	240, 245
de Cindio, Bruno	21, 48, 203, 242
de Jong, Saskia	124
de Jongh, Harmen	124, 135
van Breemen, Lambert	117
van Hooijdonk, A.C.M.	89
van Leusden, Pauline	90
van Valenberg, H.J.F.	89
van de Velde, Fred	125
van der Goot, Atze Jan	64,75
van der Linden, Erik	14,88,89,90,125,140
	<ul> <li>Willenbacher, Norbert</li> <li>Williams, Gordon</li> <li>Wilson, D.I.</li> <li>Windhab, Erich J.</li> <li>Winkler, Hans</li> <li>Wium, Helle</li> <li>Wolf, Bettina</li> <li>Wollgarten, Susanne</li> <li>Wong, Edeline</li> <li>Wooster, Tim</li> <li>Wouters, Rudy</li> <li>Yuri, A.</li> <li>Zacharias, Jörg</li> <li>Zare, Davoud</li> <li>Zettel, Viktoria</li> <li>Zhang, Lanjun</li> <li>Zhao, Jianguo</li> <li>Zielbauer, Birgitta</li> <li>Zogzas, Nikolaos P.</li> <li>de Cindio, Bruno</li> <li>de Jong, Saskia</li> <li>de Jong, Saskia</li> <li>de Jongh, Harmen</li> <li>van Breemen, Lambert</li> <li>van Hooijdonk, A.C.M.</li> <li>van Leusden, Pauline</li> <li>van de Velde, Fred</li> <li>van der Goot, Atze Jan</li> </ul>

### Abstract Book of the 7<sup>th</sup> International Symposium on Food Rheology and Structure

#### Contents

Opening Lecture TALKS
<b>Structural and mechanical anisotropy in sheared colloidal gels</b> 2 Jan Vermant*
Keynotes TALKS
<b>Food rheology and structure for nutritionally enhanced foods</b> 4 Bettina Wolf*
Effect of competition for $Ca^{2+}$ on the morphology and the elasticity of mixed gels of $\kappa$ - carrageenan and $\beta$ -lactoglobulin 5 Taco Nicolai*, Bach Nguyen, Christophe Chassenieux, Lazhar Benyahia
<b>Casein hydrogels: Water holding capacity related to swelling and rheology of caseinates</b> 6 Kees De Kruif*, Skelte Anema, Christina Coker
Insights into food design and oral processing using soft matter physics, thin film rheology, and tribology Jason Stokes*
Mixing of construction materials on different length scales 8 Nicolas Roussel*
Gastric Structuring: How food structure and rheology impact on fat digestive processing9 Tim Wooster*, Andreas Steingötter, Li Day, Matt Golding, Peter Clifton
<b>Interplay of rheology and functional structure processing along the food value chain</b> 10 Erich J. Windhab*
Biopolymer Solutions and Gels TALKS
<b>Rheological behavior of dense milk protein suspensions in the presence of minerals</b> 12 Peggy Thomar <sup>*</sup> , Taco Nicolai
<b>Solvent transportation behavior of mechanically constrained agarose gels</b> 13 Isamu Kaneda*, Sayuri Iwasaki
Molecular interactions, morphology and macroscopic properties of mixed protein-protein systems Carsten Ersch*, Paul Venema, Erik van der Linden, Anneke Martin
Local diffusion in heterogeneous food microstructures determined by quantitative confocal microscopy 15 Niklas Lorén*, Joel Hagman, Erich Schuster, Mats Rudemo, Anne-Marie Hermansson
Ultrasonic effect on the rheology of protein solutions16Jonathan O'Sullivan*, Ian Norton

<b>Rheological properties of milk derived peptides in high-protein matrices</b> 17 Lélia Lacou <sup>*</sup> , Marie-Hélène Famelart, Julien Jardin, Valérie Briard-Bion, Sébastien Lê, Stéphane Pezennec, Valérie Gagnaire
Pre-gelatinized tapioca starch and its mixtures with xanthan and ι-carrageenan18Natalie Russ*, Birgitta Zielbauer, Thomas A. Vilgis18
Process stable whey protein-pectin complexes as new structuring elements in fat reduced food systems
Kristin Protte*, Orquídea Menéndez-Aguirre, Alina Sonne, Jochen Weiss, Jörg Hinrichs
Polymeric assembly of gluten proteins in an aqueous ethanol solvent20Amélie Banc*, Mohsen Dahesh, Agnès Duri, Marie-Hélène Morel, Laurence Ramos20
A rheological and microstructural characterisation of monoglycerides/olive oil organogels 21
Valeria Greco, Francesca Romana Lupi*, Domenico Gabriele, Noemi Baldino, Bruno de Cindio, Peter Fischer
Electrostatic gels between $\beta$ -lactoglobulin and xanthan gum: Properties and potential applications 22 Xuan Thang Le, Sylvie L. Turgeon*
The influence of pH on the proteolysis and self-assembling of partially hydrolyzed bovine $\alpha$ -lactalbumin into nanotubes and gels 23 Xiaolu Geng*, Jacob Judas Kain Kirkensgaard, Lise Arleth, Morten Jannik Bjerrum, Jeanette Otte, Richard Ipsen
Changes in viscoelastic properties as an effect of maltodextrin paselli SA2 addition on polysaccharides helix aggregation in sheared gel mixtures24Izabela Gladkowska Balewicz*, Ian Hamilton, Ian Norton24
<b>Re-entrant isotropic-nematic phase behavior in polymer-depleted amyloid fibrils</b> 25 Jianguo Zhao*, Chaoxu Li, Raffaele Mezzenga
<b>Effect of fluid elasticity on swallowing</b> 26 Mats Stading*, Waqas Qazi, Magda Nyström, Marco Berta, Margareta Bülow, Olle Ekberg
Flow, diffusion and microstructure in capillary alginate gels27Erich Schuster, Kristin Sott*, Anna Ström, Annika Altskär, Niklas Lorén, Anne-Marie Hermansson
Mechanistic insights into self-assembly and gelation in biopolymers, proteins, and complexfluids through combined DLS-optical microrheology and raman spectroscopy28Samiul Amin, Steven Blake, E. Neil Lewis, Linda Kidder, Stacy Kenyon, John Duffy*, StephenCarrington
New strategies for tailoring the mechanical properties of ethylcellulose oleogels for food applications 29
Andrew Gravelle*, Maya Davidovich-Pinhas, Reed Nicholson, Shai Barbut, Alejandro Marangoni
Acid-induced protein gels: From gelation to stress-induced failure30Mathieu Leocmach*, Christophe Perge, Thibaut Divoux, Sébastien Manneville30

Carrageenan aggregation and gel transition31Florian Nettesheim*, Juan David Londona, Prashanth Badrinarayanan, Christopher J. Rasmussen,Dennis Walls, Yefim Brun, Laura C. Clinger, Christopher David Chan, Anne Golematis
Anomalous stiffening and ion-induced coil-helix transition of carrageenan in monovalent salt conditions 32 Larissa Schefer*, Ivan Usov, Raffaele Mezzenga
Alteration of the textural properties of inulin gels33Steffen Beccard*, Thomas A. Vilgis, Jörg Bernard, Rudy Wouters, Karin Gehrich33
Dough TALKS
<b>Relaxation dynamics, glass transitions, and microstructure of hydrated gluten networks</b> 36 Vassilis Kontogiorgos*
Assessing the handleability of bread dough 37 Christine Tock*, Fred Gates, Charles Speirs, Gary Tucker, Phillip Robbins, Philip Cox
<b>Rheology of maize, wheat, teff, and cassava starches with stearic acid and xanthan gum</b> 38 Mohammad Naushad Emmambux*
Starchy foam tailored with cellulose derivatives and transformed in non-reactive sponge cake after baking 39 Josselin Bousquières*, Catherine Bonazzi, Camille Michon
The porous structure of dough characterised using in situ Synchrotron X-ray computertomography40Thu McCann*, Sherry Mayo, Jennifer Favaro, Helen Tuhumury, Anton Maksimenko, Li Day
Instrumentally determined sensory moistness in yeast-raised toast bread41Helle Wium*, Niels Christensen
Effect of jet milling on whole wheat flour quality and bread digestibility42Styliani V. Protonotariou*, Ioanna Mandala, Cristina M. Rosell
Unraveling the contributions of starch, gluten, water content and mixing time to the rheology of wheat dough 43 Mathieu Meerts*, Ruth Cardinaels, Filip Oosterlinck, Christophe M. Courtin, Paula Moldenaers
Evolution of adhesion between bread dough and a solid surface during baking: effect on rheological properties and physico-chemical properties of dough44Lucie Huault, Bertrand Heyd, Sophie Bistac-Brogly, Pierre Giampaoli, Véronique Bosc*44
A mechanistic model describing the growth dynamics of bubbles in viscoelastic dough during proving André Braun*, Christoph Kirse, Heiko Briesen

Identification of gluten-free dough properties relevant for aeration and foam stabilization Dana Elgeti\* 46

Structural and rheological characterization of laminating shortenings47Braulio Macias-Rodriguez*, Fernanda Peyronel, Alejandro Marangoni47
Rheological optimization of durum wheat dough mixing by uniaxial extensional properties $48$
Noemi Baldino*, Ilaria Carnevale, Domenico Gabriele, Francesca Romana Lupi, Bruno de Cindio
Structural impact of butter in puff pastry49Daniel Kalnin*, Sophie Breau, Philippe Saguez, Franck Sobolewski49
Influence of Processing on Structure and Rheology TALKS
Measurement of sol-gel-transition of skim milk and microfiltrated skim milk retentates bymeans of oscillation rheology and dynamic light scattering52Johannes Schäfer*, Christian Schmidt, Jörg Hinrichs
Rheological properties of novel whey protein emulsion fluid gels53Richard Moakes*, Antonio Sullo, Ian Norton53
Shake it but don't break it: Structure formation under oscillatory shear54Alejandro Marangoni*, Edmund Co
Effect of accumulated shear work on the rheology, structure, and melt functionality of a mozzarella-type cheese 55 Prateek Sharma*, Peter A. Munro, Tzvetelin T. Dessev, Peter G. Wiles
<b>Process-induced structural changes of casein micelles and casein micro-particles</b> 56 Ronald Gebhardt*
AFM studies on the impact of different grinding techniques on sucrose surfaces and resulting flow properties of chocolate model suspensions 57 Dana Middendorf*, Knut Franke, Ute Bindrich, Petra Mischnick
A mechanistic approach to deliver functionality into complex food systems via extrusion processing 58 M. Azad Emin*, Heike Petra Schuchmann
Dependency of moisture barrier properties of fat blends on their fat crystal network struc- ture and possibilities for influencing thereof 59 Knut Franke*, Ute Bindrich
<b>Rheological characterization of commercial thickening agents for dysphagia patients</b> 60 Sofie Morren*, Sanne Lenaerts, Johan Claes
Application of NMR techniques for improved understanding of molecular and structural changes during ingredient processes61Mattia Boiani*, Like Mao, Richard J. FitzGerald, Phil Kelly
<b>Rheological properties of baby foods: Dependence on recipe and type of process</b> 62 Marialuisa Pinto, Marianna Gallo, Ninoi Grizzuti, Andrea Budelli, Rossana Pasquino*, Roberto Nigro

Microstructure and texture of stirred yoghurt made with different exopolysaccharide pro- ducing cultures and subjected to mechanical treatment after fermentation63Lanjun Zhang, Richard Ipsen*, Ditte Marie Folkenberg
<b>Understanding structure formation in dense biopolymer mixtures</b> 64 Birgit Dekkers*, Atze Jan van der Goot
Application of the thixotropic elasto-viscoplastic model as a structure probing techniquein weak gel suspensions65Marjan Javanmard*, Jason Stokes, Tony Howes, Edeline Wong
Rheological Methods TALKS
Simulating food mastication by applying integrated 'compression-shear-decompression' cycles using a Kinexus rotational rheometer 68 Cheryl Chung, Philip Rolfe, Adrian Hill*, David Julian McClements
Analysis of fat crystallization thanks to microrheology69Maxime Bazin*, Roland Ramsch, Giovanni Brambilla, Mathias Fleury, Gérard Meunier
The single phase shear rheology of molten instant coffee70Michael Sargent*70
Spontaneous gelation of wheat gluten proteins in a food grade solvent71Laurence Ramos, Mohsen Dahesh, Amélie Banc*, Agnès Duri, Marie-Hélène Morel71
Active laser tweezers microrheology of inhomogeneus mixtures72Biljana Stojkovic, Simon Sretenovic, Iztok Dogsa, David Stopar, Igor Poberaj*72
<b>Micro-rheology as a tool for the characterization of food</b> 73 Mathias Reufer*
Investigation of texture-structure-perception relationships of protein-enriched yogurts with different fat contents 74 Delphine Huc*, Véronique Bosc, Cyril Chaudemanche, Nicolas Demetri, Jean-Marc Sieffermann, Camille Michon
Combining NMR relaxometry and rheology to predict structural properties of SPI-WG blends 75 Atze Jan van der Goot*, Kasia Grabowska
Flow-Viz, a fully integrated and commercial in-line fluid characterization system for indu- strial applications 76 Johan Wiklund, Mats Stading*
Advanced rheometric tools for food applications 77 Jörg Läuger*
Impact of structuring of mixed gels on oral breakdown and sensory perception78Laura Laguna*, Anwesha Sarkar

Predicting sensory parameters such as fat perception by tribology measurements79Henrik Kragh*, Finn Madsen, Stine Møller
Identification of formulation effects on cake softness using a technique combining alveolarstructure analysis with mechanical properties characterization80Marine Dewaest*80
Yield stress determination – impact of sample handling and loading, measuring method and type of evaluation 81 Fritz Sörgel*, Fabian Meyer, Francesca De Vito
A novel process viscometer for food rheology applications 82 Joe Goodbread*
Particle Image Velocimetry and its potential to improve high shear rate processing of food suspensions 83 Steffen Jesinghausen*, Hans-Joachim Schmid
Emulsions and Interfaces TALKS
<b>Coarsening mechanisms of alkane-in-water okra pectin stabilized emulsions</b> 86 Katerina Alba*, Vassilis Kontogiorgos
Multiple W/O/W emulsion systems with adaptive transient thickening behavior 87 Jana Bahtz*, Zeynel Deniz Gunes, Erich J. Windhab
Temperature is key to yield and stability of protein stabilized microbubbles88Tijs Rovers*, Guido Sala, Erik van der Linden, Marcel Meinders
<b>Effect of processing conditions on the foaming behavior of casein micelle dispersions</b> 89 Min Chen*, Guido Sala, Marcel Meinders, H.J.F. van Valenberg, A.C.M. van Hooijdonk, Erik van der Linden, Leonard Sagis
Microencapsulation systems for heat sensitive, oil-soluble components90Pauline van Leusden*, Erik van der Linden, Leonard Sagis
Bacterial cellulose as an alternative thickener for o/w emulsions: Comparison with common thickeners 91 Paraskevi Paximada*, Apostolis Koutinas, Elke Scholten, Ioanna Mandala
Food grade ingredients and their effects on the production and stability of air filled emul- sions 92 Ali Green*, Philip Cox
<b>Relating foam and interfacial rheological properties of</b> $\beta$ <b>-lactoglobulin solutions</b> 93 Norbert Willenbacher <sup>*</sup> , Meike Lexis
Dynamic interfacial tension measurements during the first milliseconds of emulsion forma- tion 94 Kelly Muijlwijk*, Claire Berton-Carabin, Karin Schroën

Interfacial behavior and rheology of oxidized proteins and lipids95Claire Berton-Carabin, Ana Rovalino, Anja Schröder, Karin Schroën, Leonard Sagis*
Interfacial viscoelasticity of protein-polysaccharide composite layers at an oil/water inter- face 96
Davoud Zare*, Nathalie Scheuble, Peter Fischer, Kathryn McGrath
Advanced rheological measurements in food emulsions: Interfacial double wall ring and microscopy 97
Carlos A. Gracia Fernandez*
Cheese, Chocolate, and Health TALKS
Laboratory scale cheese with model filler particles: How structure controls rheological properties 100
Orianne Thionnet*, Graeme Gillies, Palatasa Havea, Mita Lad, Matt Golding
Tailoring flow behavior and texture of water continuous cocoa suspensions101Norbert Willenbacher*, Susanne Wollgarten, Erin Koos
A molecular dynamics approach to explore the interaction of lecithin molecules and sucrose particles in chocolate 102 Moritz Kindlein*, Maximilian Greiner, Heiko Briesen
Effect of varying insoluble volume fraction on rheological and recovery properties of mixed colloidal suspensions 103 Wensheng Lim*, Teck Ann Ong, Matt Golding, Kelvin Goh, Jason Hindmarsh, Richard Archer
<b>Two rheological methods to approach the gastric behavior of infant formulas</b> 104 Sophie Chever, Marie-Hélène Famelart*, Olivia Ménard, Claire Bourlieu, Didier Dupont
Controlled mechanical and structural properties of biopolymer adsorption layers under gastric conditions
Nathalie Scheuble*, Thomas Geue, Frederic Carriere, Erich J. Windhab, Peter Fischer
Interactions of delivery systems with gut epithelial mucus studied by drop tensiometrycoupled with relevant in vitro absorption models106Milena Corredig*, A. Yuri, Y. Li, E. Arranz106
<b>Mechanistic approaches towards understanding the kinetics of gastric digestion</b> 107 Qi Luo*, Remko M. Boom, Anja E.M. Janssen
Rheo-SALS, SANS, SAXS TALKS
Geometric and material effects on sensory properties of confectionery wafers and similar extruded products 110 Saba Butt*, Maria Charalambides, Gordon Williams, Hugh Powell
Small-angle neutron and X-ray scattering of pH-responsive micelles based on short, medium and long chain saturated fatty acids111Elliot Gilbert*, Stefan Salentinig, Stephanie Phan, Tamim Darwish, Nigel Kirby, Adrian Hawley, Ben Boyd111

The nano-to meso-scale solid structure of edible fats as seen by USAXS: Static versus dynamics results 112 Fernanda Peyronel*, Alejandro Marangoni, David Pink
Modelling shear and solid fat aggregation in edible oils 113 David Pink*, Bethany Townsend, Bonnie Quinn, Adam Macdonald, Tyler Gordon, Charles Hanna, Alejandro Marangoni
Rheological Modelling and Numerical Methods TALKS
A modified Giesekus model 116 Natalie Germann*, Soroush Hooshyar
Micromechanics of cheese sensory texture117Timo Faber*, Han Meijer, Lambert van Breemen117
Simulation of a liquid filament under Rayleigh breakup conditions applied to emulsion structures 118 William Case*, Franz Tanner, Kathleen Feigl, Erich J. Windhab
Numerical investigation of in-nozzle drop breakup conditions for emulsion sprays 119 Kathleen Feigl*, Ahmad Baniabedalruhman, Franz Tanner, Erich J. Windhab
Numerical correction factors of the Couette inverse problem in a Herschel-Bulkley fluid 120 Naser Hamedi*, Johan Revstedt, Eva Tornberg, Fredrik Innings
<b>Simulations of peristaltic flows for Newtonian and non-Newtonian fluids</b> 121 Franz Tanner*, Samer Alokaily, Kathleen Feigl, Abdallah Al-Habahbeh, Erich J. Windhab
Semi-Solid Foods TALKS
<b>Dynamic energy dissipation in protein-networks to determine the texture of food gels</b> 124 Saskia de Jong, Harmen de Jongh*
Tribological properties of microparticulated whey protein as a fat replacer in liquid and semi-solid model foods 125 Kun Liu*, Markus Stieger, Erik van der Linden, Fred van de Velde

Modulating state transitions and visco-elastic behavior of zein resins by plasticizers and co-proteins interactions 126

Katleen Vallons\*, Stefano Renzetti, Daniel Erickson, Albert Jurgens, Osvaldo Campanella, Bruce Hamaker

Measurement of pH micro-heterogeneity in cheese matrices by fluorescence lifetime imaging 127

Zuzana Burdikova, Zdenek Svindrych, Cian Hickey, Martin G Wilkinson, Mark A.E. Auty\*, Diarmuid JJ Sheehan

Particle interactions and structure development in high-protein bars 128	5
Sean Hogan*, Ian O'Loughlin, Phil Kelly	
Pathway of lipid molecules in fat-based food products129Svenja Reinke*, Josélio Vieira, Stefan Palzer, Stefan Heinrich129	I
Biopolymer Solutions and Gels POSTERS	
Oleogels of canola oil with candelilla wax: Rheological characterization and utilization as	
a shortening replacer 132 Suyong Lee*, Woosung Bae, Sanghoon Ko	
<b>Passive microrheology on biopolymers</b> – <b>structure, gel point and applications</b> 134 Roland Ramsch, Mathias Fleury*, Giovanni Brambilla, Gérard Meunier	
Setting the conditions for protein exchangeability by the energy balance and mechanical	
responses Anneke Martin, Jissy Jose, Lenka Tonneijck-Srpova, Harmen de Jongh*, Laurice Pouvreau	
<b>Retention of esters by gellan and pectin solutions or their mixtures</b> 136 Vasiliki Evageliou*, Konstantina Papastamopoulou, Despoina Frantzeskaki, Christalli-Christina Chri- stodoulidou	
<b>Rheological and structural properties of heat induced mixed globular proteins</b> 137 Jissy Jose*, Laurice Pouvreau, Anneke Martin	
Effects of whey protein particles on rheological and microstructural characteristics of an acidified milk model system 138 Guanchen Liu*, Richard Ipsen, Marie Greve	
<b>The effect of surfactant addition on the rheology behavior of ethylcellulose oleogels</b> 139 Maya Davidovich-Pinhas*, Shai Barbut, Alejandro Marangoni	I
Protein aggregation in the presence of cellulose140Jinfeng Peng*, Paul Venema, Krassimir Velikov, Erik van der Linden140	I
Interactions between different milk protein ingredients and exopolysaccharide-producing cultures and their effects on microstructure and textural properties of yogurt 141 Patrizia Buldo*, Connie Benfeldt, Rodrigo Bibiloni, Ditte Marie Folkenberg, Hanne Bak Jensen, Jose Manuel Amigo Rubio, Sander Sieuwerts, Richard Ipsen	
The effect of hydrocolloids on rheological properties of food matrices containing emulsified fat droplet and starch gel 142 Yong-Ro Kim*, Shinjae Park, Sehun Mun	
<b>Physical and rheological study of frozen aqueous solutions containing food biopolymers</b> 143 Cátia Saldanha do Carmo*, Lídia Pinheiro, Catarina Maia, Maria do Rosário Bronze, Catarina Duarte	

#### Tribological and rheological properties of acidified milk drinks prepared with different stabilizers 144

Heidi Liva Nielsen\*, Janne Riis Hansen, Sara Pedersen

# A rheological study of the temperature dependence of the gel-sol and sol-gel transition in gelatin-water systems $$146\end{tabular}$

Thomas Goudoulas\*, Natalie Germann

## Ultrasound homogenization to alter the physical properties of bacterial cellulose aqueous suspensions 147

Paraskevi Paximada\*, Eleni Alkmini Dimitrakopoulou, Styliani V. Protonotariou, Costas Fasseas, Apostolis Koutinas, Ioanna Mandala

#### Microstructural and mechanical properties of alginate gels containing parallel aligned capillaries 149

Anna Ström, Erich Schuster, Anne-Marie Hermansson, Anette Larsson, Kristin Sott\*

### Preparation and rheological characterization of food grade gellan hydrogels with calcium ions as gelling agent 150

Emmanuel Neba Ambebila\*, Diego Mínguez Martínez, Jose Maria Gutierrez, Carmen González Azon

Application of bagasse cellulose nanofibers to make nanocomposite film for food packaging Seyed Mohammad Mousavi\* 151

**Rheological properties of mucilage aqueous solutions (Opuntia ficus indica L. Mill)** 154 Claudia Quinzio, Beatriz Mishima, Laura Iturriaga\*

# Study of the rheological behavior of mixed gels of carrageenan and goat milk whey protein concentrate 155

Anabel Ayunta, Cecilia Puppo, Laura Iturriaga\*



POSTERS

bough root	
Effects of gel from ground chia (Salvia hispanica L.) on the rheological properties of whe dough and bread 1 Viktoria Zettel*, Florian Hecker, Anna Krämer, Bernd Hitzmann	eat 159
Influence of processing on the structure and function of wheat components 1 Linda Brütsch*, Andreas Baumann, Erich J. Windhab	60
<b>Cake ingredients and processing influence batter rheology, cake volume, and texture</b> 1 Maria Christaki*, Tim Van Dyck, Peter Goos, Pieter Verboven, Bart Nicolaï, Johan Claes	61
Effect of blend levels on composite wheat doughs performance made from yam and cassa native starches and bread quality 1 Charlemagne Nindjin*, Georges N'Guessan Amani, Marianne Sindic	<b>iva</b> 162
Rheological behavior of wheat and quinoa blend sourdough and its impact in texture bread Ana Yanina Bustos, Graciela Font De Valdez, Maria Pia Taranto, Laura Iturriaga*	<b>of</b> 163
Influence of Processing on Structure and Rheology POSTE	RS
<b>Rehydration study of instant noodles: Texture versus thermal conductivity</b> 1 Suyong Lee*, Areum Jang, Sanghoon Ko	66
<b>Stabilization of açai juice using homogenization treatments</b> 1 Delphine Huc*, Véronique Bosc, Damien Binois, Gérard Cuvelier	L67
How to enhance satiating ability: rheological and structural approaches related to or processing of yogurt 1 Pere Morell*, Isabel Hernando, Susana Fiszman	<b>ral</b> 168
Powder-liquid mixing process for production of homogeneous food composite structur with low water or fat Katarina Slettengren*, Erich J. Windhab	res 169
Redesigning cheese powder for omission of emulsifying salts 1 Anni Hougaard*, Camilla Varming, Inger Hansen, Ylva Ardö, Richard Ipsen	170
<b>Dissolution and functional properties of pea protein isolate</b> 1 Noel McCarthy*, Krishtina Thapa, Phil Kelly, Deirdre Kennedy, Mark A. Fenelon	171
<b>Rheological and textural properties of cheese chyme during in vitro gastric digestion</b> 1 Xixi Fang, Laurie-Eve Rioux*, Steve Labrie, Sylvie L. Turgeon	172
<b>Contraction measurements of cocoa butter and chocolate with a normal-force controller</b> <b>rheometer</b> Lucie Rejman*, Peter Braun, Erich J. Windhab	l <b>ed</b> 173

Lucie Rejman\*, Peter Braun, Erich J. Windhab

The effects of hydrocolloids on final texture and synersis of native corn starch paste using a Rapid Visco Analyzer 17 Noushin Hosseini*, Amir Farokh Mazaheri	-
Simulation of the flow of semi-dilute guar gum and kappa/iota hybrid carrageenan gun solutions through symmetric and asymmetric contractions.17Douglas Gibson*, M.D. Torres, B. Hallmark, D.I. Wilson17	
Damage of large food particles in simple shear flow17André Braun*, Jennifer Dirscherl, Jörg Zacharias, Heiko Briesen17	6
<b>Changes in rheological and physico-chemical properties of starch during chuño processin</b> Gonzalo Cruz Ortiz, Cristina Ferrero, Laura Iturriaga* 17	-
Microscopic and dynamic vapor sorption characteristics of spray dried model infant formul powders 17	
Deirdre Kennedy*, Mark A.E. Auty, Mark A. Fenelon	
Modulating protein-protein interactions for in-process stability during manufacture of infan formula	
Alan Goodison, Deirdre Kennedy, Alan Kelly, James A. O'Mahony, Mark A. Fenelon*	
<b>Effect of concentration on heat induced formation of whey protein aggregates</b> 18 Aoife Buggy, Sean Hogan, Jennifer J. Mc Manus, Andre Brodkorb, Mark A. Fenelon*	0
Rheological Methods POSTER	S
Application of the rheological measurements to determination of amylolytic activity of malt used for bozo beverage 18 Janyl Iskakova*, Jamila Smanalieva, Asylbek Kulmyrzaev	
Microscopic precursors of fracture in amorphous solids18Med Yassine Nagazi, Mathias Fleury*, Maxime Bazin	4
Interfacial wall-slip and its consequences on Lissajous plots 18 Fabian Birbaum*, Sanna Haavisto, Antti Koponen, Erich J. Windhab, Peter Fischer	5
Gluten free porridge with accustomed rheological properties18Viktoria Zettel*, Julia Gehringer, Bernd Hitzmann18	6
<b>Impact of preshearing conditions on rheology of non-colloidal suspensions</b> 18 Thomas Goudoulas*, Natalie Germann	7
<b>Extensional rheological characterization of food suspensions for optimizing spraying an</b> <b>3D printing</b> Fritz Sörgel*, Fabian Meyer, Francesca De Vito	

Fritz Sörgel\*, Fabian Meyer, Francesca De Vito

<b>Development of a new rheological method in the rennet and lactic coagulation</b> 190 Carlos A. Gracia Fernandez, Michael Schopferer*	I
<b>Temperature ramp rheological method to measure five kinds of mozzarella cheese</b> 191 Carlos A. Gracia Fernandez, Michael Schopferer*	
<b>Determination of flow stability of dietary food fibers by using powder rheometer</b> 192 Fatma Altuntas, Mahmut Dogan, Vildan Gurmeric*, Duygu Aslan	
<b>Viscosity and powder rheology of a traditional powdered dessert: Incir Uyutmasi</b> Sidika Kalitekin, Mahmut Dogan, Duygu Aslan*, Vildan Gurmeric	
Emulsions and Interfaces POSTERS	
Rheology, microstructure and particle size of oil/water/cellulose emulsions used as fat replacers Teresa Sanz*, Ana Salvador	
<b>Rheological properties of O/W/O emulsion containing resveratrol and milk protein</b> 197 Elham Ghorbani Gorji*	
<b>Emulsifying and stabilizing properties of functionalized orange pulp fibers</b> 198 Joël Wallecan*	1
Characterization of olive oil emulsions formed by catastrophic phase inversion using bac- terial cellulose and whey protein isolate 199 Elli Panagopoulou, Apostolis Koutinas, Ioanna Mandala, Vasiliki Evageliou*	
The use of green biopolymers in the formation of double emulsions200Elli Panagopoulou*, Apostolis Koutinas, Ioanna Mandala, Vasiliki Evageliou200	I
Incorporation of epigallocatechin-3-gallate (EGCG) in emulsions containing bacterial cel- lulose 201 Paraskevi Paximada*, Evangelia Papadopoulou, Kleopatra Tsatsaragkou, Elli Panagopoulou, Vasi- liki Evageliou, Apostolis Koutinas, Ioanna Mandala	
<b>Rheological investigations on foaming properties of gas-saturated O/W emulsions</b> 202 Volker Lammers*, Angelika Morant, Erich J. Windhab	
<b>Effect of pectin on the interfacial rheology of</b> β <b>-casein layers at the air/water and oil/water</b> interfaces 203 Noemi Baldino*, Stefania Luzzi, Domenico Gabriele, Francesca Romana Lupi, Bruno de Cindio	

**Preparation of water-in-water emulsions based on gelatin/pectin solutions for food systems** Kateryna Vengel\*, Carmen González Azon, Jose Maria Gutierrez 204

# Impact of structural factors on the fat release from emulsions during digestion in vitro and in vivo 205

Daiva Leskauskaite\*, Ernesta Malinauskyte, Milda Kersiene, Ina Jasutiene

#### Application of response surface methodology and central composite rotatable design to the analysis of egg white protein-pectin electrostatic interaction in a high sugar content system on foaming capacity and foam rheological properties 206 Mitie Sonia Sadahira\*, Maria Isabel Rodrigues, Mahmood Akhtar, Brent S. Murray, Flavia Maria

Netto

Influence of pH on foaming and rheological properties of aerated high sugar system with egg white protein and hydroxypropylmethylcellulose 207 Mitia Senia Sedebire\* Maria Jackel Bedrigues Makmaed Alkhter Brent & Murray Elavia Maria

Mitie Sonia Sadahira\*, Maria Isabel Rodrigues, Mahmood Akhtar, Brent S. Murray, Flavia Maria Netto

The effect of ultrasound on the rheological properties of emulsions208Duygu Aslan\*, Mahmut Dogan208

Better foam stability via interplay between β-lactoglobulin and HM-pectin209Dilek Ercili-Cura\*, Riitta Partanen, Martina Lille, Päivi Myllärinen, Pirkko Forssell

**Soft tribology of oil-in-water emulsions: Whey protein versus silica nanoparticle stabilization** Lubica Macakova\*, Johan Andersson, Jan-Willem Benjamins 210

N.

#### Cheese, Chocolate, and Health

# Rheological and sensory properties of instant hot chocolate: Application different multi-<br/>criteria decision techniques to determine optimum fat composition212Mahmut Dogan\*, Duygu Aslan, Tugba Aktar, Meryem Goksel212

POSTERS

Rheo-kinetics of the enzymatic degradation process of thickened fluids for dysphagia patients 213

Getachew Assegehegn, Mihaela Turcanu, Sebastien Secouard, Nadja Siegert, Edmundo Brito-de La Fuente, Críspulo Gallegos Montes\*

Influence of alpha-amylase addition on the elongational behaviour of thickened fluids for dysphagia management 214

Getachew Assegehegn, Mihaela Turcanu, Sebastien Secouard, Nadja Siegert, Edmundo Brito-de La Fuente, Críspulo Gallegos Montes\*

Effect of saliva (mouth digestion) and in vitro stomach digestion on the rheological properties of vegetable purees made with hydrocolloids to enhance satiety 215 Ana Salvador\*, Teresa Sanz

Intragastric rheological properties and their relationships with the satiating capacity of cheese pies with glucomannan 216

Susana Fiszman\*, Johanna Marcano

<b>The structural properties of puffed breakfest cereals with plant based fibre additives</b> 217 Dace Klava*, Vitalijs Radenkovs, Daiga Konrade, Solvita Kampuse, Tatjana Rakcejeva, Jekaterina Litvinova
Specific and unspecific adhesion of gut bacteria218Patrick Rühs*, Lukas Böni, Lukas Böcker, Peter Fischer, Christoph Jans, Tomas De Wouters218
Is physical strength related to oral processing of food structure?219Richard Asensio-Barrowclough, Laura Laguna*, Anwesha Sarkar
Lipase-induced structural modification of anhydrous milk fat 220 Sean Hogan*, Phil Kelly
Colloidal Dispersions POSTERS
Rheological interactions of natural micro-particle food polymers in different temperaturesMahmut Dogan*, Duygu Aslan, Vildan Gurmeric222
Structure properties and crystallization kinetics of diverse palm oils for nougat spread production with small amounts of high melting triglycerides and different particle size distributions of sugar 223 Stephen-Sven Hubbes*, André Braun
<b>Effect of structural parameters on rheological behaviour of apple cells dispersions: A com- parison of real systems and model systems.</b> 224 Cassandre Leverrier*, Giana Almeida, Gérard Cuvelier
Characterisation of protein aggregates in milk protein concentrate226Sophie Gaspard*, James A. O'Mahony, Alan Kelly, Mark A. Fenelon, Andre Brodkorb
Characterisation of the flowability of the instant pudding and baking powder by using powder rheometer 227 Mahmut Dogan*, Vildan Gurmeric
<b>Composition, morphology and pasting properties of orchis anatolica tuber gum</b> 228 Birsen Bulut-Solak*, James A. O'Mahony
Rheological Modelling and Numerical Methods POSTERS
Modelling the growth of bubbles in a range of non-isothermal generalised Newtonian fluids Michael Sargent* 231
Fractional equations for the firmness, rubberiness, and springiness of food gels232Timo Faber*, Aditya Jaishankar, Gareth McKinley
Particle-filled meat protein gels: Investigating the role of particle size and protein/filler interactions 234 Andrew Gravelle*, Alejandro Marangoni, Shai Barbut
Computational fluid dynamics for pasta extrusion236Richard Vonlanthen, Jean-Philippe Dellemann*236

Semi-Solid Foods POST	ERS
Investigation of rheology of semi-hard cheeses using fluorescence spectroscopy and o mometrics Zhyldyzai Ozbekova*, Asylbek Kulmyrzaev	c <b>he-</b> 238
Influence of chemical and biochemical parameters on texture of Appenzeller cheese Dominik Guggisberg*, Hans Winkler, Ueli Buetikofer, Daniel Wechsler	239
Effect of storage conditions on quality characteristics of dietary biscuits made with curr paste Eirini K. Nikolidaki*, Styliani V. Protonotariou, Andriana Lazou, Vaios T. Karathanos, Ioa Mandala, Spyros E. Papadakis, Nikolaos P. Zogzas	240
Effect of carob flour particle size on the gelling properties of rice flour Kleopatra Tsatsaragkou*, Theodora Kara, Christos Ritzoulis, Ioanna Mandala	241
Rheological investigation of starch based edible coatings for ready-to-eat potato produc Olga Mileti, Francesca Romana Lupi*, Noemi Baldino, Domenico Gabriele, Bruno de Cindio	
Relationships among small-strain rheological behavior, large-strain rheological behavior and structure of different mashed potato preparations Helen Joyner*, Alexander Meldrum	<b>vior,</b> 243
Physicochemical and viscosity of honey from Côte d'Ivoire Amoin Georgette Konan, Charlemagne Nindjin*	244
Factors affecting shelf life and quality attributes of currant-paste Eirini K. Nikolidaki*, Georgia S. Skoumpi, Spyros E. Papadakis, Nikolaos P. Zogzas	245







# **Opening Lecture**

# Structural and mechanical anisotropy in sheared colloidal gels

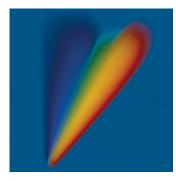
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Colloidal gels are important in many areas of science and technology, including foods. Their mechanical response is particular as they undergo a solid-to-liquid transition as a function of the applied shear stress. In terms of material functions, the rheological properties of aggregated suspensions are described by an elasticity that depends on shear history, a yield stress, and a viscosity that not only changes reversibly with shear rate but also with time. The latter effect is known thixotropy. These rheological effects find their origin in the microstructure and how it reacts to flow. At rest, colloidal gels are typically made up of a network of reversible colloidal clusters or flocs. The mechanical response of this structure is solid like, and stresses are transmitted by direct contact forces with the magnitude of the moduli strongly depending on concentration and the number of contact points. However, the difficulty with this kind of network structures is that they are inherently metastable. Hence, in addition to physicochemical details (the volume fraction, the pair potential), the flow history plays an important part in the details of the microstructure. Most theoretical concepts are based on an isotropic gel structure, and once the systems starts to flow, the non-Newtonian response is often attributed to a shear rate dependence of an isotropic floc size and density. Microstructural studies reveal a pronounced anisotropy in the structure factor, which is then often ignored, because as yet there was no evidence for pertinence of mechanical anisotropy in these systems. In the present work we demonstrate the so far unaccounted presence of a spectacular anisotropy in the mechanical response of a pre-sheared gel, with especially the elastic moduli differing by as much as two orders of magnitude. This implies that directional nature by which stresses are transmitted in these systems have to be taken into account in modeling efforts, features shared with materials near jamming transitions or granular media







Keynotes

# Food rheology and structure for nutritionally enhanced foods

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The development of novel food manufacturing processes, food product formulations and ingredients is increasingly driven by the growing demand for nutritionally enhanced foods. The nutritional requirements for processed foods vary across the globe, e.g., from less energy dense to more energy dense, from protein enriched to salt and sugar reduced, or substitution of ingredients with natural alternatives. In recent years we have been looking at hydrocolloid ingredient processing as well as complex emulsion design based approaches to contribute to the formulation of salt reduced foods. The hydrocolloid processing route confirmed the unusual behavior of xanthan gum with regard to oral release of tastants compared to other molecularly dissolved hydrocolloid thickeners. Only in presence of saliva, rheological analysis highlighted different behavior. Similarly, considering the functionality of saliva and the mechanical action of oral processing in the design of complex emulsions opens up new formulation strategies for oral release of tastant. Combined with recent advanced in the understanding of emulsion formulation based on particulate emulsifiers, an example of a salt releasing emulsion will be shown.

7<sup>TH</sup> INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

#### Effect of competition for $Ca^{2+}$ on the morphology and the elasticity of mixed gels of $\kappa$ -carrageenan and $\beta$ -lactoglobulin

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The polysaccharide  $\kappa$ -carrageenan is sometimes added to milk based products in order to improve the texture.  $\kappa$ -carrageenan gels reversibly below a critical temperature that can be reduced by adding small amounts of specific ions such as  $K^+$ .  $\beta$ -lactoglobulin is a globular protein and is the major protein component of whey.  $\beta$ -lactoglobulin aggregates irreversibly when heated and gels above a critical concentration. The aggregation and gelation of both  $\kappa$ -carrageenan and  $\beta$ -lactoglobulin are very sensitive to the presence of calcium ions that are most often present in the food products. Therefore it is important to understand the effect of  $Ca^{2+}$  on gelation of mixtures. It was reported that  $\beta$ -lactoglobulin in aqueous solutions at neutral pH forms microgels when heated in the presence of  $CaCl_2$ . Here the effect of adding  $CaCl_2$  to mixtures of  $\kappa$ -carrageenan and  $\beta$ -lactoglobulin microgels at neutral pH on the morphology and the elasticity is presented. The  $\beta$ -lactoglobulin microgels were formed by heating either before or after mixing with  $\kappa$ -carrageenan. It will be shown that both  $\beta$ -lactoglobulin and  $\kappa$ -carrageenan specifically bind  $Ca^{2+}$  and that in mixtures they compete for  $Ca^{2+}$ . This influences aggregation and gelling of each biopolymer. In addition, thermodynamic incompatibility drives micro phase separation above a critical  $\kappa$ -carrageenan concentration with  $\beta$ -lactoglobulin microgels. An attempt is made to disentangle the effects of competition for  $Ca^{2+}$ and thermodynamic incompatibility on the structure and the elasticity of the mixtures.

# Casein hydrogels: Water holding capacity related to swelling and rheology of caseinates

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We developed a new method to characterize water holding capacity (WHC) by immersing defined pieces of a gel in solutions with varying salt concentration, pH and temperature. The gel pieces swell or shrink depending on conditions and after some time they attain an equilibrium volume which can be defined as the WHC. Flory-Rehner [1] theory extended with a Donnan osmotic pressure term [2] described the experimental data quite well. Theory suggests that the properties of the individual polymers (caseins) and their crosslinking determine the WHC. We therefore investigated the low to medium concentration properties of different caseinate dispersions. It was found that the overlap concentration correlated directly with the WHC. Caseinate dispersions enriched in  $\alpha_{s1}$ -casein showed an overlap concentration which was almost a factor of two lower than of sodium caseinate. We ascribed the improved WHC to the telechelic character of  $\alpha_{s1}$ -casein which can be considered as a protein polyelectrolyte with two hydrophobic blocs connected with a hydrophilic block and therefore increases the interaction between the casein molecules. The working hypothesis was verified by mildly crosslinking caseins using trans-glutaminase [3, 4].

[2] Shibayama M, Tanaka T: Volume phase-transition and related phenomena of polymer gels. Advances in Polymer Science 109 (1993) 1-62.

[3] de Kruif CG, Anema SG, Zhu C, Havea P, Coker C: Water holding capacity and swelling of casein hydrogels. Food Hydrocolloids 44 (2015) 372-379.

[4] de Kruif CG, Bhatt H, Anema SG, Coker C: Rheology and water holding capacity of caseinate fractions. Submitted.

<sup>[1]</sup> Flory PJ, Rehner J: Statistical mechanics of cross-linked polymer networks II Swelling. Journal of Chemical Physics 11 (1943) 521-526.

# Insights into food design and oral processing using soft matter physics, thin film rheology, and tribology

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An emerging driver for consumer choice and acceptability of foods and beverages is their mouthfeel and texture. These are challenging to consider since they evolve during and after consumption and they are not an explicit physical property. Rheology is routinely used in food design due to its relationship to physical stability and initial texture perception [1]. However, as food is transformed during oral processing, the physical mechanisms contributing to the dynamics of texture and mouthfeel perception in the oral environment are complex and not well understood. Here we highlight recent advances in oral processing and our own research to combat such complexity and develop physical techniques that enable in vitro measurement of relevant properties of food systems, which span liquids, multiphase fluids and brittle solids. These techniques seek to probe the physics experienced by the food components during oral processing as well as their interaction with saliva and the mucosal films lining the oral cavity. As well as considering oral physiology, we also develop multi-scale deformation processes that includes tribology (friction) and thin film rheology that characterizes the micromechanics of food and/or food-bolus [2]. Uncovering the physical basis of texture and mouthfeel provides potential approaches for rational design in order to meet the challenge of re-engineering foods and beverages with superior mouthfeel and acceptability whilst delivering benefits to health and well-being of consumers.

Selway N, Stokes JR: Annual Review of Food Science and Technology 5 (2014) 373-393
 Stokes JR, Boehm MW, Baier SK: Current Opinion in Colloid and Interface Science 18 (2013) 349-359

Keynotes

# Mixing of construction materials on different length scales

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Many construction materials share the amazing property of being shapeable during the first hours of their industrial life. These complex granular pastes can therefore be molded, sprayed, vibrated, extruded, roller-compacted or injected to produce elements displaying strength and durability properties mimicking natural stones. These materials also share the uncommon property of being heterogeneous at length scales of observations only one order lower than the length scale of the final elements of industrial interest. Most of the above shaping processes require therefore that local heterogeneities and their consequences on the hardened element properties stay at an acceptable level for the industrial application. This all starts at the mixing process level, during which drastic concentration gradients have to be erased in less than a few hundreds of seconds. There exists for the most common mixing processes in the construction industries some specific literature dedicated to advanced computational fluid mechanics or distinct element methods allowing for the detailed prediction of the processes themselves. In order to capture the general underlying physics, we will however not enter here the details of these studies but rather describe industrial construction materials mixing processes through the use of various dimensionless numbers from the chemical processing and suspension rheology literature (diffusion-based and convection-based mixing, shear induced particle migration, granulation), which dictate the dominating physical phenomena and therefore the final success and duration of the entire mixing process.

### Gastric Structuring: How food structure and rheology impact on fat digestive processing

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Fat digestion (lipolysis) plays an essential, if somewhat controversial role in our diet. Lipolysis facilitates the absorption of essential lipid/lipid soluble nutrients particularly during in early years, yet over consumption of fat is cited as a leading cause of obesity in adults. Hence, understanding and manipulating fat digestion has been an active topic of research for more than four decades. The simple fact that lipases are activated upon adsorption to emulsion interfaces means that food structure can play a central role in understanding and 'control' of fat digestion. This article presents an overview of our recent work examining how control of emulsion (in)stability within the stomach can be used to impact the speed of fat absorption from the intestine. Starting with model systems the mechanisms behind the emulsion destabilization are described using in vitro and in vivo data. MRI imaging studies are then used to follow the dynamics of; i) initial gastric structuring and then ii) droplet re-dispersion. Finally emulsions are incorporated into different biopolymer networks to understand how different matrix rheologies impact the dynamics of emulsion structuring. Throughout this story simple food structuring concepts are used to understand how foods are processed by the stomach during digestion and how they can be used to achieve dramatically different lipid absorption kinetics.

#### Interplay of rheology and functional structure processing along the food value chain

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Our process engineering and material science integrating research approach follows a holistic consideration of the entire food value chain from the raw material production to the sensory and metabolic responses of the human body. To optimize related functionality, multi-scale Process-Structure-Property relationships (S-PRO2) are taken into account throughout the entire food value chain (FVC) and in a reverse engineering order. Rheological characteristics of multiphase liquid or semi-solid food systems are non-Newtonian and mostly viscoelastic. Tailoring food product rheology is of relevance for consumers' oro-gastro-intestinal (OGI) processing to satisfy preferences, acceptance and needs (PAN). Related 'rheo-functional optimization' is most versatile, flexible, and efficient if considered during each processing step along the FVC. Accordingly we investigated novel processing modules to expand the frame for optimization of rheological food characteristics in the context of related food quality and sustainable food production improvements. Processing domains identified along the FVC are (i) field pre-processing, (ii) food factory processing, (iii) home processing, and (iv) OGI processing. The functionalized food structure perspectives (i - iii) generate what (iv) disintegrates while releasing the consumer-relevant target functions in a controlled manner concerning site, kinetics and physiological interaction. A detailed insight into related food processing developments within the mentioned modules (i - iv) will be given and related impacting factors on the processed food rheology be addressed. Accordingly we refer to the technologies of rubbery milling, dynamic membrane dispersing/encapsulation, spray-drying/chilling, and emulsion powder reconstitution. Finally related rheologically modified model food systems were tracked into the OGI flow processing domain. Within this domain we started dealing with in vitro and in vivo experimental as well as numerical simulation based flow characterization addressing the human mouth, esophagus, stomach, and small intestine. Rheology and related OGI flow characteristics determine the transport phenomena of functional food components to specific receptors. This is equally relevant for flavor perception in the mouth and micronutrient availability in the small intestine. Within this work we mainly focus on emulsions/emulsion droplets as macroscopic, their interfaces as mesoscopic and their main constituents as macromolecular structural food entities and follow their track along the food value chain. Consequently it's the non-Newtonian rheology of emulsions being particularly addressed. In order to characterize velocity fields in industrial processing and OGI flows, least invasive methods like magnetic resonance imaging, ultrasound-Doppler velocimetry, or high resolution computer tomography were applied.

<sup>[1]</sup> Feigl K, Tanner FX, Holzapfel S, Windhab EJ: Chem. Eng. Sci. 116 (2014) 372.

<sup>[2]</sup> Nahar S, Jeelani SAK, Windhab EJ: Chem. Eng. Comm. 200 (2013) 820

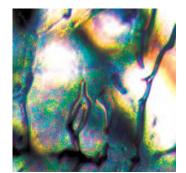
<sup>[3]</sup> Rühs PA, Affolter C, Windhab EJ, Fischer P: J. Rheol. 57 (2013) 1003.

<sup>[4]</sup> Tanner FX, Al-Habahbeh AA, Feigl K, Nahar S, Jeelani SAK, Case WR, Windhab EJ: Appl. Rheol. 22 (2012) 63910.

<sup>[5]</sup> Scheuble N, Geue T, Windhab EJ, Fischer P: Biomacromolecules 15 (2014) 3139.







## Biopolymer Solutions and Gels

#### Rheological behavior of dense milk protein suspensions in the presence of minerals

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Caseins, the most abundant protein in milk (80%), are assembled into spherical complexes with a radius of about 100nm called casein micelle. The casein micelles are held together by nanoclusters of colloidal calcium phosphate (CCP). When the CCP is removed by acid precipitation, the casein molecules can be resolubilized with NaOH to obtain sodium caseinate (NaCas). In aqueous solution NaCas assembles into spherical particles with a radius of about 10nm. The viscosity of dense NaCas suspensions strongly increased with increasing concentration due to jamming of the particles. Increasing the temperature led to a decrease of the viscosity because the repulsion between the particles and thus their effective volume fraction decreased. Oscillation shear measurements showed that the dense suspensions were visco-elastic with a terminal relaxation time that increased with increasing concentration and decreased with increasing temperature. In most food applications different types of minerals are present and it is thus important to characterize their effect on the texture of dense NaCas suspensions. In first instance, we have studied the influence of calcium ions on the structure and the rheological properties of dense NaCas suspensions. It was found that the terminal relaxation time increased with increasing  $Ca^{2+}$  concentration. However, the high frequency elastic modulus decreased with increasing  $Ca^{2+}$  concentration. Microscopy showed the formation of micron sized dense flocs above a critical  $Ca^{2+}$  concentration. We will show that these results caused by attractive interactions induced by  $Ca^{2+}$ . Secondly, we have studied suspensions of NaCas that contained orthophosphate in addition to calcium. The calcium and phosphate were either introduced as pure minerals or by mixing NaCas with casein micelle that contain CCP. It led to an increase of the viscosity of the dense suspensions or even gelation. The effects on the rheology at a given protein concentration depended in a subtle manner on the molar ratio of phosphate and calcium, the pH and the temperature.

### Solvent transportation behavior of mechanically constrained agarose gels

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Agarose gels are extensively applied in processed foods in Japan. It is an important issue to investigate the solvent transportation behavior form the edible gels since it is strongly relates to the flavor release. Although solvent transportation from edible gels has been extensively studied, most of these studies focused on the passive diffusion of the flavor from edible gels. In this study, we attempted to investigate the solvent transportation behavior when the gels constrained. The mode of transport of solvent from compressed agarose gels that were quenched at various temperatures was investigated. The compression load and the volume of the agarose gels decreased with time during compressive restraint. The decrease in volume was induced by squeezing of the solvent from the gel by compressive restraint. Relaxation of the compression load and the decrease in volume of the gels could be analyzed using a stretched exponential function; moreover, interestingly, both time constants were coupled. It is proposed that relaxation of the compression load was induced by solvent transportation (squeezing out). To study the relationship between the structure of the gels and the solvent transportation behavior, the gels were prepared at various quenching temperatures and investigated. The hydrogel prepared at just below the sol-gel coexisting temperature  $T_{qel}$  exhibited distinguishing behavior, characterized by an increase in the rate of the solvent transportation. It is postulated that micro-phase separation occurred in the sample quenched close to  $T_{ael}$  due to spinodal decomposition, with consequent formation of relatively large solvent paths in the gel. Turbidity measurements and small angle light scattering data supported this postulate.

### Molecular interactions, morphology and macroscopic properties of mixed protein-protein systems

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Selective mixing of gel forming ingredients can be used to produce foods with a wide range of textural properties. Whereas protein/polysaccharide mixtures are widely studied, protein/protein mixtures are researched to a much lesser extent. Rheological and microstructural properties of different globular protein/gelatin mixed gels were investigated. The molecular interactions between these proteins were quantified in terms of their virial coefficient and used to explain microstructural and rheological changes during independent gelation of each of the proteins in the presence of the secondary protein. Results showed that only when analyzing mixed systems over a large range of length scales (molecular to macroscopic, nm to cm) one is able to explain properties observed on a macroscopic level, such as fracture properties or water holding. In mixtures of globular proteins (soy, whey) and gelatin both proteins were able to form continuous networks if triggered to gel. The presence of the other, secondary protein allowed alteration of the gel properties (on micro and therefore macroscopic length scales) dependent on the size and interaction between proteins. The gained insights into mixed protein systems point out the possibilities how intentionally choosing proteins based on their size and interaction allows one to steer microstructure development during gelation. This is interesting not just for research but also for the development of protein gels with desired texture and sensory properties.

### Local diffusion in heterogeneous food microstructures determined by quantitative confocal microscopy

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Diffusion is vital for many food properties such as water management in pasta and pastry products, oil migration induced fat bloom in chocolate and oral taste release. It is equally important for controlled release of drugs, liquid management in hygiene and wound care products and chromatography applications. These examples show that it is important to have good control over the diffusion properties to obtain desired functionality. Therefore thorough understanding of structure - mass transport relationships at different length scales in the structure and good measurement techniques for global and local for diffusion are essential. In this talk, the coupling between structure and diffusion at different length scales in Foods and soft porous heterogeneous materials will be discussed [1]. Quantitative microscopy allows for simultaneous determination of the detailed microstructure at micrometer level and local quantitative information regarding mass transport, electrostatic interactions, rheological properties etc. A brief overview of different microscopy based techniques to characterize local diffusion will be given in this presentation. Confocal laser scanning microscopy (CLSM) in combination with flourescence recovery after photobleaching (FRAP) or raster image correlation spectroscopy (RICS) are versatile methods to determine quantitative diffusion properties locally directly in the microscope. They can be used in many types of soft porous homogeneous and heterogeneous foods and biomaterials. In FRAP, fluorescently labelled molecules are bleached locally and the rate of intensity recovery is proportional to local diffusion rate. In RICS, the concentration fluctuations are analyzed to enable information about the local diffusion rate. Here, examples from the use of FRAP and RICS will be presented. Food properties change as a function of time and surrounding conditions. CLSM-FRAP combined with different stages to control surrounding conditions is powerful to monitor effects of kinetics on the diffusion properties. Here, results on microstructure and probe diffusion in phase separated biopolymer mixtures determined by FRAP will be presented. Possibilities of CLSM-FRAP and new pixel-based models to quantitatively monitor diffusion in alginate gels, and fat crystallization in chocolate will be shown [2 - 4]. It was found that tempering and solid particle additions have a large impact on the time-dependent fat migration in chocolate model systems. Recent advances in cell science that allow for determination of interaction parameters using FRAP has been applied to biopolymer gels. Very recent results that reveal the effects of charge density, size and concentration on diffusion of negative probes in positively charged  $\beta$ -lactoglobulin gels will be presented [5].

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- [2] Deschout et al.: Optics Express 18 (2010) 22886-22905.
- [3] Schuster et al.: Soft Matter (2014) DOI: 10.1039/c3sm52285g.
- [4] Svanberg et al.: Food Research International 44 (2011) 1339-1350.
- [5] Schuster et al.: Biophysical J. 106 (2014) 253-262.

#### Ultrasonic effect on the rheology of protein solutions

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Ultrasound processing has many applications in a wide range of sectors, yet the fundamentals of the ultrasonic effect upon biopolymers are to be fully understood. Little research involving the ultrasound treatment of proteins has been conducted, in particular the effect of ultrasound treatment upon the rheological properties of protein solutions. Proteins presented in our study, three dairy, three animal and three vegetable, were treated with ultrasound and probed to understand how ultrasound impacts the rheological properties of proteins. Solutions of sodium caseinate (NaCas), whey protein isolate (WPI), milk protein isolate (MPI), bovine gelatin (BG), fish gelatin (FG), egg white protein (EWP), rice protein isolate (RPI), soy protein isolate (SPI) and pea protein isolate (PPI) were ultrasound treated for two minutes with a power intensity of  $\sim 34W/cm^2$ . The effect of the ultrasound treatment on protein structure was investigated using size measurements and SDS-PAGE, and rheologically using bulk viscosity, intrinsic viscosity and critical concentration. Ultrasound treatment reduces the aggregate size of all proteins due to the hydrodynamic shear imparted by ultrasonic cavitations. SDS-PAGE confirmed that ultrasound provides insufficient energy to cause scission the primary amino acid sequence, therefore no hydrolysis. The bulk viscosity of protein solutions was measured at a range of protein concentrations. Ultrasound treatment of protein solutions yielded a consistent reduction in bulk viscosity, due to the reduction in protein size. Ultrasound treatment reduces the intrinsic viscosity of proteins, a measure of the hydrodynamic volume of proteins in solution, which is consistent with size measurements. The critical concentration of proteins increased upon treatment with ultrasound. In conclusion, ultrasound treatment reduces the protein size by disrupting inter- and intramolecular forces. SDS-PAGE confirmed that scission of the primary amino acid sequence does not occur. The bulk viscosity of protein solutions is reduced due to the reduction in hydrodynamic volume of proteins, as measured by both size measurement and intrinsic viscosity.

#### Rheological properties of milk derived peptides in high-protein matrices

<u>Lélia Lacou</u><sup>1</sup>, Marie-Hélène Famelart<sup>1</sup>, Julien Jardin<sup>1</sup>, Valérie Briard-Bion<sup>1</sup>, Sébastien Lê<sup>2</sup>, Stéphane Pezennec<sup>1</sup> and Valérie Gagnaire<sup>1</sup>

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Proteins are one intrinsic part of many food products. They can be hydrolyzed into peptides during food processes or storage. Protein hydrolysis can enhance the functional properties of the food products depending on the type of peptides produced. Most of information on peptide functional properties currently comes from dilute solutions, in which both the composition and the protein concentration are far away from food products. However, additional information is required to understand the self-assembly of proteins and peptides in complex matrices. Pools of peptides are usually not characterized and a fortiori it is difficult to determine how, and to which extent, the protein functional properties are modified due to protein hydrolysis. To answer these questions, we produced pools of peptides with defined physicochemical characteristics (size, charges and hydrophobicity) through the action of two enzymes. Sodium caseinate was hydrolysed by trypsin and Glu-C, giving three pools per enzyme, which contain peptides quantitatively and qualitatively different. Peptides were identified by high resolution tandem mass-spectrometry and their structure was determined by Fourier transform infrared spectroscopy. Controlled amount of these peptides were incorporated into high-protein matrices made of casein micelles. Some of these matrices are liquids whereas the others are gels. Then, rheological properties of matrices were studied by flow and small amplitude oscillatory measurements. Advances in sequence identification and structural determination have made possible to characterize the peptides present in the hydrolysates and those that interact with casein micelles. Under our conditions, peptides with specific physicochemical and structural characteristics modified the rheological properties of matrices. This innovative approach gives new ways to establish a relationship between physicochemical, structural characteristics of peptides and their functional properties in complex matrices. This method could be enlarged to various food matrices.

### Pre-gelatinized tapioca starch and its mixtures with xanthan and *ι*-carrageenan

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Starch is one of the most widely used stabilizers, thickening and gelling agents in the food industry. Already since the early history, humans have always eaten starchy foods derived from seeds, roots or tubers. Tapioca starch is obtained from the roots of the cassava plant and due to its relatively low amylose content ( $\sim$  17 %) the food application concentrates mostly on thickening and stabilizing effects. At low concentration ( $< 10 \ \text{w/w}$ ) tapioca starch builds high viscous and translucent pastes with no taste of its own. In general, the application of starch as thickening or gelling agent always requires the addition of heat. In this work, a cold water soluble tapioca starch is produced by spray-drying, a very sensitive and common drying process. After this procedure, the paste of the rehydrated tapioca starch shows a significant loss in elasticity. The combination with other food hydrocolloids, like xanthan or *i*-carrageenan, helps to rebuild the elasticity and creates a new tapioca starch-based paste with a stable structure. Rotation and oscillatory rheological measurements show significant differences in viscosity, shear-thinning behavior, temperature dependence, stress relaxation and strain dependence between the native tapioca starch paste and the spray-dried one. The addition of the non-gelling agents, xanthan and  $\iota$ -carrageenan, alters these behaviors for the spray-dried tapioca starch much more pronounced. Here, the mixture is more dominated by the hydrocolloid than in the native starch paste. The polyelectrolytes xanthan and  $\iota$ -carrageenan differ significantly in their chain flexibility which raises disparate effects on the mechanical properties of the pastes of the native and the spray-dried starch. It is assumed, that the mixture of starch and  $\iota$ -carrageenan leads to a coupled network with intermolecular bindings between the hydrocolloid and the starch molecules, whereas xanthan provokes a defect in the starch network resulting in a phase separation [1].

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### Process stable whey protein-pectin complexes as new structuring elements in fat reduced food systems

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Pectins and whey proteins are often used as stabilizers in diverse dairy products. Mixing these two biopolymers in an aqueous solution results in the formation of electrostatically attracting complexes. It is postulated that these complexes can be used as structuring agents in fat reduced food systems. However, since electrostatically stabilized complexes are highly sensitive to dissociation due to changes in their local environment, e.g. heating, shearing and pH change, a major challenge is to generate whey protein-pectin complexes that maintain their expected functionalities during processing and when added in different food systems. The aim of this study was to identify process conditions to generate process stable whey protein-pectin particles with improved functional properties in comparison to the biopolymers alone. For this purpose, we studied the response of those associated complexes to changes in temperature, shearing and pH. Biopolymer mixtures consisting of native whey protein and pectin with varying degree of esterification have been generated at a ratio of 5:1. The biopolymer mixtures were heat-treated for 250 s at 80 - 90°C. By post heating acidifications at pH 6.1 to 4.0, complex stability in different food matrixes such as yoghurt has been assessed. Formed complexes have been sheared at different process steps to investigate their stability during processing. Generated structures were characterized by means of turbidity, electrical charge, particle size, microscopic analysis, and fluorescence spectroscopy. Results revealed that the structural and mechanical characteristics of whey protein-pectin particles are strongly affected by heat treatment and acidification. In particular, heat treatment is a promising way to generate process stable whey protein-pectin particles. Particle size analysis indicated that pectin-stabilized whey protein aggregates are able to meet size characteristics of milk fat globules (1 - 10  $\mu$ m) and thus might have potential to replace parts of fat in fermented dairy products. This study outlines that functional properties of whey protein-pectin particles can be designed towards the desired application in food systems.

### Polymeric assembly of gluten proteins in an aqueous ethanol solvent

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The supramolecular organization of wheat gluten proteins is largely unknown due to the intrinsic complexity of this family of proteins and their insolubility in water. We fractionate gluten in a water/ethanol (50/50 v/v) and obtain a protein extract which is depleted in gliadin, the monomeric part of wheat gluten proteins, and enriched in glutenin, the polymeric part of wheat gluten proteins. We investigate the structure of the proteins in the solvent used for extraction over a wide range of concentration, by combining X-ray scattering and multi-angle static and dynamic light scattering. Our data show that, in the ethanol/water mixture, the proteins display features characteristic of flexible polymer chains in a good solvent. In the dilute regime, the protein form very loose structures of characteristic size 150 nm, with an internal dynamics which is quantitatively similar to that of branched polymer coils. In more concentrated regimes, data highlight a hierarchical structure with one characteristic length scale of the order of a few nm, which displays the scaling with concentration expected for a semi-dilute polymer in good solvent, and a fractal arrangement at much larger length scale. This structure is strikingly similar to that of polymeric gels, thus providing some factual knowledge to rationalize the viscoelastic properties of wheat gluten proteins and their assemblies.

### A rheological and microstructural characterisation of monoglycerides/olive oil organogels

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The structuration of oil phases via organogelation is an interesting physical technique to replace chemical processes, such as hydrogenation or transesterification, commonly adopted to increase consistency and structuration of the fat phase. In the present work, olive oil/monoglycerides organogels were prepared with different organogelator amounts, and their mechanical characteristics were studied with small amplitude oscillation tests within the linear viscoelastic regime, cooling the system, at a fixed cooling rate. Both onset of crystallization temperature  $T_{co}$  and gelation temperature  $T_g$  of organogels were evaluated by rheological temperature ramp tests, whereas other techniques (NMR, DSC) were used to determine only the  $T_{co}$ , confirming what found by the rheological analysis. Solid fat content (SFC) was evaluated by NMR spectroscopy, and a fractal model was used to relate rheological properties to the microstructure of the system. According to the microstructural transitions deduced by rheological results, all samples showed an evolution from a liquid to a crystallized system (comparable to a suspension of crystals in a liquid oil phase), which takes place at  $T_{co}$ . At lower temperature (named  $T_g$ ) a network formation, arisen from crystals aggregates, was also observed. When organogelator fractions are higher than 0.034 w/w  $T_{co}$  and  $T_g$  are equal suggesting that crystallization and network formation occur simultaneously. The structure development rate of organogels was determined from both rheological (SDR) and SFC data (SDR-NMR), and both of them were related to the organogelator fraction. In particular, SDR-NMR followed a power-law trend with organogelator fraction, whereas SDR curve showed also a saturation behavior for high organogelator fractions added to the system, evidenced by an almost constant region of data for monoglycerides amount larger than a specific value. The rheological and microstructural results reported in the present work could be used to predict the characteristics of a novel fat based on olive oil organogels, from both a mechanical and a thermo-rheological point of view.

### **Electrostatic gels between** β-lactoglobulin and xanthan gum: Properties and potential applications

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An interesting gelled system has been discovered using native proteins and polysaccharides at very low concentrations. Gelation was induced by in-situ acidification, at room temperature, to a pH where both molecules carry net opposite charges. The novelty of this study resides in that no thermal, enzymatic or any other denaturing treatment was applied to the protein or the mixture at any time. Therefore, this gelled system presents potential uses in food industry to enhance the stability of foods, to protect micronutrients, and to deliver, protect drugs or active molecules as application in pharmaceutical industry. Gelation processes of protein-polysaccharide systems were monitored by viscoelastic measurements and microstructure of gels was observed by confocal laser scanning microscope. The gelation mechanism was identified for a model system of  $\beta$ -lactoglobulin and xanthan gum by which gel network was based on initial network of xanthan gum,  $\beta$ -lactoglobulin aggregated along the xanthan gum chains and could be regarded as a crosslinking agent. A strong relation between gel microstructure, texture, and waterbinding properties were found, and the gel microstructure and its properties could be controlled by several environment factors including biopolymer ratio, total solid concentration, and ionic strength. Since the gel is stabilized by electrostatic interactions between proteins and polysaccharides; the gel is easily destabilized by change of pH or increasing ionic strength of medium. Interestingly, a heat treatment at 80°C during 30 minutes enhanced the gel pH stability and improved its waterbinding capacity. However, the heat treatment did not change gel's pH reversible property. This functionality is attractive for the design of physical gels geared toward biomedical applications or controlled release of bioactive molecules.

# The influence of pH on the proteolysis and self-assembling of partially hydrolyzed bovine $\alpha$ -lactalbumin into nanotubes and gels

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Partially hydrolysed  $\alpha$ -lacalbumin has been found to be able to self-assemble into nanotubes and form translucent gels [1]. These nanotubes and resultant gels have great potential for application in the food system as well as for use in the pharmaceutical and non-food fields due to their excellent properties such as thickening agent, gelation and encapsulation. The formation of the nanotubular structure and gels requires the presence of a serine protease from Bacillus licheniformis (BLP) and calcium, at neutral pH. In our study, the pH range was extended from 7.5 to 4.0 to explore the possibility of formation of  $\alpha$ -lacalbumin nanotubes at decreased pH. Furthermore the influence of pH on the proteolysis of  $\alpha$ -lacalbumin, self-assembly of nanotubes and the resultant gel properties were investigated. The methods applied included TEM, SAXS, WAXS, LC-MS, CD, and light scattering. Our results show that the partially hydrolysed  $\alpha$ -lacalbumin was able to self-assemble into nanotubes at all pH values tested in the study. Transparent or semi-transparent gels or sediment was formed depending on protein and calcium concentration and the pH value. In general, the pH has no influence on the hydrolysis, but influences the rate of proteolysis and self-assembly, as well as the gelation time and gel strength.

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# Changes in viscoelastic properties as an effect of maltodextrin paselli SA2 addition on polysaccharides helix aggregation in sheared gel mixtures

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Gelling agents extracted from red seaweed can significantly increase the viscosity of aqueous solutions or form gels at low concentrations (0.5 - 2.0 %) they are therefore widely used in the food industry as a thickening, gelling and stabilizing agents. Their gelation process involves double helix formation followed by aggregation. This aggregation process is well understood for single hydrocolloid systems including the role of ions, sugars etc. [1, 2]. Investigations of  $\kappa$ -carrageenan sheared mixtures with pregelatinized cross linked waxy maize starch and maltodextrin Paselli SA24 revealed that the addition of a neutral carbohydrate can increase aggregation rate of charged helix-forming polysaccharide and impact on gel properties. However it is still not well understood how neutral carbohydrate can impact on charged hydrocolloid viscoelastic properties when produced under shear. This research focuses on changes in viscoelastic properties due to helix aggregation of differently charged polysaccharides ( $\iota$ -carrageenan,  $\iota$ -carrageenan, furcellaran and agarose) mixed with maltodextrin Paselli SA2 when produced under shear. We describe how manipulation of applied shear rate and cooling rate during network formation and neutral polysaccharide concentration influence viscoelasticity of final gel microstructure by investigation of complex modulus G\*, elastic modulus G', viscous modulus G'', and Phase angle delta and compare the results to single hydrocolloid systems [3, 4].

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#### Re-entrant isotropic-nematic phase behavior in polymer-depleted amyloid fibrils

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High-aspect-ratio amyloid fibrils based on whey protein display an isotropic-nematic (I-N) phase transition at critical concentration ( $\sim 0.4 \text{ wt\%}$ ), which has been explored by both experimental and theoretical approaches [1, 2]. The I+N coexistence, although theoretically predicted in rod-like colloidal system [3, 4], is still elusive in these systems, most probably due to the extremely high aspect ratio, combined with weak I-N interfacial tension and low enthalpy of transition. In this study, we report for the first time a depletion-induced closed-loop of I+N coexistence, induced by adding a non-absorbing polymer up and beyond the semi-dilute polymer concentration. At low polymer concentration, the biphasic coexistence was enlarged, similar as other rod-like colloidal systems such as filamentous fd viruses. Surprisingly, this separation phenomenon tends to weaken and eventually disappears when polymer concentration is far beyond the overlap concentration in the semi-dilute regime. To justify these results we calculate the total potential among fibrils by combining depletion potentials in the dilute and semi-dilute polymer regime with the DLVO theory, and rationalize the re-entrant behavior by following the evolution of the second virial coefficient via the extended law of corresponding states (ELCS) at increasing polymer concentration. We find that the decrease of the depletion potential range in the semi-dilute regime plays a pivotal role in the observed re-stabilization phase behavior, leading to a closed-loop of I+N coexistence region.

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#### Effect of fluid elasticity on swallowing

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We all eat food and drink liquids several times every day. The chewing, taste perception and swallowing are all highly unconscious processes rendering a complete culinary experience and a hopefully pleasant meal. The swallowing is seldom considered at all by healthy individuals whereas for many others it may cause discomfort and even serious health problems. Already over 50 years of age 22 % suffer from swallowing disorders, or dysphagia, due to factors such as degenerative diseases, side effects of medication and trauma. At 70 the same figure has reached 40 %. These persons must eat texture adjusted foods, and food viscoelasticity is key to facilitate easy and safe swallowing. The effect of elasticity on swallowing has been studied using model fluids with specific rheological properties, which were given to healthy individuals for sensory evaluation of the swallowing, and to patients suffering from dysphagia in a clinical trial. Three food grade model fluids with specific rheological properties were developed, a Newtonian fluid (constant shear viscosity), a Boger fluid (elastic, constant shear viscosity) and a shear thinning fluid (elastic, shear rate dependent). The model fluids were made opaque to x-rays to enable videoflouroscopic analysis during clinical trials. The elastic properties of the two latter fluids were determined through the extensional viscosity using Hyperbolic Contraction Flow. The results from the healthy sensory panels were combined with quantitative videoflouroscopy in the clinical trial (oral and pharyngeal transit time and the degree of misdirected swallowing). The study strongly indicated positive effects of fluid elasticity on the ease and safety of swallowing. For healthy individuals it was however difficult to distinguish the specific ease of swallowing since for this group it is an involuntary and well-functioning process. The patients suffering from dysphagia benefitted from fluid elasticity, but symptoms and discomforts were highly varying. Further development of the study of the swallowing process is therefore desired, especially for the varying swallowing disorders.

### Flow, diffusion and microstructure in capillary alginate gels

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Alginate is a renewable polysaccharide extracted from seaweed. It has been used for decades as a thickener and gelling agent in the food and pharmaceutical industries. Alginate is typically gelled via careful and slow introduction of di- or trivalent cations, often calcium. Via a specific gelation method, the external gelation method, gel structures interspersed with large channels, of around 100 micron in diameter, are created. Those structures are of interest as template for sintering ceramics and in the field of tissue engineering [1]. In the present study it is used as a model system in which the uptake and influx of macromolecules, e.g. nutrients or active substances, from the channel into the gel matrix is studied. The influence of flow on diffusion [2] was quantified by confocal laser scanning microscopy in combination with fluorescence recovery after photobleaching (FRAP) and raster image correlation spectroscopy (RICS). More specifically we investigated the system at the capillary/gel interface when an external flow of a solution of macromolecules in the capillaries is present. Layered structures at the channel interface, identified by transmission electron microscopy (TEM), are found to correlate with a decreased rate of transport of molecules from the liquid filled channel into the gel structure. Additionally a higher rate of transport of molecules from the channel into the gel was found for increasing flow rates. Thus, the flow within the channel influences the apparent rate of diffusion and loading of molecules in the gel bulk material. This study yields insights into the mass transport across the channel/gel interface at high resolution and highlights the versatility of renewable biomaterials to create complex structures.

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#### Mechanistic insights into self-assembly and gelation in biopolymers, proteins, and complex fluids through combined DLS-optical microrheology and raman spectroscopy

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Biopolymers and proteins play a critical role in providing both functional and sensory benefits in many processed foods. The self-assembly in these complex materials and the associated rheological response are engineered to provide stability, texture and functional benefits. The rheology evolution in such complex fluid/soft matter systems as a function of formulation parameters (e.g. pH, ionic strength) is intimately connected to corresponding changes in micro/mesostructure and intermolecular and intramolecular associations and interactions. Most insights into the self-assembly and rheology evolution process in such systems have primarily focused on elucidating the associated micro/mesostructural changes through various scattering (light, x-ray, neutron) and imaging techniques (cryo-TEM, SEM, AFM). Furthermore detailed insights into the associated chemical conformational/molecular structural changes and various non-covalent interactions (e.g. H-bonds, hydrophobic interactions) leading to the self-assembly process has been very limited. An understanding of the molecular level structural changes as self-assembly and gelation progresses will provide new mechanistic insights that will allow better optimization of performance controlling formulation design rules. The talk will illustrate the utility of the combination of mesoscale structure-property elucidation techniques such as DLS/optical microrheology with the high resolution chemical structure/conformation elucidation techniques such as Raman Spectroscopy in generating novel mechanistic insights that will enable improved performance engineering of complex fluids and soft matter systems for food applications. This will be exemplified through studies into the self-assembly/gelation mechanism in a thermo-reversible gel forming agarose and a widely utilized food protein- $\beta$ -lactoglobulin undergoing temperature induced aggregation and self-assembly.

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#### New strategies for tailoring the mechanical properties of ethylcellulose oleogels for food applications

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Over the past decade, the structuring of edible oils low in saturated fats through alternative means has been an active area of research. One of the more promising food grade oleogelators thus far identified is ethylcellulose (EC), a derivative of the naturally occurring polymer cellulose. Gelation is induced by heating the mixture above the glass transition of EC to disperse the polymer in the oil phase and subsequently cooling the melt. EC imparts structure by entrapping liquid oil within a three-dimensional polymer network supported by intermolecular hydrogen bonds. In the present investigation, we identified several novel strategies which can enhance the mechanical properties of EC oleogels. The manipulation of solvent polarity was investigated through the addition of castor oil or mineral oil to the oil phase, increasing or decreasing the solvent polarity, respectively. Gel strength positively correlated to the bulk polarity of the miscible solvents due to improved polymer-solvent interactions. This mechanism was also supported by rheological analysis and was successfully interpreted using Hansen solubility parameters. The effect of hydrogen bonding-capable surface active small molecules was also evaluated through the addition of oleic acid or oleyl alcohol. These molecules greatly enhanced the mechanical strength at very low concentrations (< 0.5 wt%), and produced up to a 10-fold increase in hardness, as determined by Texture Profile Analysis. DSC results demonstrated a shift in the melting behavior of stearic acid in the presence of EC, suggesting a direct interaction between these two species. Processing conditions were also used to increase the mechanical strength of EC oleogels. Gentle mixing during gel setting was found to nucleate structure formation, resulting in stronger gels than those cooled in bulk . Finally, reheating set EC oleogels to 80 - 100°C temporarily weakened the hydrogen bonds, thus facilitating a restructuring of the polymer network, yielding a significantly stronger gel after cooling. These newly identified strategies for enhancing EC oleogels will be advantageous when formulating fat mimetics for food products. Furthermore, the improved efficiency of EC as a gelator should allow for a reduction in the amount of structurant required to produce functional oleogels, which will directly translate to cost reduction.

### Acid-induced protein gels: From gelation to stress-induced failure

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Biomaterials such as protein or polysaccharide gels are known to behave qualitatively as soft solids and to rupture under an external load. Combining optical and ultrasonic imaging to shear rheology we show [1] that the failure scenario of an acid-induced sodium caseinate gel is reminiscent of brittle solids: after a primary creep regime characterized by a power-law behavior whose exponent is fully accounted for by linear viscoelasticity, fractures nucleate and grow logarithmically perpendicularly to shear, up to the sudden rupture of the gel. A single equation accounting for those two successive processes nicely captures the full rheological response. The failure time follows a decreasing power law with the applied shear stress, similar to the Basquin law of fatigue for solids. These results are in excellent agreement with recent fibre-bundle models that include damage accumulation on elastic fibres and exemplify protein gels as model, brittlelike soft solids. We investigate the robustness of this scenario with varying acidulent concentrations, paying special attention to the case of over-acidified gels previously overlooked in the literature. Qualitative differences are explained by microstructural changes below isoelectric pH that we monitor throughout gelation both by rheology and confocal microscopy. As a whole, our results highlight protein gels as a versatile and model system for the study of plasticity in soft amorphous materials.

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Wed, June 10, 2015, 11:20, F30

#### Carrageenan aggregation and gel transition

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The gel transition for three different types of carrageenan ( $\kappa$ ,  $\iota$ , and  $\lambda$ ) were investigated as a function of temperature and electrolyte concentration (KCI). Carrageenans are prone to forming helical and double helical aggregates, which lead to a significant change in viscosity and eventually gel formation. These aggregates are also a significant impediment for accurate determination of molecular weight by conventional SEC methods. This complication affects understanding of most aspects of the gel transition and appropriate interpretation of material properties. We followed the transition using dynamic rheology, optical rotation, micro-calorimetry, thermal conductivity and investigated structure under the various conditions using X-ray (SAXS, WAXS) and multi-detector SEC. Local interactions are probed by local techniques, such as optical rotation and SAXS, and more global techniques probe longer range effects of gelation, microscopy and rheology.

### Anomalous stiffening and ion-induced coil-helix transition of carrageenan in monovalent salt conditions

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The macromolecular conformations of anionic polysaccharides with decreasing linear charge densities – lambda, iota, and kappa carrageenan–, in varying NaCl concentrations, are studied by single-chain statistical analysis on high-resolution atomic force microscopy (AFM) images. Lambda remains in the random coil conformation, whereas iota and kappa undergo ion-induced coil-helix transitions, with two- to three-fold increase in chain rigidity. At low ionic strengths, I, the polymer chains sequester  $Na^+$ , leading to a greater flexibility, and beyond a critical I, to the formation of an intramolecular single helix. The persistence length exhibits a sublinear dependence on the Debye screening length deviating from the classical polyelectrolyte behavior expressed by Odijk-Skolnick-Fixman (OSF) or Barrat-Joanny (BJ) models. Above a certain I, the  $L_p$  shows an upturn, resulting in polymer stiffening and a non-monotonic behavior. This phenomenon is inferred from specific ion-polymer interactions and/or nonlinear electrostatic physics involving ion-ion correlations.

#### Alteration of the textural properties of inulin gels

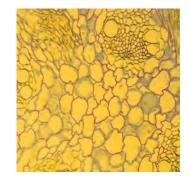
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Inulin is a well-known polysaccharide which can be easily extracted from several economically important plants, such as chicory, onion, garlic and wheat. Inulin is a dietary fibre and exhibits low calorie content, prebiotic properties and is suitable for diabetic nutrition. Inulin/water suspensions can form gels with a fat like structure. Depending on the inulin molecule's degree of polymerization (DP), inulin is commercially used as sugar replacement (low DP), texturizer (medium DP) and as fat replacement (high DP). In this work, two types of long chain inulin are examined in terms of their textural properties, depending on the applied shear rates and temperature during sample preparation. The application of a temperature profile reaching from 25 to 80°C during sample preparation, with constant stirring at 600 rpm, yields viscoelastic gels with increasing hardness for one type of inulin and decreasing hardness for the other type of inulin. By application of high shear rates (7000 rpm) at the same temperature conditions, the development of gel hardness was completely altered. Thus, the results reveal a high sensitivity of the textural properties of inulin gels to the variation of applied temperature and shearing during sample preparation. Those findings allow the controlled alteration of textural properties in food products containing inulin.









### Relaxation dynamics, glass transitions, and microstructure of hydrated gluten networks

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Thermal, mechanical and microstructural properties of frozen hydrated gluten were studied by differential scanning calorimetry (DSC, MDSC), rheometry (stress relaxation), imaging (cryo-SEM, TEM, CLSM) and theoretical modeling. This work was undertaken to investigate the thermal and mechanical relaxations observed in hydrated gluten and relate them to its microstructure. The Defay-Prigogine theory for the depression of melting point of fluids confined to capillaries was used to explain the calorimetric results and provide in-depth understanding of gluten microstructure. Results of the present investigation showed that is impossible to assign a specific glass transition value for hydrated frozen gluten because of the wide temperature range over which the gluten matrix vitrifies, and therefore the construction of state diagrams is not feasible at subzero temperatures for this material. Stress relaxation was followed over a wide temperature range (0 - 70°C). Temperature played a significant role in relaxation, allowing the construction of master curves and calculation of shift factors. Numerical calculation of stress relaxation spectra using Tikhonov regularization and the L-curve criterion identified three relaxation regimes. Stress relaxation is independent of compositional differences, although confocal microscopy showed the influence of protein composition on the morphology of the networks. Utilization of theoretical frameworks from macromolecular dynamics coupled by imaging techniques allowed in-depth understanding of the rheological properties of gluten with the aim to improve its industrial performance.

#### Assessing the handleability of bread dough

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Within the baking industry, the ability to produce dough that can be processed through large mechanized plant bakeries is of commercial importance. Dough that, once mixed, cannot be processed represents a loss of both time and product. There is, therefore, a need to assess whether dough can be processed in advance of production. The primary method for assessing dough properties is by baker's assessment of the feel of dough, assessing properties such as softness and stickiness. It is desirable for dough to be soft but not sticky and so these attributes are scored separately by the baker. There is a desire to move away from subjective assessments like these towards more quantitative instrumental techniques. A compression test has been developed in an attempt to measure these dough properties instrumentally. A wide range of non-yeasted doughs with varying softness and stickiness levels were prepared by varying flour type, mixing time and functional ingredients. Dough samples were compressed to specified strains and various measurement parameters compared with baker's assessments of softness and stickiness. Significant correlations were seen for compression energy per unit area with both properties, with softness showing the strongest correlation. One of the stages in dough processing where sticky dough causes particular problems is in sheeting where the dough passes between rollers. A quantitative dough handling assessment has been developed using a roller system and multiple passes of dough pieces. This quantitative assessment was compared with compression test results and showed significant and strong correlations. These results suggest that, with further development, this compression test could provide a limiting value below which dough could be expected to be difficult to process.

### Rheology of maize, wheat, teff, and cassava starches with stearic acid and xanthan gum

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Chemical modification is often used to improve starch tolerance to processing conditions. Consumer concerns to synthetic chemicals have led to strong preference for 'clean' label starches. Lipid and hydrocolloids are food friendly chemicals. This study determines the effects of stearic acid and xanthan gum alone and in combination on the rheology of maize, wheat, teff and cassava starches. An increase in viscosity was observed for all the starches with stearic acid and xanthan gum alone compared to the controls with cassava having the least increase. A further increase in viscosity was observed for the cereal starches with combination of stearic acid and xanthan gum. Stearic acid alone and with xanthan gum reduced retrogradation, resulting in soft textured pastes/gel. Combination of stearic acid and xanthan gum reduced the formation of type IIb amylose-lipid complexes, syneresis and hysteresis in cereal starches. Combination of stearic acid and xanthan gum produces higher viscosity and non-gelling starches with increased physical stability to freezing and better structural recovery after shear.

#### Starchy foam tailored with cellulose derivatives and transformed in non-reactive sponge cake after baking

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The sponge cake is a cereal soft product formed of a typical cellular structure with a relatively simple composition (flour, eggs, sucrose). It is a good candidate for the study of reactions that take place during cooking and affecting the nutritional and sensory quality of the final product. In order to be able to decompose the different reaction pathways (Maillard reaction, caramelization) it is necessary to focus on the specific role of the formulation (composition and process which includes foaming and baking). The originality of our approach is based on the development of a model product mimetic of a real sponge cake with similar cellular structure, but non-reactive regarding Maillard reaction or caramelization. Reactive ingredients (egg, sucrose, and flour proteins) have been replaced by non-reactive components that can provide equivalent functional properties: surfactant, thickening and gelling with increasing temperature. Hydrocolloids (cellulose derivative MC and HPMC) were selected as good candidates, to be added to starch and water for producing a solid foam. It was assumed that hydrocolloids of small molecular weight would present a better surfactant capacity due to their higher diffusion speed. On the other hand, hydrocolloids of high molecular weight would have a better viscosity capacity. We developed an approach for rational formulation, based on the reproduction of features of the suppressed ingredients. Viscosity of the liquid paste measured at high shear-rate was adjusted to 1.36 Pa.s, as in the reference dough, and the foaming capacity was controlled by measuring the volume fraction of included air. The cellular structure formed after baking was characterized by image analysis. Results showed that a mix of MC and HPMC made it possible to develop a cellular structure (size and homogeneity of cells) mimetic of that one of a sponge cake after baking. Moreover the model product was totally white and non-odorant, suggesting that no caramelization or Maillard reaction occurred during the thermal process.

### The porous structure of dough characterised using in situ Synchrotron X-ray computer tomography

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The porous structure of the dough as the result of air cell expansion during fermentation and bread baking is an important parameter in relation to the final textural properties of baked foods. The structural development of dough during bread-making is influenced by the protein content of the flour and the co-solute such as neutral salt (e.g. NaCl). X-ray computer tomography (CT), a nondestructive technique has been used to analyze the physical structure and porosity of bread, mostly focused only on the structure of either dough at the proofing temperature ( $< 37^{\circ}$ C) or products after baking. Due to a rapid change in dough structure during baking, the conventional X-ray CT method could not capture the transformation of dough to bread as a function of time. Accordingly, the high-speed synchrotron X-ray CT was used in this study to capture the kinetic evolution of the dough structure from dough fermentation (37°C) to baking at 100 to 125°C. The effect of different ionic and cationic types of salt on dough properties and how those properties are related to the dynamic structural changes in dough during fermentation and baking was also investigated. A high protein (13.6 %) wheat flour and a low protein (9.6 %) wheat flour were used to study the effect of inherent gluten proteins on dough structure at different levels and types of salts (no salt, 2% NaCl, 2% KCl and 2% NaBr). Results showed that high protein flour produced a higher loaf volume compared to low protein flour regardless of salt additions. KCl had an adverse effect on the dough resulting in low porous structural development. This dynamic study provided in situ characterization of how dough is structurally transformed into bread and how different flours and salts affect the cellular structure of dough and bread. This information could be used as a guide to select flour for suitable end-use or to control the quality of baked products.

#### Instrumentally determined sensory moistness in yeast-raised toast bread

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Sensory evaluated moist (opposite dry) crumb is an important quality parameter of yeast-raised toast bread. Toast bread will, due to staling, get firmer and also drier over time. Moistness is a sensory attribute not easily measured by anything but human senses. In this work the ability of instrumentally measured dough rheology (farinograph, Kieffer), heating/cooling dough rheology (rapid visco analyzer, mixolab, rheometer oscillation throughout heating/cooling), and final bread (sensory analysis, Texture Profile Analysis, TPA, Nuclear Magnetic Resonance, NMR, Differential Scanning Calorimetry, DSC, and bread penetration) characterization was done on yeast-raised toast bread added: emulsifiers, enzymes, and hydrocolloids, to predict sensory moistness attributes for toast bread. In the sensory analysis the same ingredients in 2 and 9 days old breads were sensorically evaluated on the same day. Quantified baking structures in yeast-raised toast bread are derived and characterized from a DOE controlled multiple stage study covering the baking process methodologies and a representative ingredient range. The (causal) baking process from dough throughout mixing, baking and of the final bread is fully covered by multiple methodologies generating a comprehensive map of quantifiable specifics which are used to discriminate between added bread improving ingredients. Splitting information is partially available for methodologies with respect to shelf-life. Dough is described using one variable, baking simulation by one variable, and final bread functionality by ten variables: four variables to describe day 2 information, two variables to describe day 9 information and another four variables to describe the difference in information between day 2 and day 9. Day 2 information most correlated with the sensory attributes evaluated by hand/fingers and in mouth (0.99) originated from the following analyses: TPA day 2 (0.89), and NMR day 2 (0.76). Day 9 information most correlated with sensory attributes evaluated by hand/fingers and in mouth (0.99) originated from the following analyses: TPA day 9 (0.99), DSC day 9 (0.99), NMR day 9 (0.97), and bread penetration day 9 (0.92).

### Effect of jet milling on whole wheat flour quality and bread digestibility

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Nowadays, nutritional habits lead to an increased whole grain consumption. Studies show that the consumption of whole grains protects against CVD, type 2 diabetes, some cancers, and improves gut health. Thus, for health reasons, a significantly higher daily consumption of whole wheat flour is desired. In recent years the use of micro-technology in food research has attracted much attention. Jet milling is an innovative technique of micronization, used for producing ultrafine powders with enhanced functional properties, finding applications in the chemical, pharmaceutical and mineral industry. The effect of different jet milling conditions on whole wheat flour functionality and the potential of those flours for bread making with the goal of improving bread quality and digestibility was investigated. Four different conditions of jet milling regarding air pressure, feed rate and recirculation were used. Scanning electron microscopy (SEM) revealed microstructure changes due to the reduction of particle size in jet milled flour with greater effect when greater intensity (high pressure, lower feeding rate and/or recirculation) was applied. As the severity of milling was enhanced protein and ash did not change, fiber content increased and starch hydrolysis rate also augmented. Mixolab data indicated that water absorption increased, while pasting temperature showed a tendency to decrease owing the milling intensity. Referring to bread jet milled flour addition reduced the specific volume, luminosity and moisture content, whereas an increase in the crumb hardness was noticed. However, these breads presented a slight decrease in estimated glycaemic index (eGI) compared to control samples suggesting that jet milling treatment could also have nutritional benefits.

#### Unraveling the contributions of starch, gluten, water content and mixing time to the rheology of wheat dough

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In a large part of the world, bread is a major constituent of the daily diet. It is obtained after baking its pre-product dough, which consists of water and flour to which small quantities of salt, sugar, yeast and enzymes are added. These ingredients are first mixed and subsequently fermented to result in a leavened piece of dough, which is then baked. The dough matrix consists of a hydrated gluten network that is intertwined with starch granules. During bread making, dough is subjected to a wide range of deformations, including both shear and extension. Hence, the rheology of dough plays a pivotal role in the bread-making process. Although bread making has been practiced for millennia, fundamental understanding of the relations between dough microstructure and rheology is surprisingly limited. In this study, first of all, protocols are developed for the systematic characterization of the rheology of unfermented dough. Linear and nonlinear viscoelastic behavior in shear is investigated by, respectively, oscillatory and creep-recovery experiments. Dough behavior in uniaxial extension is explored by means of an extensional viscosity fixture on a rotational rheometer, while for biaxial extension compression tests are implemented. Dough shows a typical gel-like behavior with a small critical strain and exhibits substantial strain hardening both in uniaxial and biaxial extension. Subsequently, the sensitivity of the different techniques to variations in dough microstructure is explored systematically. For this purpose, the contribution of each of the major constituents (gluten and starch) to the overall dough behavior is elucidated by examining the rheological behavior of both components individually, and by studying mixtures of different gluten-to-starch ratios as well as doughs made from weak versus strong flour. While the nonlinear dough behavior is primarily determined by the response of the gluten network, the linear behavior is strongly affected by the starch granules. We show that only nonlinear rheological tests are able to distinguish strong from weak flour doughs, confirming and extending the findings of previous literature. In addition, the effects of water content and mixing time (which are both important process parameters in bread making) are investigated. Small changes in water content alter the dynamic moduli substantially for dough as well as for each of the major constituents. The mixing time, on the other hand, appears to affect only the gluten network. Finally, visualization of the dough microstructure after application of shear or extension by means of confocal microscopy allows to qualitatively link dough microstructure and rheological properties.

#### Evolution of adhesion between bread dough and a solid surface during baking: effect on rheological properties and physico-chemical properties of dough

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During baking in pan, interactions occur between pan and bread that may increase the force needed to withdraw the product at the end baking time. It is known that interactions involved between a viscoelastic product and a surface depend on the rheological characteristics of this product [1, 2] as well as the properties of the surface [2]. Being established that wheat flour dough rheological properties change during baking [3], the aim of this study was to evaluate and understand the evolution of bread dough adherence during heating taking into account also the formulation of the dough. Bread dough adherence measurements during heating were carried out using an experimental device which allow to control the temperature and time of contact, the pressure applied on the dough by the surface fixed on a mobile (MCR, Anton Paar, Austria). Afterwards, the force versus distance is measured during the pull off of the mobile plate from the bread dough. The temperature of heating were chosen to refer to different time of baking. The viscoelastic properties of the product after different heat treatments were measured as well as some physico-chemical properties such as water content. These adherence measurements, carried out after heat treatment of the dough, showed a relation between adherence energy and dough rheological behavior as well as physico-chemical properties. An increase of temperature led to a decrease of adhesion. This loss of adhesive properties was attributed to a change of the structure of the product. Moreover the formulation of the dough, by adding fibers or changing the nature of fat influenced the level of adhesion. For raw dough it increased considerably in the presence of fiber whereas the viscoelastic properties did not change. It may evidence that rheological properties may not be the lone parameter that should be taken into consideration for adhesion phenomena during baking.

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#### A mechanistic model describing the growth dynamics of bubbles in viscoelastic dough during proving

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Proving of bread dough is an important step within the whole bread making process since its cellular structure is created. After the kneading, small gas bubbles along with  $CO^2$  producing yeast cells are distributed within the dough. The bubbles then grow during proving and eventually coalesce. Diffusion of  $CO^2$  from the dough to the bubbles drives the growth whereas the rheological and interfacial tension acts as a resistance. Predictive models describing the growth dynamics with respect to the material properties are therefore of great interest. The bubble growth dynamics during the first stage of proving (where bubbles grow freely without coalescence) is studied by adopting and modifying the, so called, cell model which is usually used to describe foaming in the chemical industry. In this model, the mass and momentum transfer equations between the bubble and the surrounding cell containing viscoelastic dough with dissolved  $CO^2$  are solved. A Monod type kinetic is used to describe the  $CO^2$  produced by the yeast and a nonlinear viscoelastic model featuring strain hardening is used to describe the rheological properties of the dough. The resulting set of partial and ordinary differential equations is solved with the software COMSOL. Experimental data from literature are used to validate the model. Due to the lack of data describing the material properties, a profound fitting procedure is applied. The agreement between predictions of the simulation and experimental data is good. In particular, effects of changes in the recipe and therefore in the material properties predict changes in the bubble growth dynamics adequately. A parameter study shows that the available amount of  $CO^2$  has the largest influence on the growth dynamics. An increase of the interfacial tension as well as the rheological parameters (such as relaxation time or rigidity module) slows down the bubble growth, as expected. Furthermore, we show that a more pronounced strain hardening leads to a deceleration of the growth process.

### Identification of gluten-free dough properties relevant for aeration and foam stabilization

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Gluten is mainly responsible for the stabilization of gas cells in wheat dough through the formation of a three-dimensional network, resulting in a stable sponge-like crumb structure. Thus, for the production of gluten-free bread, functional additives and novel processing approaches are required to compensate the functions of gluten. One strategy is to increase the incorporation of gas, preferably in the form of homogeneous small bubbles, by adapting mechanical and biological aeration methods. However, the success of this technical approach strongly depends on the foam stabilizing properties of the dough medium. Currently, the huge variety of recipes used by industries and researchers makes it difficult to compare and generalize obtained results. The aim of the presents study was to identify gluten-free dough properties which are required for sufficient gas stabilization in dough throughout mixing proofing and baking. In particular, surface activity and rheology were monitored since they determine the extent of surface aeration, buoyancy, coalescence, disproportionation and drainage.  $\mu$ CT allows for a correlation of the former parameters with the bubble distribution and time-stability in dough. Most of the typically used empirical methods for describing wheat dough rheology are not applicable without a gluten-network. A fundamental measuring program was developed to simulate the temperature and shear conditions which are prevailing during dough processing and baking in a rheometer. This made it possible to evaluate dough viscosity and shear-thinning behavior during mixing (shear rate: 700/s), followed by viscosity and structure regeneration during fermentation (low shearing: 0.05/s) and finally to monitor the viscosity increase through starch gelatinization (temperature ramp:  $4^{\circ}C/min$  until  $95^{\circ}C$ ) in a single measurement. The data obtained by tensiometer and rheometer measurements were correlated with the gas volume fraction of dough and bread. Consequently, different flours and hydrocolloid (HPMC) concentrations resulted in considerable differences regarding the gas volume fraction of dough (5 - 25 %) and bread density (0.42 - 0.21 g/ml) with the best volume and pore structure obtained with quinoa white flour (without bran) and 2 % HPMC. Higher power-law indices resulted in improved gas entrapment during mixing. The surface tension played a minor role in comparison to the flow behavior, revealing the power-law index ( $\sim 0.4$ ) as well as the viscosity after mixing as critical for aeration and gas stabilization.

### Structural and rheological characterization of laminating shortenings

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Laminating shortenings are fats specially tailored to prevent pastry dough layers from fusing together during the folding and rolling steps. A major problem associated with these products is their high content on unhealthy structuring fats: saturated (SFA) and trans fatty acids (TFA) (up to 66% w/w altogether). We studied different commercial laminating shortenings, and compared them to a multipurpose shortening, in order to identify key parameters underlying their macroscopic rheological properties. We characterized the solid structure using X-ray scattering in the wide angle (WAXS), small angle (SAXS) and ultra small angle (USAXS) regions. We used small and large deformation rheology to obtain: (1) parameters from creep and recovery and (2) viscoelastic moduli in the linear and non-linear regions. X-ray scattering reveals differences only in the USAXS region. All shortenings have similar polymorphic forms as seen in the WAXS region, while the domain sizes are in the range of 300 - 400  $\AA$  as determined in the SAXS region. USAXS results indicate that the aggregation of primary crystalline nanoparticles (CNPs) for laminating shortenings is either via diffusion limited-cluster aggregation or reaction limited cluster aggregation, while for the multipurpose shortening, the CNPs remain un-aggregated. Creep-recovery parameters show lower retarded compliance values and higher zero-shear viscosity for laminating shortenings when compared to multipurpose shortening. The viscoelastic moduli (G' and G': 1 - 3 MPa) and yield stresses (350 - 750 Pa) in the linear region remain unremarkable. Preliminary Lissajous curves (stress versus strain) in the nonlinear region at a frequency  $\omega = 6.28$  rad/s, suggest a less marked strain-stiffening behavior and higher viscous dissipation in laminating shortenings compared to the multipurpose one. To the best of our knowledge, this is the first time that non-linear dynamic rheological information is reported on edible fat systems. This information together with the USAXS observations provides novel insight on the structural and rheological signatures of laminating fats, and opens up the possibility for the design of healthier shortenings.

### Rheological optimization of durum wheat dough mixing by uniaxial extensional properties

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Dry pasta, a traditional cereal-based food product, recently increases its popularity in worldwide because of its accessibility, palatability, and high nutritional quality. Pasta process consists of different stages, but the first one, the mixing stage, is probably the most important. Generally, the durum wheat semolina is mixed with water in a paddle mixer, fed into an extruder, then compressed and extruded through a die. The rheological behavior of dough after extrusion is related to the development of the gluten network. In fact, the rheological properties of the final system reflect the degree and type of cross-linking of the polymers. With the aim of obtaining the optimal dough development, operating parameters, like mixing time and temperature, water amount, have to be optimized in order to produce high quality dough. In this work the extensional and shear properties of different durum wheat dough, for pasta production, were investigated aiming at optimizing the mixing time and temperature as a function of the semolina protein content by using a rheological criterion. Small amplitude shear oscillations were used to characterize dough in linear conditions, whereas uniaxial extensional measurements were carried out for non linear investigation. Dough was prepared at different temperatures and by using semolina with different protein content; samples were tested at different mixing time to evaluate the effects of both parameters on rheological properties. Strain hardening phenomena, similar to those observed for wheat flour, were evidenced during extensional tests and it was found that they are significantly affected by investigated operating conditions (i.e. time and temperature of mixing) and by the gluten content. Obtained results confirm that the rheological "optimum" of durum dough network is mainly governed by the gluten fraction and that extensional tests seem to be an interesting tool to optimize operating conditions.

#### Structural impact of butter in puff pastry

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In France, the majority of butter production is used in industrial bakery and biscuit pastry production. The largest volumes are consumed to produce puff pastry where the butter moiety is up to 30% of the product. Butter as a functional ingredient, must meet specific texture and hence structural requirements. As of today measurement tools such as texture analysis is sufficient for sorting butters to allow their use on a production lines, but this is only one aspect of functionality that is taken into account. The purpose of this study is to understand the functionality of butter through assessment of the crystalline stat combined with the SFC content measured in standard conditions supplemented by optical microscopy and differential scanning calorimetry. The combination of analytical techniques used in this study together with the French butter making industry to date allows not only to understand the density of crystal network installed in the butter but also to determine a profile of crystal sizes crystal forms as well as SFC. Previous studies have shown the relationship between the crystal size and the ability of butter to perform as a functional ingredient in puff pastry. We were able to identify critical factors for the fabrication of puff pastry in standard conditions to establish the structure functionality relationship between the butter and its performance in puff pastry.







# Influence of Processing on Structure and Rheology

#### Measurement of sol-gel-transition of skim milk and microfiltrated skim milk retentates by means of oscillation rheology and dynamic light scattering

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In fermented milk products manufactured from ultrafiltrated skim milk retentate e.g. Greek style yoghurt or fresh cheese, a high mineral content, in particular calcium ions, are often held responsible for bitterness [1]. Under conditions of ambient temperature and native pH of milk, approximately 66 % of the milk calcium is bound to the casein micelles [2]. In membrane filtration processes such as microfiltration (nominal pore size 0.1  $\mu$ m), presented as an energy efficient way to manufacture milk retentates, the casein fraction is retained so that micellar calcium is enriched in the retentate and is subsequently in the final product. The calcium ions can be washed out of the micelles by reducing the pH-value or temperature. This can be applied to lower the amount of calcium in the retentate via membrane fractionation. It's well known fact that under appropriate temperature and pH conditions, milk proteins change from sol to the gel state and a gel layer is gradually built up on the membrane surface, rendering the filtration process inefficient. The sol-gel-transition of different heat treated skim milk (protein content = 3.4 %) and microfiltrated (nominal pore size = 0.1  $\mu$ m) skim milk retentates (protein content = 8.1 to 12.0 %) between pH 4.6 and 6.8 at temperatures from 2 to 65°C was determined by means of oscillation rheology combined with dynamic light scattering. It was shown that the sol-gel-transition temperature of raw skim milk can be increased by heating to a temperature of  $95^{\circ}$ C (holding time = 256 s) from  $17.2 \pm 1.3^{\circ}$ C to  $36.0 \pm 5.4^{\circ}$ C. Further results will be presented as an evaluation of inefficient pH-temperature combinations for manufacturing skim milk retentates with a defined calcium content in an energy saving way by microfiltration.

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### Rheological properties of novel whey protein emulsion fluid gels

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In recent years the food industry has moved towards healthier and more functional food products. In response, shear gels in particular have been proposed as promising fat replacers: simulating the organoleptic properties of fat. Shear gels are formed through shear separation of a polymer undergoing the sol-gel transition. Currently much of the research investigates the preparation and resulting structural properties of polysaccharide fluid gels, however newer studies have concentrated on protein as the gelling material. The research presented here employs shear gel technology to encapsulate an oil droplet core within a whey protein (WPI) gel shell to form an emulsion fluid gel with close to 99% entrapment. The application of a shear field results in discrete micro-sized spherical particles, which, when in close proximity interact to form structures with physico-mechanical properties lying between strong and weak gels. Data obtained infers that the inclusion of oil in to a fluid gel matrix increases the effective phase volume of the particles to the extent that values above the critical phase volume are reached; in turn an interconnected structure is formed and elastic behavior observed. Within this region the storage modulus becomes independent to the oil content suggesting a system of soft deformable particles that can exceed the theoretical maximum packing fraction for monodisperse hard spheres. As a result these particulate suspensions show marked shear thinning behavior with apparent yield stresses. Additionally, once flow has been induced through shearing of the weak network, structural recovery of the system was observed over time. Furthermore the incorporation of an oil core gives the potential for new structures capable of transporting lipophilic compounds to the GI tract. These novel systems therefore present interesting rheological properties resulting from the formation of a multi-functional composite, that is not only high in nutritional value, but give the means to both structure fluids and deliver bioactive compounds to the body.

### Shake it but don't break it: Structure formation under oscillatory shear

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The influence of thermal gradients and an applied oscillatory shear field on the structure of selfassembled fibrillar networks of 12-hydroxystearic acid (HSA) in canola oil were investigated using a rheo-optical stage attached to a light microscope. This structure was greatly influenced by both the cooling rate applied to crystallize the organogel as well as by the oscillatory shear applied during solidification. Indeed, the application of oscillatory shear is shown here to create structures, in contrast to the complete destruction of structure under continuous shear. Crystallization of HSA from the melt at slow cooling rates (1 °C/min) was shown to yield fibrillar crystal morphologies. This is not unexpected given the reports in the literature of the fibrillar nature of 12-HSA organogels. In contrast, crystallization at fast cooling rates (30  $^{\circ}C/min$ ) was shown to result in a spherulitic morphology. This is also not unexpected. 12-HSA is known to undergo crystallographic mismatch branching, a phenomenon whereby a new crystalline fiber domain nucleates on the surface of an existing fiber. This causes the daughter strand to appear to "branch" off the mother strand. Extensive crystallographic mismatch branching is present in highly supersaturated systems, such as a fast-cooled organogel. The resulting microstructure of HSA is highly dependent on the cooling regime used to set the organogel. The application of an oscillatory shear to an organogel melt being cooled slowly results in the formation of thicker fibers. It has been shown using time-lapse micrographs that the thickening of these fibers can be attributable to the aggregation of individual 12-HSA fibers. The effect appears to be dependent on the magnitude of the strain being applied to the solidifying organogel. At relatively low strains, the effect is barely noticeable but at progressively higher strains, the thickening effect is observed. It appears that no further thickening of the fiber is achieved once a certain strain is achieved. The increase in the thickness of the fiber can be explained by the area swept by the fiber as oscillatory shear is applied to it. The back and forth motion of the fiber due to the oscillatory shear will increase the collision rate with other fibers in its vicinity. Collision of these fibers will cause them to aggregate. The strain-dependence of this effect suggests that a large enough area (and thus enough crystallizable matter) must be swept by the fiber before any noticeable increases in the thickness can be observed. Varying the oscillatory frequency does not result in any noticeable change other than what was already observed. The application of an oscillatory shear on an organogel being solidified under a fast cooling regime appears to enhance the nucleation of the spherulitic structures. An increase in nucleation will not only increase the number of spherulites observed but will reduce the size of these spherulites. This is observed when an oscillatory shear is applied to an organogel solidified under a fast cooling regime. Increasing the strain of the oscillatory shear appears to increase the incidence of nucleation as evidenced by the smaller and more numerous spherulites. Increasing the frequency of the oscillation also appears to increase the incidence of nucleation. At sufficiently high frequencies of oscillation, the microstructural morphology appears to transition to the fibrillar morphology observed in organogels cooled under a slow cooling regime. This study demonstrates how external fields can be used to structure crystalline fibrillar networks of organogelling materials. The rheological consequences of this structuring will also be presented.

#### Effect of accumulated shear work on the rheology, structure, and melt functionality of a mozzarella-type cheese

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The mozzarella cheese process includes cooking and stretching steps that give rise to a fibrous network. The stretching step not only brings about desirable textural changes but also helps in uniformly distributing the fat-serum channels that are important for attaining optimum melt characteristics during pizza baking. The amount of shear work given to the cheese mass during working plays a significant role in creating the desired structure and functionality. The present study, therefore, aimed to study the effect of accumulated shear work on the rheology, structure and melt characteristics of mozzarella like cheese. Cheese samples with varied amounts of shear work input were prepared by working molten cheese mass at 70°C in a twin screw cooking-stretching vessel (Blentech) for various times using three screw speeds. Batch size for each experiment was 25 kg. The power law model fitted well to steady shear rheology data so consistency coefficient, K and flow behavior index, n were useful rheological parameters. Frequency sweeps and temperature sweeps were used to measure changes in viscoelastic behavior. Confocal laser scanning microscopy was used to explore differences in structure. Melt functionality was assessed using a modified Schreiber melt test. Rheology, structure and melt functionality were found to be strongly dependent on accumulated shear work when either screw speed or shearing time were increased. A non-linear increase in K and decrease in n were observed with increasing amounts of accumulated shear work, indicating work thickening behavior. A 10 fold increase in K was recorded with an increase in accumulated shear work from 8 to 80 kJ/kg. Excessively worked cheese samples exhibited poor melting. Excessive working (> 70 kJ/kg) led to breakdown of the fibrous structure of the cheese matrix and also caused syneresis. Fat globule size in the cheese decreased with increasing accumulated shear work. We concluded that the dominant contributor to the changes in properties with increased shear work was shear induced structural changes to the protein matrix. A good correlation was found between the steady shear rheological properties and the melting properties of test cheeses.

#### Process-induced structural changes of casein micelles and casein micro-particles

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Caseins are one of the major proteins in milk and have a high nutritional and technological value. Caseins are prone to self-assemble because they have structural features similar to diblockcopolymers. While hydrophobic blocks cross-link the caseins with themselves, phophoserin-rich patches interact with calcium-phosphate nanoparticles. Spherical casein micelles with a diameter of approximately 150 nm or recently produced casein micro-particles (10 - 30  $\mu$ m) [1] are examples of self-assembled, higher aggregated structures. The lack of secondary and tertiary structure confers high flexibility to the protein which might explain why supramolecular casein complexes behave like soft matter. This has consequences for a number of functional and processing properties. Casein micelles, for instance, deform in films or deposits when subjected to external forces [2 - 3]. During micro-filtration casein micelles undergo a transition from spheres to ellipsoids as a result of the filtration forces, which has an impact on the filtration performance. We have developed methodology to monitor and analyze structural changes of casein micelles in deposit layers during filtration. We will report in detail on filtration setups and simulation tools which we used for surface-sensitive X-ray scattering experiments. We observed a similar deformation in casein micro-particles in blend casein/pectin films. These particles could be further functionalized and used for many purposes. A promising approach is the use of caseins for nano- or microencapsulation of bioactive substances [4]. Besides solubility properties and stability of the matrix, structural aspects play a key role for the functional properties of the capsules. For the detection of micro-particles, X-ray scattering has limits in resolution and contrast. We demonstrate application of convocal Raman microscopy as an analytical tool to resolve changes in structure and composition.

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#### AFM studies on the impact of different grinding techniques on sucrose surfaces and resulting flow properties of chocolate model suspensions

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Refining is an important step in manufacturing of chocolate. In order to obtain a good mouthfeel and melting behavior of chocolate, a smooth mass without grittiness is required. This can be achieved by grinding the particles to a size of less than about 25  $\mu$ m. Despite chocolate texture, refining is also relevant for the processing behavior as e.g. flow properties of chocolate masses are highly affected [1]. It is well known that a larger proportion of small particles increases equilibrium viscosity and yield value [2]. Thus, particle size distribution after refining should not be too broad. In chocolate manufacturing, traditionally two different processes are used for grinding: roller refining and ball mill refining. In both cases, particles are ground in an environment of liquid fat so that newly generated surfaces are prevented from air and humidity maintaining their activated and amorphous character [3]. In addition, both processes result in a good mouthfeel and melting behavior of produced chocolate. However, processing behavior of chocolate masses refined by ball milling is not as good as it is obtained from roller milling. While it is possible to optimize the ball milling process in order to obtain similar particle size distributions compared to roller refining [2], there are still differences in taste and flow behavior which could not be explained so far. The results of our study show that these differences can be attributed to altered surface properties of particles. As sucrose is one of the main ingredients in chocolate, experiments were focused on sucrose particle surfaces. Therefore, model suspensions of sucrose in a continuous phase of liquid cocoa butter were prepared. Particles were ground using ball mill, roller refiner and impact mill. Then, surface properties of resulting sucrose particles were examined in detail. Therefore, an Atomic Force Microscope (AFM) was used to characterize surface topographies and local adhesiveness at micro- and nanometer scale [4]. The results were correlated to macroscopic flow properties and immobilized fat content. Furthermore, local thermal properties of surfaces were measured providing additional information on surface state with respect to its amorphous and crystalline character.

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#### A mechanistic approach to deliver functionality into complex food systems via extrusion processing

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Health and well-being are the major drivers for the increased interest in the creation of functional food products. For this purpose, functional food ingredients that provide health benefits beyond basic nutrition can be incorporated into food products. Key challenge is the delivery of the ingredients through the food product without sacrificing the appeal, convenience and sustainable character of the food and in a manner that they will indeed promote health and well-being of the consumers. Extrusion processing possesses several advantages due to its multifunctional nature combining several unit operations, which can be manipulated to provide desired product characteristics and functional properties, at the same time. Extrusion is a flexible, economic and sustainable process with no effluents, yet it possesses many challenges with respect to strong interactions between mass, energy and momentum transfer, coupled with complex physicochemical transformations, which govern thermo-mechanical properties. For instance, extruded biopolymers, such as plasticized starch or protein, are non-Newtonian fluids and their rheological behavior is very complex, depending on different parameters such as screw speed, temperature and moisture content. Hence, an empirical research approach based only on experimental design of extrusion trials generally fails to provide fundamental understanding and is not efficient in finding a rational compromise between the large numbers of system parameters influencing the microstructure and functionality of the food product. To deal with the complexity of extrusion and deliver the desired functionality into food products, we have developed a mechanistic approach based on the fractionation of the process into logical interrelated sections. These sections can then be investigated more thoroughly and conveniently using various experimental and numerical tools, including inline multiple-step slit-die rheometer, offline shearing device, computational fluid dynamics simulation and inline infrared sensors. In this contribution, we will present this approach and the corresponding tools developed to characterize the extrusion process. The application of the approach will be demonstrated with a case study on the delivery of functional ingredients into directly expanded starch-based foods.

#### Dependency of moisture barrier properties of fat blends on their fat crystal network structure and possibilities for influencing thereof

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Fat based barrier layers within products are applied to control moisture diffusion between layers which differ in their water activity. Investigations have been carried out for an improved understanding of barrier properties depending on fat crystal network structure (FNCS). For this purpose, mixtures of low and high melting fats (stearin and olein fractions of palm fat, palm kernel fat and milk fat) were characterized after solidification with respect to solid fat content (SFC), barrier properties and FCNS. Compositions of the fat blends were selected to obtain comparable solid fat contents, but they should differ in their fatty acid distribution. Furthermore, several additives, like emulsifiers and crystallization promotors, were added to modify crystallization and resulting FCNS. The method of detrended fluctuation analysis (DFA) was applied to quantify FCNS which had been visualized using confocal laser scanning microscopy (CLSM). The scaling exponent as a result of DFA enables a mathematical description of such structures [1]. Large differences were detected for moisture barrier properties of the fat blends investigated. Lowest SFC (32 %) resulted in low barrier effect of this blend. However, blends with SFC > 35 % can reduce the moisture migration very efficiently, but can also have only a minor effect. These findings confirm the influence of FCNS on this parameter. Interestingly, fat blends with a very smooth FCNS were not necessarily the best in barrier properties. In contrast, blends possessing a very heterogeneous FCNS with very dense crystals and large areas of interconnected liquid fat in the structure showed a high barrier effect with respect to moisture migration. A correlation could be established between the scaling exponent from DFA and the barrier properties of the solidified fat blends. Higher scaling exponents indicating more heterogenic structures lead to a better barrier effect and vice versa. Addition of emulsifiers or crystal promotors to the fat blends can contribute to an improvement of barrier effects especially in the case of blends, where the barrier effect of the pure blend was relatively low. Influence of additives corresponds with changes in the FCNS due to the addition of the substances as could be confirmed by CLSM and DFA. These results can be used to optimize fat blends with respect to their performance in barrier layers and define the FCNS which are required to obtain a good prevention of moisture transfer by fat-based barrier layers.

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## Rheological characterization of commercial thickening agents for dysphagia patients

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Dementing elderly have often problems with swallowing, so-called dysphagia. To overcome these problems, commercial thickeners are available on the market. The producers of these products claim to deliver easy and practical solutions for the staff of elderly houses. Drinks with rheological characteristics adapted to the swallowing problems could be made quickly and easily. In the present research, the influence on the rheological characteristics of three types of commercial thickening agents [Nutilis Powder (Nutricia); Resource Thicken Up Clear (Nestlé); Thick & Easy (Fresenius)] in different fluids, concentrations and conditions of use were evaluated from a practical perspective. Firstly, the rheological characteristics of the three thickening agents dissolved in water was studied. All the thickening agents were dissolved in the lowest concentration indicated in the product specifications, in order to obtain a fluid with nectar or syrup thickness. A broad range of rheological measurements were carried out to obtain a full characterization and comparison of the different thickeners. Although the overall rheological behavior of the products was similar (e.g. shear thinning behavior), there was a viscosity range of 1 log unit for the different products. Also the storage and loss moduli varied with the thickener type (up to 1 log unit). Secondly, the rheological characteristics of the concentration range indicated in the product specifications were evaluated in water. All concentrations showed similar rheological behavior, with an increasing shear stress as expected. However, one of the commercial thickeners delivered (much) higher viscosity values compared to the product specifications. In a third part, the influence of different fluids (milk, orange juice, coke and coffee), was examined. All fluids delivered a lower viscosity than water. The lowest viscosity was obtained with coke, probably due to its very low pH. In conclusion, different commercial thickening agents all show similar rheological behavior, but the thickness of the final product differed considerably as function of thickener type, the (variation in) concentrations and the solvent. These influence must be taken into account when optimizing the rheological characteristics with respect to the swallowing problems.

#### Application of NMR techniques for improved understanding of molecular and structural changes during ingredient processes

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Increasing awareness of functionality changes induced by manufacturing processes and, thereafter, ingredient interactions during formulation is demanding the application of advanced analytical techniques for molecular level characterization. Nuclear Magnetic Resonance (NMR), in its various configurations, offers the possibility to detect and identify target molecules in milk and dairy products by means of direct analysis. A combination of 1H NMR, 31P NMR and 31P magic angle spinning (MAS) NMR is currently being applied to study the rehydration of milk protein powders. 1H NMR is making it possible to identify soluble molecule release according as the milk protein powder is being re-suspended. 31P NMR and 31P MAS NMR are being combined to distinguish between the different phosphorus species, particularly phosphoserine residues present in micellar casein-based powders and solutions, in order to provide greater insights into the changes occurring in the structure of these proteins during concentration and rehydration process. An additional line of investigation involves a study of proton relaxation time to follow the dehydration and rehydration behavior of dried ingredients as well as cheese maturation. In mozzarella cheese, this technique is being studied to distinguish between different types of water, and differentiate it according to the extent of its interaction with protein, thus proving that it may be a complementary tool when studying the micro-structure of this complex matrix.

### Rheological properties of baby foods: Dependence on recipe and type of process

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Baby food production is increasing due to the increasing request of working mothers and to the handiness, cleanliness and safety of this kind of food. Many different properties determine the nutritional profile and the physico-chemical behavior of baby food. Among them, the rheological behavior is important both during the process (i.e. for the proper design of some components of the production plant) and in the final use, where consumer satisfaction is the final goal. Baby food can be dry or wet and is obtained by mixing different ingredients. Their formulation and the processing parameters determine a very rich rheological behavior. In this work, three formulas produced by Heinz Italia have been analyzed (beef, mixed fruit with cereals and cheese), all produced with a new aseptic process, recently introduced in baby food production. In the aseptic process, the heating exchange is managed directly on the nude food during the process, by means of continuous heat exchangers. In other words, in the aseptic process the sterilization of the food and the package take place separately. Conversely, in the classical process the baby food is packaged and then heated to a specific temperature for the appropriate time. The aseptic process reduces the thermal treatment time (being also energetically favorable) and should better preserve the macroscopic food characteristics. Detailed rheological experiments have been carried out on the three samples, both in linear and non-linear regimes. More specifically, linear viscoelastic properties and steady flow behavior have been analyzed. Within the same process (either aseptic or classical), the rheological properties depend upon the food recipe (i.e. macroscopic ingredients, if compared to literature data). Within the same sample a strong influence of the used process (aseptic or classical) on the final flow properties of the baby food has been found.

#### Microstructure and texture of stirred yoghurt made with different exopolysaccharide producing cultures and subjected to mechanical treatment after fermentation

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Different yoghurts were produced by use of four starter cultures varying in level of exopolysaccharide (EPS) produced. Each starter culture was constituted of one strain of Lactobacillus delbrueckii ssp. bulgaricus (LB) and one strain of Streptococcus thermophilus (ST). Two strains of LB (high/low in EPS production) and two strains of ST (high/negligible in EPS production) were combined pair-wise resulting in generation of four starter cultures. Yoghurts were produced at pilot scale and mechanically treated after fermentation by applying various back pressures (0, 1, 2, or 4 bar). The different starter cultures and applied mechanical treatment significantly influenced the characteristics of the yoghurts in their rheology, particle size distribution and water holding capacity as well as in microstructure. The type of EPS produced, the distribution of lactic acid bacteria as well as the applied strain combinations were found to play a key role. The ST strain used was the main reason for the observed differences, with the high EPS producing strain e.g. causing increased particle size and viscosity at high shear rate  $(1000 \ 1/s)$ . The confocal micrographs revealed that the particle size of the protein gel decreasing with increasing pressure and that the distribution pattern of EPS within the protein gel was very dependent on the strain combinations. The results thus show that EPS producing starter cultures differ in their ability to provide mechanical stability to stirred yoghurt and the present study can aid in selection of appropriate strain combinations and processing parameters for yoghurt production on industrial scale.

### Understanding structure formation in dense biopolymer mixtures

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Plant-derived products with pronounced fibrous structures are of interest as a replacement of meat products. For this research a high temperature shearing device is used for making fibrous structures with soy proteins and polysaccharides. Previous research showed that the combination of proteins and polysaccharides is needed to obtain fibrous structures. Soy protein concentrate (SPC) could be transformed into anisotropic structures with pronounced fibers under certain processing conditions and certain concentrations, while only homogeneous structures were obtained with soy protein isolate (SPI). Therefore the role of polysaccharides, mainly cellulose and pectin, as the most predominant polysaccharides present in SPC, was investigated. A combination of pectin and cellulose (ratio 1:1) was added to SPI while keeping the dry matter constant (45 wt%). Besides, a blend of soy with the separate polysaccharides was made. The morphology of the structures was analyzed with the SEM. The anisotropic index (AI) of a structure was used to quantify the fibers of a sample as measured with a texture analyser. The combination of pectin and cellulose in a ratio 1:1 resulted in all cases in anisotropic structures (Al 1.8 - 3.2). The optimum Al of 3.2 was found for the polysaccharides protein ratio of 1:6.5. A combination of pectin and soy protein isolate resulted in a fibrous structure that lacks coherence. While the combination of cellulose and pectin resulted in a macroscopically phase separated matrix without fibers. Therefore it can be concluded that both polysaccharides are important for fibrous structure formation.

#### Application of the thixotropic elasto-viscoplastic model as a structure probing technique in weak gel suspensions

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A new methodology for probing size characteristics using thixotropic elasto-viscoplastic models is described and its application is demonstrated using a weak gel suspensions. From these constitutive models a structural parameter is derived. The structural parameter is a scalar quantity that represents the structuring level of the suspension. We used the structural parameter as an indirect method for evaluating the structure and compared it to a direct method (i.e. particle characteristic size with a Mastersizer) of structure evaluation. The new approach was applied to acid milk gels, a weak food gel suspension, to investigate breakdown and build-up of the suspension aggregates through shear. The objective of both the direct and indirect method is to ultimately link the structural characteristic size with the fluid mechanics. This has been done by relating the structural parameter to measurable flow properties. The agreement between the predictions with the model and the measurements using a Mastersizer was very good, provided that samples were diluted and sheared in the Mastersizer. Results suggest that the thixotropic elasto-viscoplastic model has predictive capability in pertaining the structural state of the acid milk gels.







## Rheological Methods

#### Simulating food mastication by applying integrated 'compression–shear–decompression' cycles using a Kinexus rotational rheometer

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During mastication, food is subjected to a number of different external stresses applied by the combined action of the tongue, teeth, jaw and palate. The timescale over which these stresses are applied depend on how long the food is chewed for, while the temperature at which mastication takes place is dictated by whether the food is consumed hot or cold. The action of saliva initiates the breakdown of the food during mastication, a process associated with a change in rheological behavior. We have attempted to simulate this mastication process using a Kinexus rotational rheometer (Malvern Instruments) performing integrated compression--shear-decompression motions to mimic the motions of the tongue against the palate in the presence of saliva. To assess the validity of this approach a number of model sauces were characterized using this instrumental technique and the parameters derived from the analysis were correlated to those derived by sensory analysis. The optical properties (lightness) and microstructure (light scattering and microscopy) of the sauces were also measured. A good correlation ( $r^2 > 0.95$ ) between the "consistency" (maximum peak force) derived from the simulated mastication method and the "viscosity/thickness" derived from sensory analysis was found [1]. The microstructures of sauces masticated within a human mouth and within the simulated mastication instrument were also similar. Overall the results suggest that the simulated mastication method can be used to characterize the textural properties of semi-solid foods within the mouth.

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#### Analysis of fat crystallization thanks to microrheology

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The crystalline form of fats in chocolate, butter and vegetable oils was studied thanks to microrheology. Passive microrheology studies the mobility and displacement of micron sized particles [1]: we used Multi Speckle Diffusing Wave Spectroscopy (MS-DWS) coupled with a temperature ramp in order to probe the particle displacement to analyze the viscoelastic properties of an opaque product. Under heating or cooling conditions, particle movements can be related to the crystalline form of the fat: the rearrangements occurring during melting or during crystallization provide crucial data about the fat's polymorphic transitions. Crystalline form and melting temperature of fats are important data for the elaboration of new products or for quality control of finished products. In the case of chocolate, the microrheology analysis during melting can identify the crystalline form of finished chocolate products, and so help to predict its stability against blooming. Moreover, microrheology can be used to study the impact of formulation and process on melting temperatures of low-fat butters. In addition to the analyses of crystalline forms of fat, the MS-DWS provides data on viscoelastic property changes.

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#### The single phase shear rheology of molten instant coffee

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Solid instant coffee is typically formed through capital intense drying processes. Alternate forming methods include extrusion as a high temperature, high pressure melt that helps to induce an open porous structure which assists in the dissolution of the product. A knowledge of the melt rheology is required to be able to understand the extrusion process and control the structure of the product. The single phase melt rheology was tested primarily using the Cambridge Mulitpass Rheometer, which is a fully enclosed capillary rheometer capable of testing the same sample repeatedly under a range of conditions. The entry and exit losses from the capillary were estimated using ANSYS Polyflow to model the specific geometry used for a range of different generalized Newtonian fluid models. Molten samples of commercially available instant coffee were prepared in situ at 110°C and then cooled to the desired temperature(s) for each test, down to a practical minimum of 80°C. The back pressure on the capillary was varied from 20 to 60 bar. The accessible range of wall shear rates was 0.01 to 100 1/s, over which the melt showed shear thinning behavior with an approximate power law index of 0.8. The melt exhibits self-similarity over the different temperatures, whereby the viscosity as a function of shear rate is shifted but the trend is the same. Sample to sample variation was high, and can be attributed to plasticisation by differing water content. Back pressure has no effect on the viscosity of the melt. Hysteresis is observed when samples are subject to increasing and decreasing shear rates, indicating that the melt is thixotropic. This effect persists for a number of hours, and may be attributed to a microstructure present in the melt. A parallel plate rheometer was also used to test the shear and oscillatory rheology at atmospheric pressure. Disc shaped samples were prepared using a high aspect ratio pill press at 120°C, and then solidified by cooling. These samples also exhibit shear thinning behavior, but were found after testing to contain gas bubbles which were not present before testing. This leads to the conclusion that samples prepared from commercially available, porous instant coffee require pressurization for the pore gas to remain in solution.

### Spontaneous gelation of wheat gluten proteins in a food grade solvent

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Producing wheat gluten gels with tunable mechanical properties via simple sol-gel routes would facilitate their processing into plant protein-rich food products. However, standard gluten is a very elastic mass with a high concentration of proteins in water and is hardly processable except using high shear harsh extrusions. The wheat proteins are responsible for the viscoelastic properties of standard gluten and dough and are among the most complex proteins families, with extremely broad polymorphisms and polydispersities. They are moreover insoluble in water, rendering rational studies difficult. Thanks to a novel protocol for the gluten proteins extraction that we have recently developed, stable ethanolic suspensions of gluten proteins are obtained for a wide range of protein concentrations. In this talk, we will present the viscoelasticity of those suspensions and show that they exhibit a spontaneous and concentration-dependent gelation, which we find to be driven by the slow formation of hydrogen bonds. We successfully rationalize our data using percolation models and relate the viscoelasticity of the gels to their fractal dimension measured by scattering techniques. The novel gluten gels display self-healing properties and their elastic plateaus cover several decades, from 0.01 to 10000 Pa. In particular very soft gels as compared to standard hydrated gluten, suitable for processing, can be produced.

## Active laser tweezers microrheology of inhomogeneus mixtures

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Laser tweezers are well established tool for noncontact manipulation of micro and nano-particles and for measuring forces between them. They can also be applied for measuring rheological properties. Compared to macro rheometers laser tweezers based micro-rheometers enable measurement of local viscoelastic properties on a length scale of a few micrometers. This may be a significant advantage if the material under investigation is inhomogeneous. Here we present the results of laser tweezers based active micro-rheology study of inhomogeneous mixture of DNA and levan. Both polymers are important components of bacterial biofilm matrix. At low DNA concentrations, DNA-levan mixture is homogeneous. At a certain DNA concentration threshold levan starts aggregating into a few micron big clusters. Microrheology measurements revealed that the viscosity and elasticity within levan clusters increases, while the local viscosity of DNA regions decreases compared to homogeneous mixture. The results of macro- and microrheology measurements in inhomogeneous mixture differed significantly which demonstrates the importance of microrheological measurements for better understaning of viscoelastic properties of inhomogeneous materials.

#### Micro-rheology as a tool for the characterization of food

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In this presentation we explore the potential of microrheology based on diffusing wave spectroscopy (DWS) to access the rheological properties of food in research, production, and quality control. DWS is a modern light scattering technique that allows the quantitative measurement of microscopic motion in soft mater systems and its main application is DWS-microrheology. This technique has several potential advantages over mechanical rheology such as the contact-free nature and the fast and reproducible data acquisition. Moreover, because the sample is not mechanically deformed, many potential artifacts like non-linear behavior, shear banding, and thixotropy can be avoided. This significantly simplifies studies on aging and gelling behavior of shear sensitive products. We present applications of DWS-microrheology on emulsions, dairy products, and xanthan and gelatin solutions. In particular we demonstrate the ability to measure the elastic modulus G' and viscous modulus G" over a huge frequency range at acquisition times of about 3 minutes. Moreover, we show how the gel point of gelling samples can be determined with high reproducibility in a fully automated manner. In addition, DWS microrheology enables aging and stability studies to be performed over an unlimited period of time with only one sealed sample. Since DWS is contact free, one does not have to load the sample chamber with new sample material each time a measurement is conducted, as in the case of a mechanical rheometer measurement. Combined with the fast measurement time, this greatly simplifies screening and quality monitoring on a large number of samples over an extended time. We show some first results for stability studies conducted on mayonnaise.

#### Investigation of texture-structure-perception relationships of protein-enriched yogurts with different fat contents

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Consumer acceptance of popular products such as yogurts is highly dependent on their texture properties, which can be greatly impacted by protein and fat contents. The increased interest in more healthy products, such as low-fat yogurts, requires a better understanding of their structure and the consequences of the formulation changes. The objective of the present study was to investigate the relationships between composition, texture and sensory properties of protein-enriched yogurts with various fat contents. A combination of several techniques was carried out, in order to obtain a multi-scale characterization of the products, which could enlighten structure/texture/sensory perception relationships. First of all, particle size analysis and Confocal Laser Scanning Microscopy (CLSM) were performed for investigating particle size distribution, aggregation and organization of protein matrix and/or fat globules. Rheology and tribology were combined to better understand texture properties both at the bulk and the interface scales. Principal Component Analysis (PCA) was performed on instrumental data to discriminate the products. Finally, Quantitative Descriptive Analysis (QDA) was carried out with 12 trained judges evaluating 25 descriptors, covering visual and mouthfeel texture, but also taste and color. The combination of techniques appeared very necessary to describe the set of 12 products since friction coefficient (tribology) and viscosity (rheology) brought complementary information, as they were orthogonal on the instrumental Principal Component Analysis (PCA) map. Particle size and quantitative image analysis data obtained from CLSM drove the third main axis. The 12 yogurts were well represented on the F1-F2 and F2-F3 PCA maps, with 88% of information covered by instrumental data. Moreover, the sensory descriptors assessed by the trained panel were in very good accordance with analytical data, revealing that rheology is important for mouthfeel understanding, tribology is linked with fat-related perception (such as oily film, butter taste, and cream taste), while particle size and microstructure are linked to both visual and mouthfeel particle amount as assessed by the judges.

### Combining NMR relaxometry and rheology to predict structural properties of SPI-WG blends

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Understanding and describing microstructure in concentrated biopolymer blends is still a considerable challenge. Blend morphology is visualized by means of techniques such as CSLM and SEM mainly. Then, this morphology is often related to the mechanical properties of the biopolymer blends and gels as defined by rheology. Here, we propose a new method to investigate the rheological response and morphology of a complex, semi-solid structure using the rheological properties of the individual phases present in the material and the water distribution between the phases as assessed by NMR relaxometry. The outcomes are then combined with polymer blending laws to understand blend structure and the resulting rheological behavior. We have used a blend of soy protein isolate and vital wheat gluten (SPI-WG) with a concentration of 20 wt% in water. In this blend, the rheological behavior is mainly a result of the fact that SPI absorbs most water as shown by NMR relaxometry. The fact that SPI absorbs most water and subsequently forms the continuous phase explains why the blend has much lower storage modules than the SPI dispersion at the same concentration. WG acts as a rigid, inert filler in the blend due to the high WG-concentration in this phase. The increased storage modules after heating can be explained by the fact that SPI gels upon heating and that WG absorbs somewhat more water upon heating, leading to a slightly higher SPI concentration in the continuous phase.

#### Flow-Viz, a fully integrated and commercial in-line fluid characterization system for industrial applications

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A new fully integrated ultrasound based in-line fluid characterization system "Flow-Viz" has been developed especially for opaque, non-Newtonian industrial fluids. The new embedded and commercially available system is designed to meet industrial requirements. Flow-Viz enables true noninvasive, real-time Doppler measurements and is able to visualize and rheologically characterize industrial fluids continuously while providing continuous feedback to an existing process control system for enhanced efficiency and productivity. The Flow-Viz platform consists of an operator's panel a multi-touch monitor, an industrial CPU unit and a previously presented pulser-receiver electronics that has now been improved and extended by adding a second ultrasound channel, augmenting the on- board data processing for obtaining the optimal performances in high attenuating suspensions. A new Baseboard combined with a sbRIO board provides an additional 8-channel digital and analog input and output capabilities for simultaneous data acquisition and processing from a multitude of sensors. The new electronics is combined with a non-invasive ultrasound sensor unit, which allows measuring the flow velocity profile even through industrial high grade stainless steel pipes. This unique solution makes possible to adapt the high resolution UVP+PD technique for complex processes performed at high temperature or under pressure with all kind of industrial fluids. The Flow-Viz system is now commercially available and already installed in industry, e.g. for chocolate and grouting applications. An international patent has been filed.

#### Advanced rheometric tools for food applications

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Food products are rather complex multicomponent systems. Rheometric techniques have been used for gaining a better understanding of the structure but also of the behavior during the oral processing of food samples. The aim of this paper is to highlight some new techniques which further extend the rheometric tool box available for the food scientist. Due to stability reasons a structure is often highly desirable in food samples. On the other hand, since a good mouth feeling is required, these structures are mostly very week and are influenced at already very small stresses and strains. The measurement of a small elasticity in samples with low viscosity is therefor quite challenging. A new rheometer concept based on two air bearing supported electronically commutated (EC) synchronous motors provides an increased sensitivity. With one motor as the drive and one motor a kept at a fixed position and operated solely as a torque transducer the device is turned into an enhanced separate-motor-transducer rheometer (SMT) for rotational and oscillatory tests at a wide measuring range down to extremely low torques and normal forces. By selecting a measuring geometry which reduces the influence of fluid inertia and surface tension measurements of weakly elastic samples are possible. In the counter-rotation test mode the motors are set to rotate in opposite directions. The pre-set speed is divided and shared by the two motors, whereas the torque and normal force are measured at the upper motor unit. This mode is an invaluable option for microscopy applications by creating a stagnation plane allowing the observation of the structure elements of the sample during shear. New accessories allow controlling additional parameters like the humidity during rheological testing. Research in the last years showed that not just rheological but also tribological parameters are important for the mouth feeling. Special accessories turn the rheometer into a tribometer. Examples of tribological measurements and their correlation to the perception of certain attributes are presented.

### Impact of structuring of mixed gels on oral breakdown and sensory perception

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The aim of this study was to examine the effect of the composition, microstructural complexity and gel strength of mixed biopolymer gels on oral breakdown and sensory perception. Model mixed gel systems were prepared using  $\kappa$ -carrageenan and alginate at concentrations ranging from 0 - 2 % of the former. In one of the mixed gel system, calcium alginate beads were prepared first and then immobilized within the  $\kappa$ -carrageenan gel. The microstructure of the gels was characterized by electron microscopy and the rheological properties by dynamic oscillatory measurements and large deformation compression test. Sensory profiling by twelve trained panelists was also performed to evaluate the sensory properties. Different texture attributes including firmness, elasticity, extensibility, adhesiveness, melting rate in the mouth. Perceived oral processing time and difficulty were also recorded. Dynamic oscillation results showed that increase in concentrations of each of the biopolymer led to an increase in the storage modulus G". The mixed gels did not show significant difference in G' and the kinetics of gel formation as compared to pure  $\kappa$ -carrageenan gels at similar biopolymer concentration. The fracture behavior revealed that mixed alginate-K-carrageenan gels were more brittle. However, in case of gel structure containing calcium alginate beads entrapped within the carrageenan gels, there was significant increase in hardness, which might be attributed to the reinforcement of the gel structure by the presence of the beads or due to the residual calcium ions. Microscopical observation suggested that  $\kappa$ -carrageenan formed a continuous network with calcium alginate beads as the discontinuous phase. Preliminary probes of sensory profiling revealed that breakdown mechanisms depending upon the gel strength and the structural complexity were the major factors in driving the sensory perception of the mixed gels. This study confirmed the essential impact of structuring in sensory perception of mixed gels

### Predicting sensory parameters such as fat perception by tribology measurements

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Making correlations between instrumental measurements and sensory evaluations has long been a wish in the food industry. Rheology, texture analysis and other instrumental methods have had some success in predicting sensory parameters such as thickness, firmness, melting, breakdown, etc. However, when it comes to terms that we often associate with the word "mouthfeel" such as creaminess, smoothness, fattiness, slipperiness, coating, etc. it has been more difficult to extract this information from rheological measurements. A range of yoghurts with variations of fat, hydrocolloids and cultures have been examined by tribological measurements. In tribology the result in contrast to rheology is not a measure of sample properties alone. It reflects the interaction between two surfaces and the sample. Therefore the choices of surfaces are of great importance. A range of surfaces have been investigated to find the ones that work the best in predicting the "mouthfeel" parameters in dairy products. Information obtained by rheological and tribological measurements are compared and discussed. Finally the aim has been to investigate which parts of the tribology curves that best reflects specific sensory parameters. It has been investigated using chemometric tools such as iPLS and rPLS [1 - 2]. This has helped in interpretation of the tribological results and gives a quick guide to future investigations on which parts of the curves to focus on. Even though tribological parameters are not always perfect in predicting "mouthfeel" characteristics, it is a good supplement to traditional rheological and textural methods as a fast screening method in development of new products where these sensory parameters are in focus.

[1] Nørgaard L, Saudland A, Wagner J, Nielsen JP, Munck L, Engelsen SB: Interval Partial Least Squares Regression (iPLS): A comparative chemometric study with an example from Near-Infrared Spectroscopy, Applied Spectroscopy 54 (2000) 413-419.

[2] Rinnan A, Andersson M, Ridder C, Engelsen SB: Recursive weighted partial least squares (rPLS): An efficient variable selection method using PLS, Journal of Chemometrics, DOI: 10.1002/cem.2582.

#### Identification of formulation effects on cake softness using a technique combining alveolar structure analysis with mechanical properties characterization

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Cake softness is a sensory descriptor not clearly defined. It is difficult to instrumentally characterize softness. Generally it is limited to the analysis of the mechanical properties of the aerated crumb. Indeed, thickness of alveolar walls, size of alveoli and openness of the crumb structure are all structural criteria which may have an effect on mechanical properties and thus, plays a key role in textural properties of soft cakes. The aim of this study was to characterize cake softness by combining alveolar structure analysis with aerated crumb and cell walls mechanical properties analysis. First investigations were made on quantifying the impact of formulation (including ingredient storage) on cake alveolar structure by using an image processing technique based on the mathematical morphology method combined with data analysis by Principal Components Analysis (PCA). PCA simplified the interpretation of the image processing results by classifying crumb structures according to their granulometric curves. Product mapping was depending on two axes that could be related to crumb fineness and heterogeneity. It appeared that flour storage temperature and time and emulsifier quality play a major role on cake crumb fineness and heterogeneity. Further investigations on mechanical properties of both aerated cake crumb and alveoli walls were then carried out to better understand effects of formulation on mechanical properties of cake crumb. Emulsifiers were found to have an effect not only on alveolar structure (alveoli size and heterogeneity) but also on aerated crumb mechanical properties and on cell walls firmness. Therefore, it was possible to explain which mechanical properties are due to either aerated cake structure or cell walls firmness.

#### Yield stress determination – impact of sample handling and loading, measuring method and type of evaluation

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The yield stress affects stability as well as texture, mouthfeel, perception and finally consumer acceptance of many processed foods. Therefore, it is desired to determine the yield stress in a quick, reliable and reproducible way, particularly in quality control (QC). However, the result is strongly dependent on sample handling and loading as well as the used measuring method and type of evaluation [1]. Moreover, because of the variety of available approaches, many users face conceptual confusion when trying to extract "the" yield stress. The aim of this contribution is to show the differences among the rheological methods and evaluations and provide a guideline on how to set up SOPs (Standard Operating Procedures) for sample handling and sample loading. A new mechanical bearing QC rheometer was used to characterize different pourable and spreadable commercial foods. A detailed comparison of the results obtained with rotational, oscillatory and vane rotor measurements as well as the different evaluation methods is presented. Our results show that the most suitable methodology for each food depends on the sample properties as well as particular needs in QC.

[1] Dapcevic T, Dokic P, Hadnadev M, Pojic M: Determining the yield stress of food products: Importance and shortcomings, Food Processing, Quality and Safety 36 (2008) 143-149

### A novel process viscometer for food rheology applications

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Vibrational process viscometers have been known for nearly 60 years but have found only modest application in food manufacturing processes, where control of product consistency is a high priority. This is due to the ingrained belief on the part of process operators that only measurements defined shear are capable of predicting the behavior of the usually non-Newtonian, often inhomogeneous products that they manufacture. A new instrument, the Rheonics SRV process viscometer, is descended from a line of vibrational viscometers that have found wide acceptance in the food process industry because they have proven themselves to be capable of controlling consistency in complex processes such as batter mixing, cheese coagulation, and beer mashing. The SRV builds on this heritage and adds to it a novel balanced resonator structure that makes it compact, simple to install, and free from the sensitivity to mounting conditions that has plagued earlier designs that lack its symmetric structure. Since vibrational viscometers function by measuring the rate of energy loss of a resonator immersed in the fluid under test, any additional energy losses due to vibrations transferred to surrounding structures place limits on the stability and reproducibility of the instrument's measurements. The Rheonics balanced resonator (patent pending) ensures the highest possible reproducibility and stability in a resonant instrument by eliminating transmission of the resonator's vibrations to surrounding structures. Further improvements to traditional vibrational viscometers include patented systems for rejection of ambient vibration due to pumps and other process machinery, as well as advanced electromagnetic transducers with unprecedented rejection of magnetic interference from neighboring machinery. The Rheonics SRV has already proven itself in a complex slurry-coating process line for which the operator initially believed that only a rheologically accurate instrument could bring the process under control. The operator found that the high sensitivity and stability of the SRV enabled control of the process by a single point measurement, whereas a battery of other instruments capable of measuring density and solids content were not sufficiently sensitive to measure the small variations in the process stream that were crucial to a uniform, adherent coating.

#### Particle Image Velocimetry and its potential to improve high shear rate processing of food suspensions

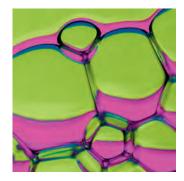
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Over the last decades Particle Image Velocimetry (PIV) has become a standard application for flow related research of any kind. Through recent developments in digital imaging quality and low cost computing, PIV setups are widely accepted and affordable. They allow for an almost on the fly imaging of a pure fluids flow field in its entirety. PIV has its limitation when it comes to nontransparent fluids, for instance most suspensions and emulsions. Nevertheless, if careful preparations are carried out, it is possible to create transparent suspensions with a particle fraction of over 55 %by volume. There are even some examples of colloidal suspensions that are naturally transparent, e.g. some starch based suspensions. The mobility and the low risks and efforts concerning security measures of the PIV technology allow investigating flows directly where they occur if a transparent apparatus can be provided. The flows of food suspensions can be traced and rheological parameters like the shear rate can be derived directly from the velocity profile. So it is also feasible to investigate the flow in rheological measurement devices like rheometers. In the present study, we show the possibilities of PIV rheology of suspensions by means of wall slip investigations in a transparent slit die as well as the influence of the yield stress and the resulting deviation from of standard rheology results. A thoughtful preparation to achieve highly transparent suspensions is outlined and some troublesome effects like the temperature dependence of the refractive index are dealt with. Furthermore, the potential to directly observe wall slip behavior and the distorted velocity profile which prevents the classical rheological interpretation of the capillary rheometer can be seen in this research. A possible application in food rheology is exemplarily shown by the analysis of starch based fluids. With this setup we aim for the enhancement of suspension flow models and the study of flow profiles in real applications.







# Emulsions and Interfaces

### Coarsening mechanisms of alkane-in-water okra pectin stabilized emulsions

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The influence of oil (n-hexadecane, n-dodecane) and emulsifier type (okra pectin extracted at low and high pH values) on coarsening of acidic oil-in-water emulsions was studied. Ultrasound-assisted emulsification at pH 2.0 was employed in order to fabricate emulsions of smaller droplet sizes and improved stability on aging. Emulsions were characterized by means of static light scattering to evaluate the droplet size distribution (PSD), zeta-potential measurements, rheometry and confocal laser scanning microscopy. Interfacial composition analysis was performed by determining protein, pectin and acetyl contents at the of oil-water interface. Time-dependent destabilization kinetics of emulsions was followed by the Lifshitz-Slyozov-Wagner theory. Okra pectin extracted at higher pH showed greater emulsion stabilizing capacity than those extracted at lower pH values, as revealed by PSD analysis and the rate of destabilization kinetics. Ostwald ripening was assigned as the governing destabilization mechanism for emulsions stabilized with pectin extracted at high pH. Emulsions stabilized with okra pectin extracted at low pH values exhibited a rapid droplet growth, due to Ostwald ripening-induced coalescence as evidenced by the development of the PSD curves on storage. It was also shown that the high molecular weight improves the emulsification capacity of okra pectins as indicated by long-term stability tests. Findings show that okra polysaccharides demonstrate remarkable differences in their stabilizing properties and magnitudes of Ostwald ripening and coalescence events depending on the employed isolation protocol. Overall, pectins extracted at higher pH values have greater stabilizing potential than those extracted at lower pH values. The present study shows the potential of okra pectins as emulsifiers under acidic conditions and serves as the basis for the development of effective strategies to prevent emulsion instability in such systems.

### Multiple W/O/W emulsion systems with adaptive transient thickening behavior

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Multiple water-in-oil-in-water (W/O/W) emulsions consist of simple W/O emulsions, which are further dispersed in an outer aqueous phase. A special benefit of W/O/W emulsions is that in the case of an osmotic imbalance between both water phases, water transport over the oil layer is induced. Water transport towards the inner water droplets results in droplet swelling and thus thickening of the emulsions. A controlled swelling enables the adaptation of the rheological properties of W/O/W emulsions after processing. The water transport mechanism was frequently discussed in literature. However, a comprehensive study of the impact of several compositional parameters on the water transport and thus clear evidence of combined mechanistic effects are still outstanding. In this study the water transport mechanism was investigated by reducing the complexity of W/O/Wemulsions. The production of monodisperse W/O/W emulsions with a single internal water droplet by microfluidics permitted a very precise study of the swelling kinetics. This method allowed not only investigations of the long-term evolution, but also early stage investigations. Two different stages of swelling were identified, a lag stage and an osmotically dominated stage. We gave evidence that an experimental decoupling of the transport mechanisms in these stages is needed to determine their nature and related parameters. The actual swelling which was caused by the osmotic pressure difference between both water phases, started after a certain lag time. Yet, the osmotic pressure difference had no significant influence on the duration of this lag time. Thus, the osmotic imbalance drives the water transport in W/O/W emulsions, but it does not initiate it, as widely assumed. In fact, the osmotic pressure difference can only act if both water phases are in full thermodynamic contact. This requires an oil layer, which is extremely thin and/or highly permeable to water. It was concluded that the lag time is jointly determined by the oil film thinning due to droplet sedimentation and a water film formation over the oil layer, the latter being supported by the spontaneous formation of water droplets in the oil phase induced by the lipophilic surfactant.

### Temperature is key to yield and stability of protein stabilized microbubbles

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The effect of preparation and storage parameters on the number, size and stability of microbubbles covered with bovine serum albumin (BSA) was investigated. A large number of microbubbles with a high stability were obtained at protein concentration of 7.5% or higher, at pH between 5 and 6, at a ionic strength of 1.0 M and at a preheating temperature of 55 - 60°C. Microbubbles stored at 4°C were more stable than those stored at room temperature. This was observed for a specific commercial BSA batch. We found that optimal preparation parameters strongly depend on the batch. Certain BSA batches were found not to lead to microbubbles at all. Microbubbles made with different protein concentration and preheating temperatures shrunk in time to a radius between 300 nm and 350 nm, after which the size remained constant during further storage. We argue that the constant final size can be explained by a thickening of the microbubble shell as a result of the microbubble shrinkage and thereby withstanding the Laplace pressure. The effects of the protein concentration, pH and ionic strength on the number of obtained microbubbles directly after sonication can be ascribed to the influence of these parameters on the adsorption speed and ability to cover the surface of air bubbles formed during sonication with enough proteins to stabilize the bubble against coalescence and dissolution. We suggest that the effect of temperature during sonication on the formation of microbubbles can be related to thermally induced proteinprotein interaction at the air-water interface. After formation these interactions have a temperature dependency, which might explain the difference in stability during storage at 4 and 21°C

### Effect of processing conditions on the foaming behavior of casein micelle dispersions

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Various studies indicated that the foaming properties of milk are strongly influenced by the composition of the milk as well as the conditions applied during processing [1]. However, the role of surface and bulk components in complex food systems like milk on foam formation and stability is still not well understood. For example, it often happens that a certain batch of milk does not foam properly while others do, even if they are processed in the same way. It is still unclear what causes this and how it can be solved. Previous studies indicated that casein micelles play an important role in the stability of milk foams. Therefore, we studied, at 20°C, the foaming behavior, interfacial, and thin film properties of casein micelle dispersions (CMD) that were initially prepared at different temperatures (4 and 20°C). CMD prepared at 4°C, with more aggregates of casein micelles and  $\beta$ -casein in the serum, resulted in much more stable foams than CMD prepared at 20°C. We investigated the linear and nonlinear surface rheology of air/water interface stabilized by CMD at different frequency and strain. Large deformation surface rheology analyzed by Lissajous plots showed significant strain hardening in compression and a yielding behavior of the interfacial structure when the interface is expanded. The frequency dependence of the dilatational modulus is significantly different between CMD's prepared at 4 and 20°C, and at low frequencies the modulus of the CMD prepared at 4°C is about two times higher than the modulus of the CMD prepared at 20°C. This correlates well with the observed higher stability of the according foams. Atomic force microscopy was conducted to study the structure of the air/water interface of CMD. Thin film structure and stability were also investigated. By analyzing correlations between the findings obtained with these experiments and foaming behavior, we assessed the role of processing conditions on the stability of CMD foams.

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#### Microencapsulation systems for heat sensitive, oil-soluble components

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Oil-soluble food ingredients sensitive to degradation can be protected from their environment by microencapsulation. In an O/W1/W2 system, these oil soluble compounds are dissolved in small oil droplets, dispersed within a gelled matrix microbead (W1), which forms a barrier from outside influences. The stability and degree of protection delivered by the gel matrix depends on its structure and strength, and these in turn depend on the gelling process. For heat sensitive ingredients this process is typically a cold-set gelling process. Here we investigated the effect of variations in matrix polymer (alginate and WPI aggregates), the gelator (acid and calcium nanoparticles), and method of gelation (internal and external) [1 - 3], on the ability of microcapsules to retain oil droplets, and maintain their integrity during the production process. The effects of these parameters on gel strength were tested on macroscopic gels, the smoothness of the surface of microcapsules without oil was investigated with SEM, and the integrity of microcapsules with oil was investigated with CLSM. External gelation with calcium chloride nanoparticles gave the smoothest and strongest microcapsules for both the protein and alginate microcapsules. Internal calcium gelation of microcapsules (induced with calcium carbonate nanoparticles and GDL) produced a mixture of smooth and rough microcapsules, most likely because of the difficulty of ensuring a completely homogeneous distribution of the calcium nanoparticles and GDL on the small scale of a microcapsule. The internally gelled alginate microcapsules were all destroyed in the production process while about half of the internally gelled protein microcapsules were destroyed. Microcapsules produced by acid induced gelation of WPI aggregates (using GDL to lower the internal pH), gave slightly rough microcapsules of which about half were destroyed during the production process. When the pH was brought further from the isoelectric point, the capsules became rougher and showed less integrity. These observations will help in designing new gel-based microcapsules with stability and release patterns tailored for a wide range of sensitive, oil-soluble ingredients.

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[2] Paques JP, Sagis LMC, van Rijn CJM, van der Linden E: Nanospheres of alginate prepared through w/o emulsification and internal gelation with nanoparticles of  $CaCO_3$ , Food Hydrocolloids 40 (2014) 182.

[3] Paques JP, van der Linden E, van Rijn CJM, Sagis LMC: Alginate submicron beads prepared through w/o emulsification and gelation with  $CaCl_2$  nanoparticles, Food Hydrocolloids 31 (2013) 428.

### Bacterial cellulose as an alternative thickener for o/w emulsions: Comparison with common thickeners

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Cellulose is the most abundant natural polysaccharide, being the major structural component of plants. Chemically modified celluloses are well known food thickeners. Recently, besides them, bacterial cellulose (BC) has received attention. BC has good water binding capacity and sufficient network formation through formation of fiber-like structures. The role of BC as a thickener in the continuous phase of emulsions has not been extensively studied and the effect on the rheological behavior of emulsions is not well known. Xanthan gum (XG) and locust bean gum (LBG) are hydrocolloids commonly used as thickeners in emulsions. However, both XG and LBG are high priced and the food industry would benefit from cheaper alternatives. Hence, the objective of the present study was to investigate the effect of BC (0 - 1 %wt) addition in whey protein isolate (WPI) (2 - 5 %wt) stabilized emulsions on the properties of the emulsions in terms of droplet size, stability and rheological behavior and to compare the rheological behavior to emulsions thickened with xanthan gum (XG) and locust bean gum (LBG). At lower BC concentrations (0.5 - 0.7 %wt), extensive aggregation led to unstable emulsions due to bridging flocculation. However, at higher concentrations (1 %wt BC), stable emulsions were obtained due to either steric stabilization or network formation of BC fibrils. The viscosity profile of the BC emulsions showed three regions, including two shear thinning regions separated by a constant viscosity plateau (Newtonian). To compare the ability of BC to act as a thickener with XG and LBG, emulsions were prepared with different thickener concentrations. Their rheological profile showed that BC experienced similar shear thinning behavior as XG, but smaller amounts of BC was needed to obtain the same zeroshear viscosity. These results showed that BC is a good alternative for commonly used thickeners with future applications in the food industry.

### Food grade ingredients and their effects on the production and stability of air filled emulsions

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Air filled emulsions have been shown to be a promising material for the improvement of food stability and the reduction of fat within emulsion based food products. While air filled emulsions are still a relatively new material within the food industry, to prove that air filled emulsions are safe and effective within a variety of food products, not just a simple oil-in-water emulsion stabilised with a single emulsifier, attention must be made to their interactions and behaviour when they are combined with the materials that they would likely encounter either during food production or within the foods themselves. Therefore this study has been undertaken to determine the effect that pre- and post- production mixing, with various food grade materials, has on the air filled emulsions both mixed with and independent of a food grade oil-in-water emulsion.

### Relating foam and interfacial rheological properties of $\beta$ -lactoglobulin solutions

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We have determined bulk rheology of  $\beta$ -lactoglobulin (BLG) foams and surface viscoelasticity of corresponding protein solutions varying pH as well as type, valency and concentration of added salt in a wide range. Foam rheology was characterized by the storage modulus  $G_0$ , the apparent yield stress  $\tau_y$ , and the critical strain  $\gamma_{c,foam}$  defining the cessation of linear viscoelastic response. These quantities were determined at gas volume fractions  $\phi$  between 82 and 96 %. Surface viscoelasticity was characterized in shear and dilation, corresponding shear and dilational moduli  $G_i$ ', E' as well as the critical stress  $\tau_{c.surface}$  and strain  $\gamma_{c.surface}$  marking the onset of non-linear response in oscillatory surface shear experiments were determined at fixed frequency. Beyond the widely accepted assumption that  $G_0$  and  $\tau_y$  are solely determined by the Laplace pressure within the droplets and the gas volume fraction we find that both quantities strongly depend on corresponding interfacial properties.  $G_0$  increases linearly with  $G_i$  and even stronger with E',  $\tau_u$ varies proportional to  $\tau_{c,surface}$  and  $\gamma_{c,foam}$  scales linearly with  $\gamma_{c,surface}$ . Furthermore, deviations from these simple scaling laws with significantly higher reduced  $G_0$  and  $\tau_u$  values are observed only for foams at pH 5 and when trivalent salt was added. Then also the dependence of these quantities on volume fraction  $\phi$  is unusually weak and we attribute these findings to protein aggregation and structure formation across the lamellae then dominating bulk rheology [1].

[1] M. Lexis, N. Willenbacher: Relating foam and interfacial rheological properties of beta-lactoglobulin solutions, Soft Matter DOI:10.1039/C4SM01972E (2014)

### Dynamic interfacial tension measurements during the first milliseconds of emulsion formation

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Many food products are emulsion-based systems, and generally they are made using high shear devices that induce fast droplet break-up (i.e., in the millisecond range) in the presence of emulsifiers. Emulsifiers have two roles; decreasing the oil-water interfacial tension, and further stabilizing the droplets. It is expected that emulsifier adsorption occurs at similar time scales as droplet breakup during emulsification; it is therefore crucial to understand the early steps of emulsion droplet formation and emulsifier adsorption. However, conventional tensiometry techniques are not able to measure changes in interfacial tension related to emulsifier adsorption at such small timescales. A device that can enable the measurement of interfacial tension the millisecond range is the microfluidic Y-junction, which is the focus of the present work. Here, the droplet size depends on the shear of the continuous phase, and on the interfacial tension. A reduction in the interfacial tension as a result of emulsifier adsorption therefore leads to the formation of smaller droplets. Based on the droplet size and flow rates, the interfacial tension at the moment of droplet breakup, also called dynamic interfacial tension, can be measured in the millisecond range. A model was constructed that relates the droplet size to the shear exerted by the continuous phase and the interfacial tension. With that model the interfacial tension at the moment of droplet formation (typically between 0.5-10 milliseconds) could be calculated. The surfactant coverage of the oil droplet depends on the process conditions, leading to different values of the dynamic interfacial tension at the moment of droplet break-up, and we were able to link this to droplet formation time and emulsifier concentration. The proposed method allows exploration of surfactant and protein behavior at the oil-water interface at very short time-scales, which is not possible through any other technique. It is expected that this information will help the food industry to optimize their ingredient formulation and processing conditions, using adsorption behavior as a starting point of their product and process designs. In that respect, the suggested technique could revolutionize the way emulsions are made.

#### Interfacial behavior and rheology of oxidized proteins and lipids

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Food proteins such as dairy proteins are widely used in food applications, for example, in emulsion or foam-based products. Often production processes for such products involve thermal or mechanical stresses, which can damage the native structure of proteins, and affect their functionality. In addition, in the presence of other reactive molecules (e.g., oxidizing unsaturated lipids), proteins can be subjected to chemical modifications (such as oxidation). However, little is known on how such modifications of proteins affect their interfacial behavior and their emulsifying properties. We investigated the effects of oxidation of dairy proteins, and of lipid oxidation products, on protein adsorption kinetics and surface rheological properties of oil-water interfaces stabilized by whey proteins, using a drop tensiometer. The effects on dilatational properties were determined in large amplitude oscillatory deformations, and analyzed using Lissajous curves. Interfacial films formed from oxidized whey proteins were less elastic at small deformation values, compared to native whey proteins, and displayed strain hardening in compression and strain thinning in extension, upon increasing deformation. The presence of oxidized lipids also greatly decreased the elasticity of native protein-based interfaces. Protein and lipid oxidation can thus alter the formation and properties of oil-water interfaces, which may in turn affect the quality of food emulsions.

#### Interfacial viscoelasticity of protein-polysaccharide composite layers at an oil/water interface

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Proteins and polysaccharides are the essential functional ingredients determining the texture and shelf-life of most food products, hence understanding components interactions at the interfacial level, including the rheological properties is essential if advances are to be made in controlling and manipulating production, usage efficacy and consumer perception of food/pharmaceutical products during fabrication and storage. Here, we study the kinetics of adsorption and interfacial viscoelasticity of laminated layers of a milk protein ( $\beta$ -lactoglobulin)-polysaccharide (sodium alginate) at the oil/water (O/W) interface, differing in aqueous phase pH and ionic strength when subjected to oscillatory shear deformations. We were able to monitor multilayer(s) formation at the O/W interface. Our results showed that the protein adsorption and the formation of an elastic mono/multilayer at the interface are fast. Upon lamination of the next layer(s) via electrostatic interaction complex viscoelastic behavior exhibited with a high elastic modulus and smaller critical value of surface stress above which, the layer(s) are disrupted and shows purely viscous properties. Qualitatively similar rheological properties of the layers at different ionic strength and pH were observed. The results can be discussed with respect to ascertaining the correlation and causation relationships between the possible molecular mechanisms of generation of the elastic films at the O/W interfaces, microstructure of the layer(s) and the rheological response.

#### Advanced rheological measurements in food emulsions: Interfacial double wall ring and microscopy

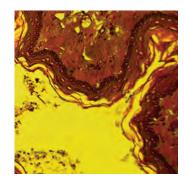
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The rheological characterization of thin liquid layers at interfaces is motivated by their intensive use in a variety of industries, ranging from enhanced oil recovery, food technology to biomedical applications. The most used devices for measuring interfacial properties that can be attached to a commercial rheometer are the Du Noüy ring, disc and bi-cone geometry. The Du Noüy ring is only suitable for purely viscous interfaces. The bi-cone and disc geometries can be used for viscous and viscoelastic interfaces, but the sensitivity is limited because these devices intrinsically possess a large area in contact with the bulk fluids. Newly developed Double Wall Ring (DWR) geometry is used with a standard rotational rheometer to characterize both viscous and viscoelastic properties of interfaces. It has the advantages of a good intrinsic sensitivity due to a small area in contact with the bulk phase and well defined flow fields that enable bulk and surface contributions to be separated. The double-wall ring geometry has a square edged cross-section and is used with a temperature controlled bottom circular channel arrangement to contain the fluid. Results on model interfaces show that the new geometry is able to measure viscous as well as viscoelastic interfaces in both continuous and oscillatory shear experiments. In this work, we used the DWR to study the interfacial viscoelastic properties of two food emulsifiers. One is a low molecular weight monoglyceride and the other is a high molecular weight sodium caseinate protein. Both are emulsifiers widely used in the food industry. One of the main applications of these emulsifiers is the stabilization of food emulsions. Analysis of their mechanical properties will be shown along with the evolution of interface formations due to the molecules migration to the water/oil interface. Structural information of both interfaces will be compared. On the top of that, a microscope attached to the transparent lower plate is used to probe the technique in food emulsions and to obtain better understanding of the processes involved.







# Cheese, Chocolate, and Health

### Laboratory scale cheese with model filler particles: How structure controls rheological properties

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The addition of various fillers (e.g. hydrocolloids) as fat replacers in cheese has been of particular interest in dairy research. In order to control the effect of these fillers, the mechanisms behind the way they affect the cheese structure and properties need to be fully understood. This study investigates the influence of incorporating non-fat filler particles (glass beads, silicone beads, and agar beads) on the structure, material, and functional properties of cheese. The samples, prepared using a Rapid Visco Analyzer, were analyzed with confocal laser microscopy (CLSM) and small amplitude oscillatory rheology. CLSM images showed that the glass beads were distributed within the protein matrix of the cheese, whereas the silicone particles were located within the fat phase. Rheological measurements revealed that, when increasing the volume fraction of glass beads, the complex modulus of cheese increased, following Krieger-Dougherty and Kerner equations. However, in the case of silicone microspheres, these equations over-predicted the experimental data. Located in the fat phase, the silicone fillers exhibited the same mechanical response as that of increasing fat content. When adding silicone microspheres in fat-free cheese, the equations predicted the effect of volume fraction and particle hardness on the cheese modulus more accurately. Agar microgels were also added into fat-free cheese. Oscillatory measurements revealed that the microgels decreased the complex modulus and the crossover temperature, indicator of the melting point, of the cheese. It is proposed that the water would have diffused from the microgels to the protein matrix, under osmotic pressure gradient, therefore altering the properties of the cheese. In conclusion, the influence of filler particles on the rheological properties of cheese depends on their nature and on their location in the protein or in the fat phase. This study highlights how the material properties of cheese can be governed by its microstructure.

### Tailoring flow behavior and texture of water continuous cocoa suspensions

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The microstructure of low fat food suspensions must be carefully adjusted to meet customer expectance. Here we discuss how appropriate heat treatment or the addition of an adequate immiscible secondary fluid can be used to tailor the texture and flow of water based cocoa suspensions in a wide range. Comparing suspensions of untreated and organic solvent (n-hexane and ethanol) extracted cocoa particles we demonstrate that the residual cocoa butter is decisive for the control of the structure and flow behavior of these suspensions. Upon heating to 30 - 35°C the low shear viscosity and apparent yield stress of suspensions from untreated particles increases by more than two orders of magnitude and this is attributed to the formation of a particle network hold together by the sticky, semi-crystalline cocoa butter leaking from the interior of the particles. This structure remains stable upon cooling to 20°C but it vanishes when the suspension is heated to temperatures  $T > 40^{\circ}$ C. Then the cocoa butter detaches from the cocoa particles forming separate emulsion droplets as confirmed by fluorescence microscopy. A similar structure build-up can be achieved at room temperature if trace amounts of polar organic solvents like oleic acid, octanoic acid or octyl acetate are added. This is attributed to the partial solubility of cocoa butter in these solvents and the formation of a sticky surface layer formed by these butter/solvent mixtures as confirmed by small angle neutron scattering experiments. The solubility rate determines the kinetics of particle network formation. The creamy texture provided by the formation of a particle network either using heat treatment or addition of a secondary fluid opens up new pathways for the formulation of low fat food products based on suspensions of cocoa or other finely dispersed particles.

# A molecular dynamics approach to explore the interaction of lecithin molecules and sucrose particles in chocolate

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It is well known that lecithin lowers the viscosity of liquid chocolate mass because of the amphiphilic molecular structure of its components. Lecithins are a natural mixture of components with hydrophobic tail groups of variable length and degree of saturation and varying hydrophilic head groups. These different molecules have a different impact on reducing the viscosity. This work presents a molecular dynamics approach to characterize different lecithin molecules regarding their impact on the chocolate conching process. We are using a so-called steered molecular dynamics method, the pull-code, to explore the detachment energies of particular lecithin molecules from a sucrose crystal surface embedded in a cocoa butter medium. For the lecithins, we focus on the influence of different head groups like phosphatidylcholine (PC), phosphatidylethanolamine (PE) and phosphatidylinositol (PI), while cocoa butter is modelled using three major triacylglycerides: 1,3-rac-palmitoyl-stearoyl-2-oleoyl glycerol (POS), 1,3-distearoyl-2-oleoyl glycerol (SOS), and 1,3dipalmitoyl-2-oleoyl glycerol (POP). The sucrose surface is built by replicating the crystal unit cell in all three spatial dimensions. The pulling is done with constant velocity at realistic conching temperature. The work needed to detach the molecule is the integral of the force over the pulled distance and can be interpreted as an upper estimate for the free energy difference. Our results show that PC molecules and PI molecules require a higher work to get detached from the sucrose surface than PE molecules. For the molecular dynamics simulations, the General Amber Force Field was chosen to describe the inter- and intramolecular interactions of the compounds, which was designed to cover a very wide range of organic molecules. The Gromacs Software package was used to calculate all simulations. The presented methods provide insight into challenges in molecular dynamics simulations of complex food systems and demonstrate possible solutions.

#### Effect of varying insoluble volume fraction on rheological and recovery properties of mixed colloidal suspensions

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The rheological behaviour of food materials at rest, during shear and recovery are critical considerations in the production and performance of many food systems. At present, relatively little research work has been done to explore the fundamentals of flow and recovery behaviour of mixed colloidal systems (consisting of soluble and insoluble hydrocolloids). The presence of insoluble particles confined within a biopolymer matrix can affect the flow and recovery behaviour. Understanding the particle-biopolymer interactions will provide insights assisting the design of highly responsive pastes suitable for 3-D printing and aiding the development of food products with rapidly alterable textures. In this study, the effects of insoluble volume fraction, concentration and flow behaviour of continuous phase on the flow and recovery behaviour were studied using a model system of microcrystalline-cellulose (MCC) dispersed in a continuous phase of glycerol and xanthan gum in water.

### Two rheological methods to approach the gastric behavior of infant formulas

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It is well known that one key parameter for food digestion is the rate of emptying of the stomach. However, during its transfer through the stomach, due to the physico-chemical conditions of this compartment (pH, content in digestive enzymes, rate and intensity of contraction), the structure of foodstuff and their rheological properties are strongly modified. This affects the rate of transfer of food into the intestine. The gastric step is particularly essential for colloidal emulsions such as dairy liquids that will gel or precipitate during that step, with changes on their residence time in the stomach. So, our aim is to understand how the structure and the rheology of dairy colloidal emulsion evolve during their digestion and impact the hydrolysis rates of proteins and lipids and the bioavailability of these macro-nutriments. Taking into account these structural and rheological changes is particularly crucial in neonatal nutrition to develop optimized infant formulas. We performed in vitro dynamic gastrointestinal digestions of different infant formulas [1]. At some specific steps during the digestion, we determined the structure and the rheology of the bolus, together with the hydrolysis rates of proteins and lipids. Rheological measurement can be performed on aliquoted samples by shearing at a low rate. But, only limited sampling can be practiced because digestion systems are limited in volume and sampling the bolus for rheological measurements can introduce bias by applying additional shearing to the sample. Thus, we proposed performing continuous rheological measurements in parallel to the dynamic digestion study. Two continuous rheological methods were tested on different infant formulas, by shearing or small amplitude oscillation. The changes evidenced with these two methods and the one observed in the digestion system were compared. In conclusion, these two methods gave relevant results and were shown to be much simpler than the digestion system. They could be applied to predict the rheological behavior of formulas. Moreover, the methods allowed discriminating the infant formulas in relation with the technological process applied for their manufacture.

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#### Controlled mechanical and structural properties of biopolymer adsorption layers under gastric conditions

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Human lipid digestion begins at the interface of oil and water by interfacial adsorption of lipases. Tailoring the available surface area for lipase activity can lead to specific lipid sensing in the body, hence defined satiety hormone release [1]. This surface area is linked with the stability of the lipid emulsion, and thus is mainly decreased in gastric environment [2]. As emulsion stability is directly influenced by the interfacial structure, we tailor the composition of interfacial active material to generate interfacial membranes, which vary in their gastric stability [3]. The viscoelasticity of membranes formed by biopolymers was monitored online by interfacial rheology applying several in vitro digestion steps. These observations allowed understanding and thus manipulating their viscoelastic layer evolution during in vitro digestion. Whereas a protein based membrane (beta-lactoglobulin) softened and finally degraded during proteolysis, its combination with methylcellulose thermogelled and was stable during enzymatic degradation (gastric lipolysis and proteolysis). Coexistence of the two emulsifiers at the air/water interface is evidenced by neutron reflectometry measurements, where morphological information is extracted. Emulsions formed by these emulsifiers showed that gastric lipolysis of MCT oil depend not only on the interfacial composition, but also on the thickness of the interfacial biopolymer layer. At a critical concentration of methylcellulose gastric lipolysis was completely inhibited. The utilized layers and their analysis provide knowledge of physicochemical changes during in vitro digestion of interfaces, which promote functional food formulations.

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#### Interactions of delivery systems with gut epithelial mucus studied by drop tensiometry coupled with relevant in vitro absorption models

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The structure of delivery matrices in foods needs to be carefully designed to impart stability during processing and storage, but even more importantly, to ensure optimal delivery and absorption of the bioactives in the gut. Various in vitro digestion methods have been designed to mimic digestion and absorption, but little is understood on mucus nutrient interactions in the intestinal tract, albeit this may be a major factor affecting bioactive uptake. In this study a physiochemical characterization of mucus- nutrient complexes by means of drop shape tensiometry was employed and coupled with in vitro cell absorption using a human intestinal cell culture model producing mucus. Mucus was isolated form human intestinal adenocarnicoma cells, Ht29-MTX growing after confluency. Two different human cell models were used, one mucus producing a co-culturing of adenocarcinoma Caco-2 cells and Ht29-MTX cells and a non-mucus producing, Caco-2 cells alone. The differences shown in absorption were correlated with the different behaviors displayed at the interface measured by drop tensiometry. Two different delivery models are presented: protein particles carrying polyphenols and liposomes carrying polyphenols or  $\beta$ -carotene. Results showed that mucus - protein complexes formed at the interface changed the interfacial rheology of the mucus layer. In addition polyphenol binding affected the behavior of the complexes formed at the interface leading to the formation of stronger interfacial networks. The results demonstrated that a material science approach combined with the use of relevant in vitro models can aid in understanding the changes occurring during digestion to food delivery systems and can help identify the factors affecting the relationships between matrices structure and delivery of bioactives.

### Mechanistic approaches towards understanding the kinetics of gastric digestion

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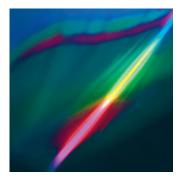
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The gastric digestion of food into dissolved or dispersed nutrients is an essential step in human nutrition and health. Although there are various studies on digestion, food scientists still need to better understand its underlying mechanisms. However, the gastric digestion of food is often regarded as a black box and only the outcomes of the black box are examined, since the process is extremely complex. The complexity lies in the diversity and complexity of food, in the deformation dynamics of food structures, in the enzymatic kinetics for the non-ideal conditions, and in the release of nutrients from heterogeneous matrices. We are trying to dismantle the digestion process from a process engineering perspective. The gastric digestion process consists of the physical disintegration, the chemical breakdown and the release of nutrients, we interpret them as texture/structure transformation, reaction kinetics and mass transfer processes, and we regard the stomach as a bioreactor. To understand these processes, interdisciplinary approaches were applied. Whey protein gels were used as a model for protein-based solid food. A degree of hydrolysis assay and Size Exclusion Chromatography were used to determine the enzymatic kinetics in the gel digestion. Scanning Electron Microscopy (SEM) was used to observe the microstructural changes of protein gels during digestion. Fluorescence Correlation Spectroscopy was applied to investigate the diffusivity of pepsin in gel matrices. We found that the gel structure was hindering the hydrolysis of protein. However, the hindrance is not simply slowing down the hydrolysis, but also altering the enzyme kinetics to some extent [1]. SEM images showed that the microstructure of gel was changed during digestion, the microstructures were characterised with image analysis. Overall, these approaches are able to bridge the digestion process with theories from physics and chemistry, which may lead to better knowledge on the underlying mechanisms of protein digestion.

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# Rheo-SALS, SANS, SAXS

#### Geometric and material effects on sensory properties of confectionery wafers and similar extruded products

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Traditional baking is the most common way of producing confectionery wafers, however over the past few decades, the extrusion process has become an increasingly important food manufacturing method and is commonly used in the manufacturing of breakfast cereals and filled snack products. This study aims to characterize products made via each of these manufacturing processes in order to understand the important parameters involved in the resulting texture of confectionery products such as wafers. Both of the named processes result in brittle, cellular foams comprising of cell walls and cell pores which may contain some of the confectionery filling to make the material an open or closed cell foam. The mechanical response of the cell wall material and the geometry of the products influence the consumer perception and preference. Xray micro tomography was used to generate geometry of the microstructure which was then fed to Finite Element for numerical analysis on both products. The Finite Element models were used to determine properties such as solid modulus of the cell walls, Young's modulus of the entire foam and amount of solid material within the cell faces. Results showed that the extruded product was a much stiffer product with a higher Young's modulus and was found to be a closed cell foam as compared to the wafer which was found to be an open cell foam. Numerical results were validated by comparing with experimental results where possible. It is concluded that the differences in the microstructures of the two products resulted in differences in the mechanical properties and therefore texture and sensory properties of the products.

#### Small-angle neutron and X-ray scattering of pH-responsive micelles based on short, medium and long chain saturated fatty acids

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Free fatty acids play a vital role as fuel for cells and in lipid metabolism. During lipid digestion in the gastrointestinal tract, triglycerides are hydrolyzed resulting in the amphiphilic products of free fatty acids and monoglycerides. These components, together with bile salts, are responsible for the transport of lipids and poorly water soluble nutrients from the intestine into the circulatory system of the body. In this study we show that the self-assembly of digestion products from short, medium and long chain triglycerides (caprylic, lauric, and myristic) in combination with bile salt and phospholipid is highly pH responsive. Fatty acid within the mixed colloidal structures is mapped using a combination of neutron scattering with both solvent contrast variation and selective deuteration as well as synchrotron-based small angle X-ray scattering [1, 2]. Modeling of the scattering data shows transitions in size and shape of the micelles with a transfer of the fatty acid from the core of the micelles to the shell or into the bulk water upon increasing pH but to an extent that is chain-length dependent. The results help to understand the process of lipid digestion with a focus on colloidal structure formation and transformation for the delivery of triglyceride lipids and other hydrophobic functional molecules.

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[2] Salentinig S et al.: Langmuir 30 (2014) 7296-7303.

#### The nano-to meso-scale solid structure of edible fats as seen by USAXS: Static versus dynamics results

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Ultra small angle X-ray scattering (USAXS) is a novel in-situ technique that has been applied to soft material like edible fats only in the last two years. This technique allows the surfaces characterization of the primary crystalline nanoparticles (CNPs) and either the solid structure, or the oil-filled-nanospaces, on spatial scales of a few nanometers to a few micrometers. This work presents the findings obtained for different edible fat systems strategically chosen to contain particular amounts of tri-, di- and mono-saturated fats in tri-unsaturated oils. The results are compared to the model system of tristearin in triolein [1, 2]. In that work, six structural levels were identified for scattering pertaining to the range from angstroms to micrometers: polymorphism (Level 1, wide angle X-ray scattering), molecular aggregates (Level 2, small angle X-ray scattering) and larger-scale aggregation (Levels 3 to 6, USAXS). The USAXS experiments were carried out at the Advanced Photon Source (APS) at Argonne National Laboratories, USA. Here we concentrate on Levels 3 to 6 to identify the average radius of gyration of the CNPs and of their aggregates. The fractal dimensions of the CNP' surface and the mass fractal dimension, Dm, of the aggregates are also reported. The results for Dm are sample and processing conditions dependent but can be separated into four categories: (i) aggregation into long cylinders  $(D_m \sim 1)$ , (ii) aggregates that resemble DLCA ( $D_m \sim 1.7 - 1.8$ ), (iii) aggregates that resemble RLCA ( $D_m \sim 2.0 - 2.1$ ), and (iv) aggregates that formed open structures compared with DLCA but did not exhibit a uniform distribution in space ( $D_m \sim 2.2 - 2.8$ ). A MCR 302 Anton Parr rheometer was used to prepare sheared samples using concentric cylinders to control the cooling rate and the speed of stirring as the samples were crystallized from the melt. USAXS results for the static and the dynamic samples will be compared. The size of the CNPs, their surface fractality as well and the mass fractal dimension of the aggregates they form are reported. We will address the implications of these findings for the choice of healthy fat-replacers.

<sup>[1]</sup> Peyronel F, Ilavsky J, Mazzanti G, Marangoni AG, Pink DA: J. Appl. Phys. 114 (2013) 234902.

<sup>[2]</sup> Peyronel F, Ilavsky J, Pink DA, Marangoni AG: Lipid Technol. 26 (2014) 223.

#### Modelling shear and solid fat aggregation in edible oils

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Aggregation of solid triacylglycerol (TAG) fats in edible oils is important for determination of their functionality. At moderate to low solid fat content (SFC), large-scale aggregation begins with crystalline nanoplatelets (CNPs) [1]. Such systems have been modeled [2] and studied via Ultra Small Angle X-Ray Scattering (USAXS) [3] in the last three years in non-flowing liquid oils. Complex fluids such as edible oils, however, are inevitably sheared during processing and consumption. The effect of shearing on the formation of solid fat structures, although highly relevant, is thus far relatively unexplored theoretically. We have developed a model in order to predict the aggregation structures of solid fats immersed in liquid oil when a shear field is applied. CNPs were modeled as rigid planar arrays of spheres interacting with each other and with surrounding spheres representing the liquid oil. The model was simulated using Dissipative Particle Dynamics within the software packages ESPResSo and Fluidix(C). In addition, we modeled the system as before [2], but with an added shear field for which (liquid) oil hydrodynamics was excluded. Previously, systems of semi-solid fats without an applied shear had resulted in the self-assembly of linear stacks of CNPs, known as TAGwoods [2]. The results of this work suggest that in the presence of shear, these TAGwoods and their aggregates, if formed, exhibit a distorted (non-linear) shape as suggested by other observations. Predictions will be presented for outcomes of USAXS experiments.

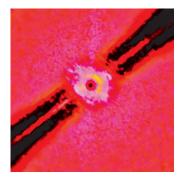
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# Rheological Modelling and Numerical Methods

#### A modified Giesekus model

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Shear banding is a ubiquitous phenomenon occurring in soft materials, including soaps, shampoos, pastes, gels, and food products. When undergoing strong shearing deformations, these materials can form two or more distinct bands of varying shear rate, known as shear bands. The Giesekus model has been widely used to predict the flow curves and velocity profiles of shear banding materials [1 - 3]. However, an unphysically large value of the solvent viscosity had to be used to capture the upturn of the shear stress at high shear rates. In this talk, we will present a modified version of the Giesekus model. The new model adequately predicts the nonlinear rheological behavior without the need for a large solvent contribution. Another advantage of the new model is that it accounts for diffusional effects leading to nonuniform concentration distributions. Like the original version of the Giesekus model, the new model can be derived using the generalized bracket approach [4] and thus satisfies the fundamental laws of thermodynamics. As the new model is still relatively simple, it can be adopted in simulations of real industrial flows.

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### Micromechanics of cheese sensory texture

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Cost price and caloric content drive cheese reformulation. This often leads to reduced firmness and increased rubberiness of the cheese, two sensory texture attributes that correlate well with intrinsic rheological material properties. The rational design of micro structures that overcome these texture deficits, requires constitutive models which handle large strains and variations in structure. Here we present such a model for semi-soft cheese, constructed through an hybrid-experimental computational approach and using Finite Element Analysis. Our micro structure model for the cheese is a filler composite. We show that the material response of the matrix of protein and water is best described by a two-term Mooney-Rivlin hyperelastic model combined with Prony series. The thermal induced softening of the cheese is well explained by the temperature sensitivity of the elastic modulus of the fat globules that fill up the matrix. Analysis of the deformed micro structure model, and the response of the matrix in a notched tensile test, reveals that the matrix fractures at the interface of two neighboring filler particles. This explains the observed plastic flow at low macroscopic strains in full fat cheese. Finally we demonstrate the use of our model as an engineering tool, for the design of alternative micro structures that represent a reformulated cheese that has a desired level of firmness without excessive rubberiness.

## Simulation of a liquid filament under Rayleigh breakup conditions applied to emulsion structures

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Complex fluid structures such as emulsions and multiple emulsions experience structure loss as a result of high shear stresses during spray processing. Previous experiments show that allowing the liquid filament to breakup under Rayleigh breakup conditions significantly reduces the structure loss in emulsions. In this presentation we demonstrate the resulting simulation of the Rayleigh breakup of a liquid filament under the stretching conditions existing in a pure centrifugal field. The stretching of the liquid filament caused by the centrifugal field causes a reduction in the filament diameter with respect to radial position, which in turn shortens the filament breakup length and reduces spray droplet size. From these results we determine the dependency of mean spray droplet size and droplet size distribution on operational parameters including rotational speed and nozzle exit velocity. The simulation results are then compared with the spray droplet distribution measured from a rotary laminar sprayer.

## Numerical investigation of in-nozzle drop breakup conditions for emulsion sprays

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A multiple or double emulsion is an immiscible liquid-in-liquid fluid system. The most common types of double emulsions are water-in-oil-in-water (W/O/W) emulsions and oil-in-waterin-oil (O/W/O) emulsions. Double emulsions have applications in the food and pharmaceutical industries where they are used in encapsulation processes for the controlled release of nutrients or drugs during digestion. However, these emulsions are known to be inherently unstable, due in part to coalescence and compositional ripening. In order to enhance the stability and shelf life of double emulsions, these fluid systems can be sprayed to produce powders. During such a spraying process, it is important that the structure of the emulsion be maintained, both inside the nozzle and during atomization and secondary drop breakup outside of the nozzle. In particular, the dispersed phase drops should not break up or coalesce since this change of structure would affect the material properties of the emulsion and the powder. The focus of this study is on the flow and drop behavior of emulsions inside a spraying nozzle. Specifically, numerical simulations are used to investigate breakup conditions of drops while an emulsion is within the nozzle. The simulations are performed by solving a two-phase flow problem in the nozzle in which individual drops are tracked through the flow field. The drops are assumed to be Newtonian, while the outer continuous phase is either Newtonian or non-Newtonian. The open source software, OpenFOAM, is used as a basis for the simulations. The numerical algorithm uses the finite volume method for solving the mass and momentum conservation equations and a volume-of-fluid approach for capturing the fluid-fluid interface. Dynamic meshing is used to maintain a sufficiently refined mesh around the drop interface as it moves through the flow field. From the simulations, the flow, material and drop conditions under which drops are broken up are investigated. Of particular interest is the minimum, or critical, drop size for breakup. The effect of the shear rates, capillary numbers, viscosity ratio, and fluid rheology (i.e., Newtonian or shear-thinning continuous phase viscosity) on critical drop sizes is determined.

#### Numerical correction factors of the Couette inverse problem in a Herschel-Bulkley fluid

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In the current research an approach based on Computational Fluid Dynamics (CFD) is applied to obtain the appropriate correction factors in Couette rheometery of a Herschel Buckley fluid. Since the frontal areas deliver their own portion to the torque measurement, two numerical correction factors (one for the yield stress part and the other for to the power-law part) are employed to calculate the actual fluid properties. Integration approach methods usually neglect the effects of the end parts on the inverse calculation of the flow properties. In the present investigation, using CFD simulation of the Couette flow in the rotational rheometer, more realistic correction factors are obtained for the flow curves. Aqueous starch solution (5 wt%) was used as the Herschel Bulkley fluid in the study. A mineral oil was selected as a Newtonian fluid to compare the results. CFD together with the integration approach was used to correct the measurement data and the calculated viscosity. The results of the wide and narrow gap rheometry were compared and the torque contribution for each part of the bob was specified. The aim of the present work is to apply a method, based on computational fluid dynamics (CFD), to find the correction factors that will in combination with the integration approach improve the accuracy of the experimental data measurements. Furthermore, we aim to quantify the torque contribution from the end surfaces and how this is influenced by the width of rheometer gap, yield stress and the power law index of the fluid. By the new approach, correction factors for the end parts was found for narrow and wide gap measurement data. The results show that comparing the integration approach, the correction factors to be larger for the wide gap. In addition, higher values of the correction factors were found for the non-Newtonian liquid. Applying the CFD correction approach highlights the end effects and precisely predict the fluid properties. The CFD approach is shown to be a useful tool to calculate the correction factors in Couette rheometery of Herschel Bulkley fluids.

### Simulations of peristaltic flows for Newtonian and non-Newtonian fluids

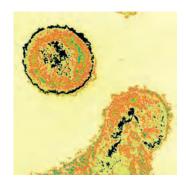
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Peristaltic motion is an important mechanism for the transport of fluids in biological systems, including in human bodies. It is the main method by which food is moved through the digestive tracts including the esophagus, the stomach and the small intestine. A peristaltic motion is a muscular constriction and relaxation of the vessel walls which induces a flow of the material inside. Such a motion can be simulated by a traveling wave which deforms the vessel walls accordingly and thus results in an overall forward motion of the inside fluid. In this study, peristaltic motion is simulated for Newtonian and Non-Newtonian fluids in deforming tubes. The computations are performed for two-dimensional planar and three-dimensional axisymmetric geometries. The twodimensional simulations correspond to a peristaltic motion in a channel, which in turn reflects a situation where a pair of cylindrical rollers are used to induce the peristaltic motion in a deformable tube. The three-dimensional axisymmetric simulations correspond to realistic tubular peristaltic flows as encountered in the small intestine. The contracting tube is modeled by means of a deforming mesh that corresponds to the traveling wave. The simulations were performed with a modified solver from the open source software package, OpenFOAM, to handle the moving mesh. The finite volume method is employed to solve the conservation equation of mass and momentum for velocity and pressure, where a Carreau-Yasuda viscosity law is used to model the non-Newtonian fluid. The influence of several material and geometrical parameters has been investigated. In particular, the effect of the traveling wave speed, the wave shape and the amount of deformation is examined in view of the systems transport efficiency. Also, the effects of a shear-thinning fluid is compared to Newtonian fluids. When available, comparisons with experimental data are made.







## Semi-Solid Foods

## Dynamic energy dissipation in protein-networks to determine the texture of food gels

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During oral processing of semi-solid (protein-based) food products entrainments speeds of 10 - 50 mm/s and contact pressures up to 20 kPa between tongue and palate are found. How the applied energy dissipates into the protein-network is critical for texture perception. The lack of a consistent description of this process makes it difficult to, for example, employ proteins from different sources or design alternative engineering routes in product (re-)formulation for the food industry. Dissipation of energy applied to a protein continuous network may occur via a number of mechanisms, like (micro)fracture, microstructural relaxation, friction caused by flow of entrapped serum or between microstructural elements, or by irreversible mechanical deformation of the material. Each of these contributions will reduce the stored (recoverable) energy and may be time- and strain-dependent. Each of the dissipation mechanisms is discussed and examples are shown for the impact on recoverable energy. To this end a variety of protein-based gels have derived that show structurally distinct network morphologies, and which are studied for their response to time-dependent (strain-/speed-)mechanical deformation. Typical findings are that the negligible contribution of stress relaxation from microstructural rearrangements in gelatin gels explains why this protein-network does not show a time-dependent behavior. Moreover, in caseinate gels the energy dissipation via relaxation processes does determine the measured RE at slow deformation speed. At faster deformation an additional contribution to the dissipated energy becomes apparent, that might point to the role of dissipation related to friction of the serum entrapped by the protein-network. It is shown in pea protein gels that either mesh size of the protein-continuous phase or water mobility within the pores of the network can be used to control the effect of this latter contribution. The role of microstructural friction is demonstrated for densely packed egg white protein gels. The inherent differences in mechanical responses between storage proteins from different plant species (pea versus soy) is highlighted. The occurrence of (micro-)fracture events is suggested to be the most relevant contribution to energy dissipation lowering the recoverable energy. This work illustrates how engineering of different network properties like porosity, crack stopper/initiators, serum viscosity, allows one to balance the different dissipation contributions and thereby the recoverable energy.

### Tribological properties of microparticulated whey protein as a fat replacer in liquid and semi-solid model foods

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The current understanding of the mechanisms underlying the fat mimicking properties of fat replacers such as microparticulated whey protein (MWP) is limited. MWP is known to provide fat-related mouth-feel in a limited range of foods, such as yoghurts and cheeses. Rheological and tribological properties are well known to contribute to the perception of fat related attributes. The aim of this study was to investigate the tribological and rheological properties of MWP in liquid and semi-solid model foods to reveal the mechanism underlying the fat mimicking properties of MWP. A mouth-mimicking tribometer was used to determine the tribological properties of MWP in four matrices: liquid (MWP in water; MWP in o/w emulsions) and semi-solid model foods (MWP in gelatin gels; MWP in emulsion-filled gels). In liquids, increasing the concentration of MWP from 0.1 to 8 % significantly reduced the friction coefficient. The reduction in friction coefficient with increasing MWP concentration was accompanied by an increase of viscosity. After considering the impact of viscosity on lubrication, a significant reduction of friction coefficient with increasing MWP remained. This suggests that the decrease of friction coefficient is not only caused by the increase of viscosity but probably by a ball-bearing lubricating effect of MWP due to its spherical shape and small size. In semi-solid gelatin gels, the addition of MWP reduced the friction coefficient of broken-down gels, but to a smaller extent compared to liquid foods, probably due to the presence of semi-solid gel particles. In emulsion-filled gels, addition of MWP reduced the friction coefficient. The binding properties of fat droplets to the gel matrix (bound/unbound) also influenced the frictional properties, which lead to very complex relationships between composition and frictional behavior. We conclude that the addition of MWP decreased the friction coefficient of liquid and semi-solid foods probably due to a ball-bearing mechanism. Bulk properties of the foods, especially the oral breakdown behavior of gels, additionally influence the frictional properties of MWP containing gels.

#### Modulating state transitions and visco-elastic behavior of zein resins by plasticizers and co-proteins interactions

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It is generally known that the production of gluten-free leavened products is technologically challenging, as gluten have unique viscoelastic properties giving wheat flour its excellent bread-making functionality. Similar viscoelastic properties have been observed only in maize zein above its glass transition temperature. However, manipulation and optimization of the zein performance is needed. The applicability of zein in bakery products might be improved by interactions with a plasticizer and a co-protein. By applying different levels of oleic acid and casein to zein resins, this study aims at elucidating the mechanism of both interaction types and the relation among functional (i.e. rheological behavior at small and large deformations), material (i.e. moisture sorption and glass-rubber transitions), and structural properties (protein secondary structural features) of viscoelastic zein systems. Plasticizing the zein resins with oleic acid resulted in reduced water absorption and glass transition temperatures and formed low elasticity/high extensibility resins. In contrast, interaction with casein increased water absorption and glass transition temperatures and imparted a four-fold increase in material strength/elasticity, as compared to zein alone. Plasticizers and co-proteins influenced zein secondary structure in the resin systems by decreasing and increasing low-frequency  $\beta$ -sheet structures (1640 - 1615 1/cm), respectively. Overall, glass-rubber transitions and secondary structure were found to be inter-related and together controlled the mechanical properties of the resins. The results indicated that zein properties can be tailored for optimal performance in a variety of applications where rheological properties and glass transition behavior are critical to product performance by interactions with casein and oleic acid.

### Measurement of pH micro-heterogeneity in cheese matrices by fluorescence lifetime imaging

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Cheese, a product of microbial fermentation, may be defined as a protein matrix entrapping fat, moisture, minerals and solutes as well as dispersed bacterial colonies. The growth and physiology of bacterial cells in these colonies may be influenced by the microenvironment around the colony, or alternatively the cells within the colony may modify the microenvironment due to their metabolic activity. The aim of this study is to assess the potential of C-SNARF-4 and Oregon Green 488 fluorescent dyes as pH indicators in cheese at the microscale and extend the useful pH range of the probes using lifetime measurements. To our knowledge it is the first time when the advanced microscopic technique fluorescent lifetime imaging microscopy (FLIM) of C-SNARF-4 and Oregon Green 488 were used to determine the local pH of different types of natural cheese. The pH of cheese samples was monitored both at macroscopic scale and at microscopic scale, using a non-destructive microscopic technique employing C-SNARF-4 and Oregon Green 488 fluorescent probes. Oregon Green 488 exhibits higher photostability and lower pKa, making it a useful pH indicator in the weakly acidic range (pH 4 - 6). Bioconjugate prepared from Oregon Green 488, dextran, has the advantage of high stability and low affinity to cheese matrix components (proteins). Employing fluorescence lifetime measurements we have extended the useful pH range of the probe towards more acidic values. Besides fluorescence intensity, fluorescence lifetime (a probabilistic timescale of fluorescence emission) may provide additional information about the chemical environment (e.g. pH) of the probe [1]. We conclude from our measurement that C-SNARF-4 is not sensitive enough to detect pH differences in cheese in the pH range 4.99 - 5.2. Also, its high affinity towards the cheese matrix makes the pH calibration challenging and less reliable. We suggest the use of Oregon Green 488 fluorescent dye to measure the pH of cheese matrix. We show that its fluorescence lifetime is a sensitive indicator of pH in the range from pH 3 to 6 which is especially which us especially useful for many natural cheese types including cheddar.

[1] Szmacinski H, Lakowicz JR: Anal. Chem. 65 (1993) 1668-74.

## Particle interactions and structure development in high-protein bars

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The use of milk-derived, protein powders in high-protein bars has grown significantly in recent years. Protein bars are not in physico-chemical equilibrium, however, and are prone to deteriorative textural change (hardening) during storage. The mechanisms responsible for hardening have not yet been fully elucidated. The purpose of this study was to explore the relationships between moisture sorption behavior, particle packing, the liquid-solid transition and aging in powder-based, protein bar matrices. The stability of a whey protein isolate (WPI) powder was compared with that of three WPI hydrolysate powders, varying in degree of protein hydrolysis. The onset of solidity, defined by the frequency-dependent crossover of viscous and elastic moduli, was dependent on volume fraction and a particle interaction energy. Hydrolyzed protein bars did not harden to the same extent as those containing intact WPI. The liquid-solid boundary occurred at volume fractions of 0.73 for WPI and ca. 0.55 for two of the hydrolysates. The most extensively hydrolyzed whey powder did not exhibit an equivalent viscoelastic transition. Hardening was less extensive in systems in which the onset of solidity occurred at low volume fraction. The relationships observed between initial viscoelastic behavior and subsequent hardening, were supported by particle size analysis, glasstransition phenomena, surface energy analysis and microscopy. Rheological characterization of the liquid-solid boundary, as a function of particle interaction, provides a means by which structural development in concentrated food systems can be better understood.

### Pathway of lipid molecules in fat-based food products

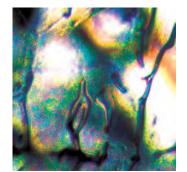
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The objective of the current project is to obtain a deeper understanding of the driving forces for migration and the preferred pathways of molecular migration in multicomponent food materials, which consist of particles dispersed in a continuous fat phase. The understanding of the mechanisms is the basis for controlling undesired migration leading to major quality issues such as fat blooming of chocolate, which is the number one consumer complaint in the confectionery industry resulting in large sales losses. Migration pathways and the driving force on different scale levels were investigated. Small angle X-ray scattering (SAXS) delivered information about the crystal configuration and migration of oil into pores. The microstructure was analyzed and migration visualized employing light microscopy and  $\mu$ -tomography. Surface roughness, which is indicative of fat blooming, was measured and correlations with lipid migration investigated. Furthermore, wetting behavior was analyzed through contact angle measurements. Thereby, the influence of composition and the nature of embedded particles, chemical composition and content of fat phase and storage conditions on fat migration were examined.







## Biopolymer Solutions and Gels

## Oleogels of canola oil with candelilla wax: Rheological characterization and utilization as a shortening replacer

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An oil-structuring technique called organogelation has been receiving great attention in a variety of scientific areas. Through this organogelation, organic liquid can be entrapped in a thermoreversible and three-dimensional gel network, producing oleogels with solid-like properties. In this study, the olegoels of canola oil with candelilla wax were prepared and their rheological properties were characterized. In addition, they were utilized as a shortening replacer in baked goods. The canola oil in a liquid form was transformed into the translucent and firm oleogels at the two levels (3 and 6 %) of cadellila wax. The viscosity of the oleogels had a tendency to decrease in a non-linear way with increasing temperature. The viscosity curves were well-fitted into the Arrhenius equation and the oleogels exhibited higher values of the activation energy compared to the shortening. While the cookies prepared with shortening contained a high level of saturated fatty acids, the oleogel cookies are rich in unsaturated fatty acids (more than 90 %). Moreover, the low viscosity of the oleogels at the baking temperature imparted desirable spreadable characteristics to the cookies. When the cookies were subjected to a three-point bending test, the greater snapping force was needed to fracture the control cookies with shortening whereas the oleogel-incorporated cookies had lower values of snapping force. This study showed that the oleogels of canolalil with cadellila wax were successfully utilized to replace shortening in cookies without quality loss. The extensive use of oleogels will contribute to the reduction of saturated fatty acids and elimination of trans-fatty acids from the diet, consequently providing beneficial health effects.

## Passive microrheology on biopolymers – structure, gel point and applications

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Passive microrheology based on Diffusing Wave Spectroscopy is a new innovative technique for the study of the viscoelastic properties and the gel point transition of polymers and hydrogels. Passive microrheology consists of using micron sized particles to measure the local deformation of a sample resulting from the thermal energy, that is to say the Brownian motion. DWS is an extension of the DLS technique to opaque media. The determination of the Mean Square Displacement (MSD) curve enables the characterization of the viscoelastic properties of a sample. The technique allows to measure particles displacement, which is directly related to the sample's viscosity and elasticity. Biopolymers have been introduced to a large scale of formulation not only in the food sector. Their unique properties in stabilization, structuration as well as texturation made them to extensively used components in formulations in food, pharmaceutical and cosmetically products. Especially the stabilization capacity for emulsions and formulation is of outmost interest. This work presents several examples how passive microrheology can be used for the characterization of formulations using biopolymers. Information about elasticity and viscosity can be obtained as well as the gel point for gelling systems. The Time-Cure superposition (TCS) method [1 - 4] allows the precise determination of the gel point according to the Winter-Chambon-Criterion. This method can applied on gelations depending on time, temperature and concentration (e.g. gelling agent concentration).

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### Setting the conditions for protein exchangeability by the energy balance and mechanical responses

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The understanding of (oral) breakdown properties or macroscopic functionalities such as water holding capacity is essential in relation to sensory properties. During (oral) processing, applied force can either be stored or dissipated via a number of physical processes. The balance between dissipation modes as fracture events and flow of water through or from the gel during deformation, and the amount of energy stored was compared for plant proteins (soy, pea) and animal proteins (whey, plasma, egg white, casein). Fracture properties (fracture stress and strain, Young's modulus), water holding (WH) and recoverable energy (RE, as a measure for the amount of energy stored) were determined for protein gels with varying morphology (CLSM, SEM). Whereas plant proteins typically have low fracture stress/Young's modulus and a wide range in RE, animal proteins are limited in variation in RE but show high fracture stress/Young's modulus. In general, the occurrence of fracture events results in low values for RE. To facilitate protein exchangeability it is essential to bridge the gap in mechanical responses between plant proteins and animal proteins. Selectively mixing of plant proteins with other proteins, or tuning the processing conditions were amongst others identified as applicable strategies to modulate the mechanical responses of the gels formed. We illustrate this with findings on how to direct fracture behavior and other energy dissipation mechanisms for various protein systems and mixtures thereof. Aggregation behavior and network morphology are investigated in order to explain changes in mechanical properties. Insights from this study open up opportunities for broader applicability of plant proteins and provide the next step towards protein exchangeability.

## Retention of esters by gellan and pectin solutions or their mixtures

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The retention of three isomer volatiles (ethyl butyrate, butyl acetate, isobutyl acetate) by gellan and pectin solutions and their mixtures was studied by static headspace gas chromatography. According to our findings, the retention of ethyl butyrate was greater for pectin concentrations of 0.25 - 0.75 %wt and gellan concentrations of 0.25 and 2.0 %wt. As the concentration of the mixtures increased, its release increased. Regarding isobutyl acetate, its release decreased with increased mixture concentration as well as at the three higher gellan concentrations. Not a clear trend could be detected in the case of pectin matrices. Butyl acetate showed decreased partition coefficient values over concentration in all types and matrices. In addition, for all studied matrices, isobutyl acetate showed the greatest partition coefficient values, followed by butyl acetate suggesting that their release was affected by their structural characteristics. According to literature, when studying the retention of a certain volatile from a carbohydrate solution, the factors that can explain the observed behaviour are the viscosity of the solution and the matrix-volatile interactions. Thus, flow curves (i.e. viscosity over shear rate graphs) for all studied solutions were constructed. According to those, as expected, viscosity increased with concentration. In addition, only the 2 %wt gellan solution exhibited shear thinning behaviour whereas the remaining solutions were Newtonians. Gellan solutions were the more viscous ones, followed by the mixtures. Furthermore, the addition of the second biopolymer increased the viscosity of the resulted mixture. If viscosity is the primary factor in aroma retention, its increase is expected with increased viscosity. In good agreement with that, volatile release for most of the samples studied in the present work was governed by viscosity. However, the study of the release of all esters from matrices sharing the same viscosity revealed that volatile retention due to specific interactions was also of great importance. Thus, for the same volatile, the type and the concentration of the biopolymer was significant and not the physical properties of the thickened system.

## Rheological and structural properties of heat induced mixed globular proteins

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In the recent years there has been a growing interest in designing food products based on mixed proteins, especially mixtures of animal and plant proteins due to its exceptionally high nutritional and health benefits. However, (partially) replacing animal proteins with plant proteins is a challenge due to the negative effect on texture and sensory properties, which is less desirable. Therefore, understanding of the mechanical properties and microstructure of mixed protein systems is highly required. In the present research, we investigated the rheological properties and microstructure of mixed systems of globular proteins: whey proteins/soy proteins (WP/SP), at different ratios. To date, very little research has been done specifically on these systems. By combining small deformation, large deformation, light scattering and confocal microscopy measurements we gain insight about the evolution of the protein network, protein aggregation, fracture properties and microstructure of both single and mixed systems of WP and SP. We observed a positive correlation of gel strength, stiffness and deformability to the fraction of whey protein in the mixed protein gel. Furthermore, we also looked at the correlation of ability of whey/soy mixed gel to retain water (water holding capacity) to the non-dissipated energy (recoverable energy), which represents the energy that is elastically stored during deformation, to better understand the sensory attributes, like crumbling effort and spread-ability of the gel. This study provides insights on how selectively mixing of proteins may lead to the design of new textures with controllable sensory properties.

# Effects of whey protein particles on rheological and microstructural characteristics of an acidified milk model system

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Microparticulated whey protein (MWP) are colloidal particles usually formed by combined heating and shearing of whey protein concentrates, typically have particle sizes ranging from 1.0 to 10  $\mu$ m. Nanoparticulated whey protein (NWP), on the other hand, is also produced from whey protein but have a smaller particle size (100 to 990 nm). In this research MWP and NWP were added to non-fat milk model systems and subjected to pilot scale homogenization (20 MPa) and heat treatment (90°C, 5 mins). The model systems contained 5 % protein in total and were made at two levels of casein (2.5 and 3.5 %wt protein based on micellar casein isolate powder). Samples with added whey protein ingredients were compared to 4 references: 5 %wt MWP, NWP or casein and a sample with 2.5 %wt casein and 2.5 %wt whey protein isolate (WPI). The model systems were characterized in terms of particle size, rheology and confocal laser scanning microscopy (CLSM) during and after acidification by glucono-delta-lactone. Following heat treatment, the particle size of the MWP systems showed no significant increase. This was also the case for the systems with NWP at a high casein level, while the particle size increased significantly after heat treatment in samples with NWP at a lower level of casein. Compared with the MWP systems, the model systems with NWP provided acid gels with higher firmness and viscosity, faster gelation and lower syneresis. CLSM revealed that the gelled model milk systems with NWP exhibited a dense protein structure and low amount of serum separation. On the contrary, MWP resulted in a protein network with low connectivity and numerous serum pores in the resulting gels. In conclusion, this study shows that NWP seem to play an important role in structure formation of acid model milk gels through efficient interaction with other proteins, while the added MWP were not able to become efficiently integrated as part of the gel network in the model systems.

## The effect of surfactant addition on the rheology behavior of ethylcellulose oleogels

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The use of oleogels as a way to structure liquid oil has been examined extensively in the last decade. Ethyl-cellulose-structured oleogels have recently been shown to have a wide range of applications in food products, cosmetics and pharmaceuticals. The current research aims to characterize the effect of surfactant addition on the rheological behavior of ethyl-cellulose/canola oil oleogel systems. Four different surfactants were studied having similarity in their chemical structure, Sorbitan monostearate (SMS), Sorbitan monooleate (SMO), Glycerol monostearate (GMS), and Glycerol monooleate (GMO). The surfactants were chosen in order to examine the role of both the "head" and "tail" groups on the final gel properties. Gel point determination using frequency dependent rheological experiments at different temperatures showed a significant decrease in the gelation temperature due to surfactant addition, as compared to the native oleogel. Temperature sweep experiments demonstrated a stepwise sol-gel transition with the addition of surfactant, suggesting a structure formation during gelation. Mechanical strength analysis showed significant increase in gel hardness due to surfactant addition when compared to the native oleogel. Significantly stronger gels were obtained with the addition of GMS, while SMS, SMO, and GMO exhibited similar gel strength. Thermal analysis done on the GMS/EC oleogels illustrated a shift and a split in the GMS crystallization peak, suggesting interactions between the EC backbone and the GMS molecules. The effect of the surfactant chemistry seems to be a complex interplay between the role of the "head" and "tail" groups on the oleogel properties and therefore requires a further investigation. These results provide a first insight to the mechanism of ethyl cellulose/surfactant gelation process which promotes gel mechanical properties. Such insight could potentially contribute to the understanding of the effect of gel composition on its properties which is crucial to further applications development.

### Protein aggregation in the presence of cellulose

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There is an on-going interest in the development of new functional food ingredients that can be used to modify the textural properties of food products. Foods are complex systems that are typically structured on a mesoscopic length scale. These structures eventually determine i.e. the stability, texture, appearance, flavor release and bio-availability of the different components. By their capacity to form different structures, proteins play an important role in texturing food products. The formed structures vary from random to linear aggregates, depending on the conditions used. These protein aggregates by themselves or in combination with polysaccharides are used in many food products. However, the formation of protein aggregates and the physical properties of the final product, can be influenced by the presence of polysaccharides. Here we study the influence of cellulose fibrils on the formation of protein aggregates and on the physical properties of the mixture at different conditions. The rheological properties and microstructure of mixed systems, containing both protein aggregates and cellulose fibrils, are characterized.

### Interactions between different milk protein ingredients and exopolysaccharide-producing cultures and their effects on microstructure and textural properties of yogurt

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The textural properties of fermented milk are the major attributes of final product quality. During yogurt manufacturing, the texture is usually controlled by adding protein ingredients and/or use of exopolysaccharides (EPS) producing starter cultures. Little, however, is known about the interactions between these two components and hence the present study was undertaken. Eighteen yogurt systems, differing in protein ingredients added, caseins or different whey proteins, and EPS-producing cultures, were manufactured at pilot plant scale. Physical stability (grain formation, syneresis, water holding capacity), as well as textural properties (viscosity, firmness, cohesiveness), and microstructure of the yogurts was studied. Microstructure was investigated by confocal laser scanning microscopy, in addition particle size distribution and intramolecular strength were studied by light scattering and rheology techniques, respectively. Image analysis was performed to quantify the amount of grains, the amount of EPS and their interactions with proteins. Denaturated whey proteins and negatively charged EPS formed a strong network, causing high firmness and viscosity, low syneresis, and low amount of grains. The presence of whey proteins resulted in yogurt with high amount of grains. However, the amount of grains was reduced by presence of EPS. The effect of EPS on graininess was negligible in presence of high lactose level. The added caseins had a negligible effect on the textural properties. Textural properties and physical stability of yogurt were shown to be improved by a combination of added whey proteins and using EPS-producing culture.

### The effect of hydrocolloids on rheological properties of food matrices containing emulsified fat droplet and starch gel

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In this study, model food matrices containing rice starch gel and protein-coated fat droplets were prepared and the effect of the addition of hydrocolloids with different properties on the rheological properties of model food matrices was examined. Non-ionic (guar gum) and ionic (xanthan gum and  $\kappa$ -carrageenan) hydrocolloids was used. Dynamic viscoelastic tests revealed that the overall rheological properties of model food matrices were less affected by the presence of protein-coated fat droplets, while they were largely affected by the addition of the hydrocolloids depending on the type of hydrocolloids. The addition of xanthan had a more pronounced effect on improving structure formation of food matrices than other hydrocolloids. The addition of hydrocolloids mixtures (guar-xanthan, guar- $\kappa$ -carrageenan, and xanthan- $\kappa$ -carrageenan) provided further modification of rheological properties. These results would be helpful for achieving the food matrices with tailored properties.

### Physical and rheological study of frozen aqueous solutions containing food biopolymers

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The development of mixed systems, formed by locust bean gum (LBG), acacia gum (AG) and xanthan gum (XG) can offer new interesting applications such as the development of frozen products, due to their interactions. Food hydrocolloids (biopolymers) in the frozen phase influence frozen systems by affecting ice crystals size and morphology. The rate of crystallization as affected by food hydrocolloids depends on the type of food hydrocolloid used [1]. The knowledge of the viscosity and other rheological properties is essential for the products quality and freezing process, as ice crystal features determines the latter. Therefore, the aim of the work is to investigate the rheological behaviour of selected polysaccharides in a food model solution during the freezing process. The polysaccharide mixtures were prepared by mixing appropriate amounts of LBG, AG and XG to achieve different polysaccharide concentrations (w/w). These preparations were added to a model solution of acidified sucrose for further characterization. DLS (Dynamic Light Scattering) was used to characterize the polysaccharides in solution. Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC) and Fourier-transform infrared (FTIR) spectroscopic techniques were used to highlight the interactions between the three polysaccharides. The polysaccharide solutions were submitted to different temperatures (from ambient temperature to freezing temperatures) and rheological measurements were performed using a Brookfield viscometer, with a small sample adapter accessory with a refrigerated water bath. Optical microscopy was used to visualize the size and morphology of ice-crystals and DSC to study phase transitions and the crystallinity of solutions.

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## Tribological and rheological properties of acidified milk drinks prepared with different stabilizers

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Acidified milk drinks (AMD's) is a worldwide market where an increasing growth is seen. AMDs are popular in Asia, but there is also an increasing AMD market in the rest of the world. When producing AMDs it is necessary to add a stabilizer to prevent or reduce whey formation and sedimentation of casein micelles. In acidified milk systems stabilized with pectin less than 20 % of the pectin is directly interacting with casein micelles. The remaining 80% is involved in a network with casein/pectin complexes [1]. When using CMC to stabilize the drinks the CMC adsorbs to the casein micelle at pH below 5.2 and stabilization occurs by steric stabilizing rather than electrostatic repulsion. Furthermore, non-adsorbed CMC increases the viscosity of the serum [2]. In the present work different AMDs were prepared by adding pectin, CMC or a blend of CMC and Gellan gum to stabilize the drink. The effect of changing the stabilizer in AMD has an impact on both viscosity and friction coefficient. It is evident that using pectin for stabilizing the milk drink results in a low viscosity, and a blend of Gellan and CMC results in a very shear dependent viscosity. In general, different tribological and rheological profiles are observed whether pectin, CMC or a blend of CMC and Gellan is used to stabilize AMDs.

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### A rheological study of the temperature dependence of the gel-sol and sol-gel transition in gelatin-water systems

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Gelatin is a protein based biomaterial used in various industries. In this talk, we will discuss the effects of the cooling/heating conditions on the transition of aqueous gelatin solutions to gels and vice versa. Gels and solutions of 2 and 4 %wt gelatin in deionized water were probed under oscillatory measurements. For each concentration, temperature ramps between temperatures of 37.5°C to 4°C were implemented. The storage and loss moduli exhibited temperature loops. In all cases the melting temperature Tm was higher than the gelation temperature  $T_q$ . The  $T_q$  for 4 %wt gelatin was about 24.5°C, while for 2 %wt was 20°C. A smaller temperature difference between  $T_m$  and  $T_q$ , namely of about 10°C, was observed for the higher concentration. For the sol-gel transition, we observed a sharp increase of G' versus 1/T after  $T_q$  followed by a straight line. The behavior is typical for a hierarchically organized self-assembled microstructure. At the earliest nucleation stages, we believe that only isolated triple helices are present. As the energy barrier is low at a further temperature decrease, these basic building blocks form spontaneously protein assemblies and finally a rigid structure. The concentration is an important parameter since higher concentration means more basic units per unit volume of the material. In contrary, in case of the gel-sol transition two distinguished regions with significant small slopes confine a narrow range of temperatures at which the linear correlation of the G' versus 1/T is recorded. The first small slope region indicates only randomly untied bonds of the large scale structure of the gel. As the temperature increases, the available energy is sufficient to break the hydrogen bonds to a greater extent. Once the bundles of triple helices are loose and isolated, the system becomes a solution upon further increase of the temperature. In conclusion, the elastic modulus is a parameter directly related to the basic structure of gelatin, namely the triple helices. The time/temperature evolution of G' gives valuable insights into the kinetics of these transitions.

## Ultrasound homogenization to alter the physical properties of bacterial cellulose aqueous suspensions

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Cellulose derivatives, obtained from sources such as wood and cotton, are well known in the food industry for their stabilizing and thickening properties. Recently, bacterial cellulose (BC) has received attention as well. Although BC and plant derived cellulose have the same chemical structure, BC has distinctive advantages such as high mechanical strength, low density, high water holding capacity, high crystallinity and high purity. Thanks to these advantages, BC is currently been used in many areas such as biomedicine, cosmetics, electronics, the paper industry and many others. Komagataeibacter sucrofermentans DSM 15973 is one of the bacteria species that has the ability to synthesize cellulose. However, in the form of pellicle that is produced, it is not possible to be efficiently added in food models (i.e. emulsions). Hence, in the present study, purified wet bacterial cellulose was disrupted into cellulose aqueous suspensions (0.1 -1 %wt) and homogenized by a 12 kHz ultrasound device for 0, 1, 3, and 5 minutes, respectively. In order to evaluate the effects of ultrasonication (US) on bacterial cellulose aqueous suspensions, morphology, rheology, phase separation, water holding capacity (WHC) and charge were investigated. Morphological analysis by TEM revealed changes in microstructure and dispersion of cellulose ribbons after US treatment. The diameter of the ribbons decreased significantly with increasing the processing time. The rheological behavior of the BC aqueous suspensions showed two shear thinning events in between which the viscosity remained constant. The size of the Newtonian plateau region increased with increasing BC concentration. Furthermore, phase separation of the suspensions was decreased after US treatment. Hence, ultrasound is an efficient homogenization method to improve the physical properties of BC aqueous suspensions.

## Microstructural and mechanical properties of alginate gels containing parallel aligned capillaries

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Alginate is a readily available anionic polysaccharide, which finds commercial use in diverse areas such as wound care, pharmaceutics and food [1]. The industrial interest of alginate originates partly from its combination of being biocompatible and ability to gel under mild conditions making it a suitable candidate for several biomedical applications [2]. Ionotropic gels are rapidly formed in the presence of di- and tri-valent ions, which are chelated between negatively charged guluronate units belonging to different alginate chains [3]. The ionotropic gels formed upon the so called "internal setting" have been extensively studied with regards to network formation mechanism, mechanical properties and microstructural characterization. The ability to form alginate gel structures containing parallel aligned channels were discovered during the 1960's using directed external source of crosslinking ions [4]. The macroporous and hierarchically formed alginate structure obtained via the directed external gelation method attracted some interest during the 1960's and 1990's when the origin of the striking channels was studied. Lately, the interest for these spontaneously formed macroporous structures have increased due to their potential application as scaffold for cell in-growth e.g muscle cells. Few studies have focused on the mass transport and mechanical properties of the alginate gels containing capillaries. Here, we report on the rheological and mass transport properties of the alginate gels containing capillaries and compare these to internally set alginate gels. The microstructure of the capillary gel is studied by small angle X-ray scattering and electron transmission microscopy. Correlation between the microstructure and rheological properties of the capillary gel is made.

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### Preparation and rheological characterization of food grade gellan hydrogels with calcium ions as gelling agent

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Hydrogels are networks of hydrophilic polymer chains that are highly valued for their functionality and ability to tune physical properties in many industrial applications. Biopolymer-based hydrogels are relatively safe for applications in variety of industries for rheology control, encapsulation and targeted drug delivery due to their biocompatibility and nontoxic nature. Gellan is a bacteria exopolysaccharide capable of imbibing high volumes of water and swelling to form hydrogels with varying properties depending on the production method. In this work, gellan hydrogels were prepared and characterized by determining their rheological properties. The hydrogels were produced by ionotropic gelation of gellan solution using calcium ions as gelling agent under high shear rate (with an UltraTurrax T.25 basic IKA-Werke) as proposed by Joice Aline Pires Vilela [1]. The rheological properties were characterized by performing amplitude sweeps, oscillatory frequency sweeps, yield point testing and shear rate sweeps with a modular advanced rheometer system (HAAKE-MARS) at 25oC. The gelation point was determined and the rheological properties showed a non-Newtonian behavior with increase in gel strength as the quantity of calcium ions increased at constant gellan concentration but beyond a certain maximum Ca2+/gellan ratio, there was no further increase in gel strength conforming to hypothesis by Pérez-Campos [2]. The hydrogels showed higher rheological properties (storage modulus and Yield point values) with an increase in the shear rate of the unltraturax. The gelation of gellan occurs at remarkably low concentrations compared to other polysaccharides making the system very favorable for use in food and cosmetic applications.

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## Application of bagasse cellulose nanofibers to make nanocomposite film for food packaging

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All Cellulose Nanocomposite film was produced from sugarcane bagasse nanofibers. N,N-dimethylacetamide/lithium chloride was used as solvent. The average diameter of bagasse fibers (14  $\mu$ m) was downsized to 39 nm after disk grinding process. X-ray diffraction showed that apparent crystallinity and crystallite size decreased relatively to an increased duration of dissolution time. Thermogravimetric analysis confirmed that thermal stability of the ACNC was slightly less than that of the pure cellulose nanofiber sheet. Tensile strength of the fiber sheet, nanofiber sheet and ACNC prepared with 10 min dissolution time were 8, 101 and 140 MPa, respectively. Water vapor permeability (WVP) of the ACNC film increased relatively to an increased duration of dissolution time. ACNC can be considered as a multi-performance material with potential for application in cellulose-based food packaging owing certain functional properties (tough, bio-based, biodegradable and acceptable levels of WVP).

### Rheological properties of mucilage aqueous solutions (Opuntia ficus indica L. Mill)

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The mucilage is a complex polysaccharide present in the stems of Opuntia ficus indica (L. Mill) plants. The sample of mucilage was obtained by precipitation with ethyl alcohol from natural exudate of parenchyma tissue (MP). The aim of this work was to study the rheological properties of solutions mucilage as a function of mucilage concentration. MP solutions at all concentrations studied showed pseudoplastic behavior. An increase in viscosity with increasing concentration in the range of mucilage 0.5 to 4.5 % (w/v) was observed, accentuating the pseudoplastic behavior. Regarding the dynamic behavior solutions MP 0.5 and 1 %(w/v) showed a cross over where G' exceeds G''. The most concentrated systems (1.5 and 4.5 %(w/v)) showed macromolecular behavior concentrated solution with G' greater than G'' in all range of frequencies studied. Cox-Merz rule indicated macromolecular aggregation in the most concentrated mucilage solution (4.5 %(w/v)). The rotational and dynamic tests showed that the mucilage solutions have a similar behavior than other hydrocolloids used in the food industry. This new hydrocolloid showed similar guar gum thickening capacity, allowing its use in the food industry as a thickener and stabilizer agent.

### Study of the rheological behavior of mixed gels of carrageenan and goat milk whey protein concentrate

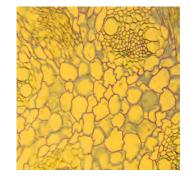
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The effect of the addition of 10 mM calcium chloride  $CaCl_2$  and heat treatment (90°C for 30 minutes) on the rheological properties of carrageenan and whey protein concentrate of goat milk mixed gels was analyzed. Gels at pH 7 were prepared at a concentration of 1 %(w/v) carrageenan and three protein concentrations 0.5, 1, and 3 %(w/v) corresponding to the following ratios of protein-polysaccharide: 1:2, 1:1, and 3:1. Gels were subjected to four types of treatments classified in: T1 (without  $CaCl_2$ , without heat treatment), T2 (with  $CaCl_2$ , without heat treatment), T3 (without  $CaCl_2$ , with heat treatment) and T4 (with  $CaCl_2$ , with heat treatment). The storage modulus G' and loss modulus G'' were obtained using dynamic rheology techniques. The hardness, elasticity, cohesiveness, adhesiveness, and water holding capacity of the gels were determined. Results show rheological behavior like gels. Protein concentration had no significant influence on the value of G' and G'', water holding capacity, modules or textural properties such as hardness and elasticity, unlike the type of treatment. In the protein-polysaccharide gels 1:1, the addition of  $CaCl_2$  was determinant in their properties. The products obtained could be utilized in the formulation of dairy desserts.









#### Effects of gel from ground chia (Salvia hispanica L.) on the rheological properties of wheat dough and bread

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The contribution presents the effects of gel from ground chia on the rheological properties of wheat dough and bread. For this purpose fundamental and dough rheological measurements were performed. All doughs were baked to analyze the baked products. The effects of addition of gel from ground chia on the process of staling of wheat bread were analyzed for 96 h through texture profile analysis. Altogether 5 doughs were prepared with 1 to 3 % ground chia (based on the amount of wheat flour) with 5 g/g and 10 g/g water forming the gel. Using a texture analyzer the maximum resistance of dough was measured and it decreases with increasing dough yield ( $R^2 = 0.98$ ). No significant differences in the gaseous release time were observed by rheo-fermentometer measurements, but an increased dough height, indicating a higher extensibility, with different amounts of chia. Frequency sweep measurements show decreasing values for the storage as well as the loss module with increasing chia gel content. Creep-recovery tests were fitted to the Burgers model and show a decreasing value for  $G_0$  a matter constant for the measured sample. Two samples behave in different experiments similar to each other: 2 % ground chia mixed with 5 g/g water and 1 % ground chia mixed with 10 g/g water. Both have the same amount of additional water that results in a dough yield of 170. The pasting properties were determined as well and with increasing chia content the viscosity of all wheat flour-chia suspensions is increased compared to the wheat flour suspension. Samples with 2 % ground chia mixed with 5 g/g water or 1 % ground chia mixed with 10 g/g water produced the best baking experiment results as the volume yield increased up to 24 % with respect to the standard wheat bread ( $R^2 = 0.94$ , excluding 3 % chia). With respect to storage the bread quality was improved as the crumb firmness measured with a texture analyzer was reduced up to 36 % compared to the breads without added chia gel.

### Influence of processing on the structure and function of wheat components

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Wheat and its flour have an essential role in human nutrition giving rise to numerous palatable and satisfying foods. Starch and proteins are the main components of flour and are detrimental for structure, functionality and quality of the final product. An in-depth understanding of process-induced structural changes is essential for future developments towards high quality good satisfying modern consumer demand. The impact of structural alterations in starch and protein were investigated from molecular- to macro-scale throughout the production line of bread. On the molecular level we focus on the structure of starch and gluten forming proteins. Analysis is performed via protein fractionation, starch damage essay as well as microscopic techniques. The interplay between starch and protein in dough samples is further investigated by rheological methods. Finally, the baking behavior and bread characteristics are analyzed to correlate micro to macro structural aspects. The established process-structure and structure-property relationships strongly prove that minor changes in milling conditions lead to alterations of the structure of starch and protein that result in major differences in the final product. A higher mechanical input results in a 3-fold increase in starch damage. This allows up to 20% increase in water adsorption which considerably prolongs product shelf-life. The protein network is also highly influenced by the milling procedure as suggested by the major observed differences in the specific volume of the bread.

### Cake ingredients and processing influence batter rheology, cake volume, and texture

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Batter ingredients and mixing process parameters determine the flow and viscoelastic properties of the batter. These structural changes impact the baking performance and thus the quality of the final baked product in terms of cake volume and texture. A complex experimental design was performed to study a cake batter system, using a limited number of observations. Flour type, leavening acid type and one process variable, namely mixing time, were varied. Two flour types (standard and pregelatinized flour) and three leavening acids with fast to slow action (Monocalcium Phosphate, Sodium Acid Pyrophosphate 28 and Sodium Acid Pyrophosphate 10) were used. Three responses were measured in this experiment: rheological properties of the batter, cake volume and cake texture. The flow and viscoelastic properties were obtained using rotational and oscillatory tests, respectively. The linear viscoelastic region was determined by an amplitude sweep for each batter and the apparent viscosity was determined as a function of shear rate. To study the properties of the batter during baking, a temperature sweep was performed. To characterize cake texture, hardness was calculated by Texture Profile Analysis. Long mixing times resulted in a decrease of both apparent viscosity and cake volume. In general, viscosity was positively correlated to cake volume. However, the replacement of standard flour with pregelatinized flour caused an increase of viscosity and a decrease of cake volume. This outcome was attributed to the larger elastic component of the viscoelastic behavior. Although large proportions of pregelatinized flour led to harder cakes, small proportions, improved the cake properties. Batter apparent viscosity decreased with large proportion of fast acting leavening acid. Moreover, the temperature sweep indicated that the batter involving a large proportion of slow acting acid set later at higher temperature during baking. Therefore, these cakes were able to rise for longer time, resulting in a higher cake volume.

#### Effect of blend levels on composite wheat doughs performance made from yam and cassava native starches and bread quality

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Due to the high cost, geographical scarcity and high demand of wheat flour, efforts are been directed toward the provision of alternative sources of flours, notably in tropical areas. However, composite wheat breads generally displayed reduction in loaf volume and impairment of sensory qualities (e.g. appearance, texture, and flavor), as the level of substitution of wheat with non wheat flour increased. Due to the fact that the main selection criteria of wheat are based on their ability to give bulky white breads, it appeared useful to seek products of substitution which had less negative impacts on the volume of the bread. The effects of refined wheat flour substitution with two native starches from yam tuber and cassava root, and two commercial products, a specialty starch, C\*Actistar and a wheat bran flour at 10, 20, 30, 40, and 60 % dry basis, on the rheological properties of dough and bread characteristics, have been examined. In general, during the mixing phase, the substitution of white wheat flour for starch or wheat bran flour had a tendency to modify the flour strength from strong to weak, depending on the nature of the added fraction and the level of substitution. Yam starch and wheat bran flour weakened dough strength to a lesser extent in comparison with cassava starch, and by far, the resistant starch, C\*Actistar. In addition, differences in dough expansion appeared among the botanical origins of composite dough and the blend proportions, during the fermentation phase. White wheat flour substitution for yam starch up to 30 % or cassava starch up to 20 % led to kinetics expansions of resulted doughs close to that of the control, while those of doughs containing C\*Actistar starch or wheat bran flour were significantly slower than that of the control, whatever the level of substitution. The baking phase showed that yam starch enriched breads from 10 to 40 % of substitution and cassava starch enriched breads from 10 to 30 % of substitution gave as bulky loaves as the refined wheat bread. Beyond these concentrations, the resulting breads were less voluminous. Hedonic tests revealed that, 30 % yam starch substitution and 20 % added cassava starch led to composite breads which met consumer satisfaction on all attributes, as the control.

### Rheological behavior of wheat and quinoa blend sourdough and its impact in texture of bread

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Sourdough is one of the oldest biotechnological processes applied in bread production to improve their nutritional and technological quality (as flavor, texture and leavened) as well as shelf life extension. Additionally, using a quinoa flour and wheat flour blend for the dough and bread making process, the protein quality, mineral bioavailability and bioactive compounds increase in the resulting product. The aim of this study was to characterize a wheat-quinoa sourdough fermented with a selected lactic acid bacterium (LAB) and evaluate the effect of the addition on the technological quality of bakery product. Blends for sourdough and bread manufacture were made with 20 %w/w of quinoa flour. Unfermented and wheat dough was used as control. The sourdough was tested in terms of acidification capacity and rheological behavior. The bread was prepared following the standard formulation replacing 20 % of the components with the sourdough and bread without fermented dough was included as control. Technological parameters as specific volume, crumb texture and colour were measured in both product. Although the doughs were prepared to get a yield of 200, showed clear differences in terms of the fundamental rheological properties. The value of the viscous G'', elastic G' and complex modulus  $G^*$  increased proportional to the angular frequency, behavior accentuated at higher frequencies, and in all cases, G' was higher than G". The highest magnitude of G\* was recorded in the blend dough unfermented. The sourdough showed a rheological behavior closer to wheat dough and G\* values were the lowest. An increase of 15 % in the specific volume was obtained in sourdough bread compared to the control. Evaluating texture variables, it was observed that sourdough bread had significantly lower hardness and chewiness values and higher resiliency than the control, desirable characteristics in fresh bread. In terms of appearance, it was observed that the inclusion of sourdough leads to an increase in crumb lightness and whiteness and decrease in yellowness and redness crumb. These results are promising for the development of a more natural, tasty and healthy bakery product with improved technological characteristics.







# Influence of Processing on Structure and Rheology

### Rehydration study of instant noodles: Texture versus thermal conductivity

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Instant noodles have been widely consumed throughout the world due to advantages such as cooking convenience, low cost, and a variety of flavors/tastes. A great deal of effort has been made to improve the quality attributes of instant noodles. Specifically, a shortened rehydration time is one of the critical attributes of instant noodles. There is however a lack of more objective and reliable studies on the rehydration of instant noodles. In this study, a novel thermal technology based on the modified transient plane source method was introduced to an instant noodle system in order to study the rehydration property of noodles. The thermal conductivity of the noodles was measured and correlated to their moisture content and extensional property during cooking. The thermal conductivity of the noodles had a tendency to increase with the increasing moisture content. Furthermore, there was a highly linear relationship between the extensional property of the noodles and their thermal conductivity depending on the moisture content. When the noodles were subjected to cooking in hot water, their thermal conductivity distinctly increased at the initial stage of the cooking and then reached a plateau. The change in the extensional property of the noodles during cooking favorably correlated to the pattern of their thermal conductivity, showing a linear correlation with a high coefficient of determination. Thus, the method applied in this study could be used as a new and promising approach to the thermal conductivity and rehydration research of instant noodles.

### Stabilization of açai juice using homogenization treatments

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Açai is an amazonian fruit very interesting for designing healthier products because of its high amount of vegetal oils, leading to a claim of high Omega-3, Omega-6 and Omega-9 contents. However, this high fat content might also be an issue for the formulation of aqueous products, like juices, because of two common destabilizations: decantation of cells and/or creaming of fat globules. The objective of this study was to investigate homogenization actions on the stabilization of açai juices without any additive or with 0.15 % xanthan. Two treatments were undertaken: shearing with Polytron (Fisher Scientific, Waltam, MA, USA), acting as laboratory equivalent of colloidal mill; and high pressure homogenization (HPH) after Polytron. A combination of several techniques was carried out, in order to obtain a multi-scale characterization of the final products, which could enlighten structure/texture/stability relationships. The kinetic stability of juices was monitored using Turbiscan (Formulaction, L'Union, France). For original juices without additive or treatment, a strong decantation appeared in the first 30 minutes, plus a creaming. Using Polytron homogenizer for 1 min at 103 rpm, the decantation was avoided but creaming was still occurring. HPH did not bring any more stability. On the contrary, while Polytron also allowed juices with xanthan to be more stable than control ones, HPH made a real difference by allowing these juices to be perfectly stable for 2 weeks, without any decantation or creaming. Therefore, while additives and strong treatments such as HPH definitively have a positive effect on stability, if one needs not to use any additive, the sole effect of colloidal mill on native juices should already help to preserve them from physical instabilities. The hypothesis made to explain these phenomena is that mechanical treatment is reducing the particles size, which can bond to each other, forming a weak gel (as underlined by viscoelastic measurements), so the juice is stabilized by its own particles. The addition of xanthan strongly increases the viscosity at low shear rate and seems to reinforce the particles network and its stability. A depletion-flocculation mechanism may be raised.

### How to enhance satiating ability: rheological and structural approaches related to oral processing of yogurt

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Protein is the most effective food macronutrient providing satiating effects. Thus, formulating dairy foods with increased protein content can raise its satiating capacity. In addition, longer residence time and more laborious handling in mouth would elicit higher expectations about satiating capacity of a food product. In this context, its rheology and structure should play an important role. In the present work, yogurts were prepared with double skimmed milk powder (MP) and whey protein (WP), as well as a control (C) without extra protein. Three more samples were prepared by adding 2 % of a physically modified starch to each (CS, MPS and WPS, respectively). Rheological tests were used to characterize the flow and viscoelastic properties of the samples before and after in vitro oral digestion, and their microstructure was observed by light microscopy. Before saliva treatment, MP showed denser areas than C owing to the extra milk protein. In WP, two protein networks could be distinguished: one with a rougher appearance corresponding to the milk protein network — as also observed in C and MP — and another corresponding to the whey protein. In the samples with added starch, starch granules were observed embedded in the protein networks. All the yogurts made with starch (CS, MPS, and WPS) showed higher viscosity than their counterparts without added starch, thus strengthening the protein network properties. After in vitro oral digestion the microstructure showed that the protein tended to aggregate and form some dense and opaque areas. The starch granules maintained their structure and were embedded in the continuous structure of the protein network, indicating that they were not broken down by the saliva due to its physical modification. The flow parameters and the viscoelastic moduli were higher in the samples containing starch than in the corresponding yogurts without starch indicating a reinforcement of the structure. The tan  $\delta$  values decreased significantly in the added-starch samples in comparison with their nostarch counterparts, indicating an increase in the relative contribution of the elastic component to the viscoelasticity of the system. This more solid-like behavior may be attributed to reinforcement by starch granules embedded in the protein network.

#### Powder-liquid mixing process for production of homogeneous food composite structures with low water or fat

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Mixing powder with a small fraction of liquid is a process step used in a wide range of industries. Adding the liquid homogeneously is however not an easy task due to liquid enriched lump formation and clogging. With conventional mixing techniques, de-agglomeration is necessary after the mixing either by mechanical treatment using "delumpers" or by adding more liquid. The high-energy input using mechanical treatment can result in the loss of functionality of sensitive materials, whereas the increase in water content mostly requires an additional time and energy consuming drying step to produce a low liquid content in the end product. A new method for adding liquid to powders has therefore been developed. The liquid is hereto transformed into a solid powder by spray chilling, forming so-called powder-liquids to be mixed with the bulk powder. The aim of this work was to evaluate the powder-liquid mixing process in comparison to conventional techniques, where the liquid is sprayed onto the powders during mixing. Fat and water based powder-liquids were produced and characterized in terms of particle size distribution, particle morphology and flow properties. The mixing quality was quantified by near infrared (NIR) spectroscopy, colorimetry and moisture analysis after various mixing times, using the empirical variance s2 as a measure of the homogeneity. The results demonstrated that the flow properties of powder-liquids, single and in mixtures, varied with the particle size, temperature and concentration. For the mixing trials, the powder-liquids mixed faster in comparison to the sprayed liquid. This was seen both for the fat and water based systems. For the lower concentrations, the powder-liquids also gave a better homogeneity of the mixture in the end.

### Redesigning cheese powder for omission of emulsifying salts

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Cheese powder is a food ingredient widely used as flavor and functional ingredient in products like snacks, soups, sauces etc. The present focus on decreasing the use of food additives as well as the general public intake of sodium and phosphate has created a need for development of cheese powders produced without addition of emulsifying salts. As in the production of processed cheese, the emulsifying salts aid in formation of a stable homogenous product, the cheese feed, which is subsequently spray dried into the final cheese powder. The rheological properties, centrifugation stability and microstructure, visualized by confocal laser scanning microscopy, of cheese feeds produced both with and without emulsifying salts have been investigated. When the emulsifying salt was removed from the cheese feed recipes substantial changes in rheology and microstructure were observed along with a marked decrease in centrifugation stability. Cheeses feeds containing emulsifying salt exhibited shear-thinning behavior and at the microstructural level a continuous protein network where the fat globules were entrapped. On the other hand, cheese feeds without emulsifying salt showed almost Newtonian behavior and had shear stresses an order of magnitude lower than the feeds with emulsifying salt at the same shear rates. Microstructural analysis revealed discrete, non-interacting particles of protein and fat. Several strategies for counteracting the destabilization of cheese feeds without emulsifying salts were studied. These included addition of other dairy based ingredients like butter milk powder and sodium caseinate, effects of using cheeses of differing ages, changes in processing parameters such as time and speed of mixing and adjustment of pH using KOH to simulate the increase in pH normally obtained by addition of emulsifying salt. Some of the studied strategies led to improvements in cheese feed stability and changes in rheology and microstructure in a direction towards the observed properties of cheese feeds with added emulsifying salt. However, only small improvements have been seen for individual changes thus the key to success may lie in combination of several of the suggested strategies and further changes in composition and processing of cheese feeds will have to be implemented in order to make complete removal of emulsifying salts from the recipes possible.

### Dissolution and functional properties of pea protein isolate

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The use of pea protein isolate (PPI) in food products is widely beneficial, due to its low economic cost, commercial availability and excellent amino acid profile, while it is also suitable for use in vegan, kosher and halal related products. The objective of this study was to determine the dissolution and functional properties of PPI. High intensity ultrasound (20 kHz, power 114.8 W) processing was used to dissolve PPI powder and the dissolution rate was compared to conventional methods of dispersion (e.g., stirring). Dissolution of PPI by stirring showed neither temperature  $(25 - 65^{\circ}C)$ nor time (up to 1440 minutes) increased solubilization of powder particles; however, sonication dramatically accelerated the dissolution process. This process achieved > 95 % levels of powder solubilization. Heating PPI dispersions (10 %wt protein) from 45 to 90°C led to an increase in the storage modulus G' at 71°C, indicating the onset of protein aggregation. Gel formation occurred at 79°C (G' > 1 Pa). Particle size of pea protein-stabilized emulsions formed using ultrasonication decreased with increasing sonication time (D50 3.83  $\mu$ m after 2 minutes) with a corresponding increase in viscosity (shear rate 300 1/s) from 8.6 to 20.4 mPas after 2 minutes. Pea proteinstabilized emulsions formed using microfluidization (50 MPA, 1 pass) resulted in the formation of cold-set gels, with gel strength increasing with increasing protein concentration or increasing oil fraction. Overall, ultrasonication ( $< 50^{\circ}$ C) can significantly improve the dissolution rate of highly insoluble PPI powder and when in the presence of oil create a stable emulsion.

### Rheological and textural properties of cheese chyme during in vitro gastric digestion

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Cheese disintegration, defined as the breakdown of cheese particles in stomach, may play an important role during the digestive process of macronutrients and their subsequent postprandial metabolic responses. The kinetics of macronutrients digestion is believed to be influenced by the viscosity and texture of food within the gastrointestinal tract. A new system was developed to highlight the changes in viscosity during the in vitro digestion of food products. A special geometry designed to maintain the cheese particles in suspension was used at different shear rates (30 and 150 1/s) to study the disintegration mechanism of cheese particles during in vitro gastric digestion. Light Cheddar and Mozzarella cheeses having a similar composition (protein and lipid content) and texture (hardness and cohesiveness) at 37°C but different structures were studied to highlight the impact of cheese microstructure on their digestive behavior. Light Cheddar and Mozzarella exhibited similar disintegration rate during the first 60 min when a low shear rate was applied (30 1/s). However, the disintegration of the Cheddar cheese reached a plateau value while Mozzarella has further decreased at the end of the gastric digestion. The protein release for both cheeses was similar at both shear rates. However, the lipids were released more rapidly for Mozzarella at 150 1/sdue to its higher level of disintegration and bigger fat globules. The viscosity of the cheese particle decreased exponentially over time for Mozzarella during gastric digestion while it remained stable for light Cheddar cheese (linear with small negative slope). These results were not fully explained by the disintegration rate but may be attributed to different texture softening phenomena during gastric digestion. Mozzarella cheese needed 60 min of soaking in the gastric juices to fracture while light Cheddar fractured at  $G_0$ . Therefore, the kinetics of cheese disintegration could be affected by cheese texture during gastric digestion, which may be influenced by water absorption, acidic and enzymatic hydrolysis. A better understanding of the role of dairy products microstructure and physical properties on nutrients' bioaccessibility will allow developing optimal food structures to deliver these key nutrients.

### Contraction measurements of cocoa butter and chocolate with a normal-force controlled rheometer

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The cooling and crystallization step during chocolate production has a major influence on the final product quality, in particular on gloss, texture and melting behavior. The crystallization-induced contraction leads to detachment of the chocolate from its mould and is of special importance as it defines the surface quality. Product faults associated with contraction and detachment are damages caused by mechanical demoulding, stress cracks due to too fast contraction and cooling spots with inhomogeneous glossiness. The aim of this work is to understand the contraction behavior of cocoa butter and chocolate to optimize the chocolate cooling process, in particular with regard to the final product quality. A normal-force controlled rheometer with temperature-controlled smooth plateplate geometry was used for contraction measurements of cocoa butter and chocolate. Cooling leads to solidification and due to density changes to a contraction of the sample. When reaching a certain pulling normal-force, the upper plate moves down to equalize it, and thus the contraction profile can be measured. The influence of different cooling rates and contents of cocoa butter crystal nuclei added to the samples was examined. Contraction starts earlier and therefore at higher temperatures when cooled faster as well as when more cocoa butter crystal nuclei are added. The latter showed a logarithmic dependency for nuclei concentrations between 0.1 and 2.0 % ( $R^2 \ge 0.95$ ). Total contraction was bigger for cocoa butter than for chocolate, and samples without added nuclei showed the lowest total contractions. Generally slower cooling leads to higher contractions (with exceptions). Contraction measurements are of significant importance for the optimization of the chocolate cooling process. It enables an in-depth understanding of process-induced changes during cooling and crystallization.

### The effects of hydrocolloids on final texture and synersis of native corn starch paste using a Rapid Visco Analyzer

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Due to huge use of starch in food industry, substitution of chemical methods with natural additives has always been a major concern. In this regard, effects of 3 hydrocolloids, Guar gum (GG), Xanthan gum (XG) and Konjac glucomannan (KGM) on final viscosity and set back of native corn starch was studied, using a Rapid Visco Analyzer (RVA). After studying the related parameters and RVA graphs the results were analyzed with the help of SPSS software. In order to consider the possibility of synergistic effects between hydrocolloids, their effects were considered both individually and in a twosome mixture with equal ratios at two concentrations of 0.1 and 0.5 %. By studying the RVA graphs it was observed that the highest final viscosity belongs to the sample of 0.5 % KGM  $(6777.00 \pm 200.73 \text{ cP})$  which indicates that KGM at 0.5 % can form the most viscous gel after processing and the difference compared to the witness sample was significant. It was also understood that the mixture of hydrocolloids at concentrations below 0.5 % cannot have synergistic effects on final viscosity, though the 0.5 % of KGM and GG could make a significant increase in the final viscosity compared to the starch itself but the difference compared to the 0.5 % of KGM was not significant. Regarding the setback, XG was the best choice to make a significant decrease which shows that XG can control synersis better, also it was resulted that XG can have better effects on setback while being used individually than being in a mixture with the other two hydrocolloids. At 0.1 % of concentration, GG can make a significant increase in final viscosity and XG can significantly decrease setback, but twosome mixtures at this concentration did not manage to make any significant difference. In order to illustrate the matter more clearly, several graphs and charts were used to show the differences between the samples.

# Simulation of the flow of semi-dilute guar gum and kappa/iota hybrid carrageenan gum solutions through symmetric and asymmetric contractions.

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The flow of non-Newtonian fluids through nozzles is an important operation in many food processes such as filling lines. The microstructural features which give rise to non-Newtonian behaviour often give rise to complex extensional flows. The microstructure in the fluid and its flow behavior are related so it is important to be able to compute the effect of nozzle geometry on both. In this case we are interested in the processing of bubbly liquids based on non-Newtonian liquid phases: previous work has shown that those studied here can be described as Giesekus fluids. The commercially-available computational fluid dynamics code Ansys(R) Polyflow was used to simulate the flow of semi-dilute guar and kappa/iota carrageenan gum solutions and their bubbly liquids through symmetric and asymmetric syringe geometries. Simulations were undertaken using a generalised Newtonian (Carreau-Yasuda) and a viscoelastic (Giesekus) model to describe the gum rheology. Separate rheometry tests yielded the model parameters. The flow pattern in the main syringe body was that of a classical Newtonian contraction flow for both models in the symmetric case, and for the Carreau-Yasuda model in the asymmetric geometry. Simulations in 3D and in axisymmetric 2D co-ordinates showed good agreement. 3D viscoelastic simulations in the axisymmetric geometry revealed the presence of a large, helical, corner vortex. The size of this vortex depended strongly on the relaxation time, but showed little dependence on the Giesekus mobility parameter and the zero shear rate viscosity. The effect of this helical vortex was to increase, substantially, the residence time of fluid in the syringe for a significant proportion of the flow, and hence expose this portion to a different shear and extension history. The shear rate distributions within the two syringe geometries were studied to investigate bubble break-up behavior. These computational results provide insight into experimentally-observed differences in the bubble size distributions that result when bubbly liquids consisting of semi-dilute guar or kappa/iota carrageenan gum solutions with an air volume fraction of 0.25 are passed through symmetric and asymmetric syringes to induce bubble break-up.

#### Damage of large food particles in simple shear flow

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Dispersions consisting of large particles (> 1 mm) such as instant soup, purees, baby food, or fruit drinks are widely used form of food products. Between the formation of the particles and the filling, the particles are exposed to mechanical and hydrodynamic stresses due to pumping, stirring, or flowing through pipes and contractions which can lead to attrition or comminution. Damaging the particles result in a modified end product and may lead to a decrease of sensory quality or/and additional costs. Past studies of large particles in process flows (e.g. in scraped surface heat exchanger and agitated tanks) attributed the damage to particle-blade collisions. The influence of the hydrodynamic stress on particle damage has not yet been investigated. Two different kinds of fruit particles were analyzed in this study: jellied fruit pieces where the juice concentrate is embedded in an alginate matrix and fruit particles hardened with calcium chloride. The particles with sizes around 3 mm were dispersed into a CMC-sugar-solution to increase the viscosity and density to avoid sedimentation at low shear rates. The suspension was sheared in concentric cylinder device with a large gap of 13.7 mm. The damage of the particles was quantified by counting, weighting, and measuring the morphology using digital image processing methods. Both kinds of particles were increasingly damaged with increasing shear stress and shear time. However, the damage of the alginate particles was rather based on abrasion since the circularity increases whereas the convexity remained the same. Surface area and mass of particle decreased. The damage of the hardened particles was based on fragmentation since the particle number increased and the convexity as well as the overall size decreased. The circularity did not change. Furthermore, a higher temperature increased the degree of damage for the alginate particles but had no influence on the hardened particles. The number of particles had no influence on the damage in the tested concentration range; particle-particle interactions can therefore be neglected. Hence, we could show that the large fruit particles can be damaged in simple shear flow and that the type of attrition depends on the nature of particle.

### Changes in rheological and physico-chemical properties of starch during chuño processing

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Rheological properties (dynamic and flow properties) were assessed in Northwest Argentinian potato cultivars (Solanum species called Rosadita) before and after the traditional freezing and sun-drying of potatoes known as chuño. Differential scanning calorimetry (DSC) was used to evaluate thermal properties (onset temperature  $T_0$ , peak temperature  $T_p$ , conclusion temperature  $T_c$  and total gelatinization enthalpy  $\Delta H_{gel}$ ). Chuño starch  $\Delta H_{gel}$  and  $T_p$  (22.7  $\pm$  1.3 J/g and 66.5  $\pm$  0.3 °C, respectively) were higher than in the starch of fresh potato (18.6  $\pm$  1.4 J/g and 65.5  $\pm$  0.1 °C, respectively). Rheological properties such us storage modulus G' and consistency coefficient K of starch from chuño were higher than native starch. That behavior was more evident at gels 10 %w/w than suspension at 5 %w/w. Determinations of amylose content AC and swelling power SP were performed. Results showed that AC and SP fresh Rosadita starch (AC = 21.1 % and SP = 18.3 g/g) were higher that Chuño starch (AC = 19 % and SP = 15.7 g/g). Wide-angle X-ray diffraction (WAXD) showed slightly differences between patterns; diffraction peaks at values of 15, 17, 22, and 24° typify B-type starches. The results suggest that the rheological properties are increased after chuño processing because of differences among temperatures during the process.

#### Microscopic and dynamic vapor sorption characteristics of spray dried model infant formula powders

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The structure and physical properties of powders, with composition similar to first stage reconstituted infant formula, i.e., 1.3 % protein (60:40 whey:casein), 7.4 % lactose, and 3.4 % fat (sunflower oil) were investigated. Liquid concentrates (pH 6.8) were heated by tubular heat exchanger to 65°C or 120°C (x 30s), homogenized and evaporated to 38, 48, or 58 % solids content prior to drying. Increasing feed concentration resulted in lower occluded air in the powders, as determined by gas pycnometry and microstructural analysis (cryo-scanning electron and confocal laser scanning microscopy). Glass transition temperature  $T_g$  of the finished powder increased by approximately 3°C when the feed solids content was increased from 38 to 58 %. Extent of denaturation did not affect  $T_g$ . Dynamic vapor sorption analysis was used to study water sorption / desorption and lactose crystallization kinetics. The relative humidity at which lactose crystallization occurred was similar for all powders, however, the amount of water sorbed by powders decreased with increasing solids content of liquid feed prior to spray drying can alter structural characteristics and hydration characteristics of model infant formula powders.

#### Modulating protein-protein interactions for in-process stability during manufacture of infant formula

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Bovine skim milk and whey protein powders are the primary ingredients used to meet the protein requirements in infant nutritional products. During processing, these ingredients are subjected to thermal treatment and evaporative concentration, modifying their rheological behavior in solution. In this study, model protein solutions (pH of 6.8) prepared from skim milk and whey protein concentrate powder (WPC80) were prepared, which on mixing, yielded a protein content of 4.7 %w/w at a whey protein to casein ratio of 60:40. The protein solutions were heat treated in a pilot tubular heat exchanger, either in combination or separately, at 85 or 120°C for 30 s, with an unheated mixture used as a control. Heating the skim and whey protein ingredients together in solution at 120°C resulted in significantly (p < 0.05) higher aggregate size (Z-Average, 265 nm) compared those mixtures whereby the skim and whey solutions were heated individually and then mixed (200 nm). Heat stability (oil bath, heat coagulation time, 120°C) was lower (24 minutes at pH 6.8) in mixtures where skim and whey were heated in combination compared to when heated separately and then mixed (> 50 minutes at pH 6.8). The control had significantly (p < 0.05) lower viscosity compared to the heated formulations. Heating to 120°C resulted in lower levels of native  $\alpha$ -lactalbumin,  $\beta$ -lactoglobulin and bovine serum albumin and higher amounts of soluble  $\beta$ casein in the serum phase compared to control mixtures. The study demonstrates that knowledge of protein/protein interactions can be utilized for development of ingredients with application in infant formulations.

### Effect of concentration on heat induced formation of whey protein aggregates

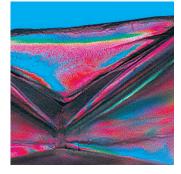
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Controlling denaturation and aggregation kinetics of protein solutions during heating provides a mechanism for altering whey protein ingredient functionality. Solutions of whey protein (1, 4, 8, and 12 %w/w, Davisco BiPROTM) were heat treated at 85°C using a pilot scale heat exchanger (MicroThermics®) to induce denaturation/aggregation (30 s, pH 6.2, 6.7, or 7.2). Aggregates formed were characterized by gel permeation HPLC, dynamic light scattering, heat coagulation time and rheology. Increasing concentration and decreasing pH resulted in a significant increase (p < 0.05) in aggregate size and decrease in the amount of monomeric species compared to non-heated control samples. Particle size, determined by dynamic light scattering increased with decreasing pH. Solutions containing aggregates with a mean particle diameter (z-average) in the range 50 - 85 nm had increased heat stability compared to those solutions with aggregates in the range 137 - 154 nm (formed at lower pH values). Solutions containing 12 % whey protein showed a significantly (p > 0.05) higher increase in viscosity on heating at all pH's compared to those heated at either 1, 4, or 8 % protein. The findings demonstrate that the size of aggregates produced in whey protein solutions is both concentration and pH dependent.







## Rheological Methods

# Application of the rheological measurements to determination of amylolytic activity of malt used for bozo beverage

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Malting is the most important stage in the processing of the Kyrgyz traditional beverage Bozo. In current study, the amylase activities of maize, millet, wheat, and barley malts were investigated by rheological methods using cooked millet porridge as a substrate for enzymes. The aim of this work has been to demonstrate the applicability of rheological measurements as alternative, rapid, safe for health and simple method for measuring amylolytic activity of grain malts. The standard colorimetric method for the determination of amylase activity was compared with rheological approach. Millet porridge exhibited pseudoplastic behavior and the Ostwald - de-Waele was used as suitable fitting model. The highest amylolytic activity was found in barley malt and the lowest in maize malt. The rheological approach is demonstrated to be advantageous, particularly with regard to speed, simplicity, no requirement for chemicals and possibility of the online monitoring of the structural changes.

#### Microscopic precursors of fracture in amorphous solids

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We use space and time-resolved dynamic light scattering in the highly multiple scattering regime (DWS), to probe the microscopic dynamics of amorphous solids during mechanical tests. Our experiment allows us to measure the microscopic motion in the sample, by measuring the local mean squared displacement down to a resolution of fraction of nm<sup>2</sup>, averaged over regions of interest (ROIs) of typical size of tens of microns. Additionally, we measure the mesoscopic drift of the same ROIs defined for the microscopic dynamics. We study a semi-crystalline polymer (PEEK) and an elastomer (PDMS). The test protocol consists of several stretching steps, each of them followed by a relaxation step during which the elongation strain is constant. During the stretching step, the measured mesoscopic drift is in agreement with the affine deformation imposed. The relaxation steps following these early stretching steps are characterized by microscopic rearrangements that slow down with time, following the relaxation of the macroscopic stress required to keep a fixed strain. A completely different scenario is observed as the material approaches macroscopic failure. The microscopic dynamics becomes order of magnitude faster than in the previous relaxation steps, unambiguous signaling microscopic rearrangements that eventually lead to macroscopic failure. Remarkably, this strong increase of the microscopic mobility occurs thousands of seconds before macroscopic failure. Then, we build a dynamic mapping (2D) to follow the microscopic dynamic of different region of interest on my sample. This Map indicates that the microscopic rearrangements present heterogeneity at relaxation phases when the sample is perfectly fixed. Moreover, if we compare the map result with an image after failure, we observe that the region with the more important microscopic rearrangements will be the region of failure. These experiments show the interest of this technique as a method to predict and localize an eventual failure of a material.

### Interfacial wall-slip and its consequences on Lissajous plots

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Emulsions and foams are generally stabilized by low molecular weight surfactants, proteins, or by particles. The high adsorption energy of particle systems results in ultra-stable foams used in various industrial applications. In this study, hydrophobic silica and clay particles are compared due to their completely different particle network properties: Silica particles build strong networks in contrast to clay particles which have weak network structure. To measure the interfacial rheological properties of these networks a biconical geometry was used and the results are analyzed in stressstrain Lissojous plots. The rheological measurements were combined with microscopy and optical coherence tomography (OCT) to visualize the flow behavior of the interfacial layer. It turned out that non-linear Lissajous plots showed a parallelogram like shape. In addition, the rheological response of silica particle interfaces showed a characteristic oscillation in the horizontal part of the Lissajous plots. The optical measurements revealed that the particular shape of the Lissojous figures is caused by interfacial wall-slip at the biconus for the silica and at the cup wall for the clay particles. We propose that the truncation of the Lissajous plots can be correlated with the wall-slip. The results emphasize in combination with morphological information, Lissajous plots can help to understand complex nonlinear rheological behavior.

### Gluten free porridge with accustomed rheological properties

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Traditionally porridge is prepared of oat flakes or other cereal flakes with water or milk. Regarding the texture, consumers of gluten free foods have to face many disadvantages. In this contribution it was tried to simulate the texture and the gelatinization behavior of a typical porridge recipe. The obtained porridge recipe should have the same texture and the gelatinization behavior like traditional prepared porridge. In the first step 12 possible cereal, pseudo cereal and other gluten free flakes, oat flakes (rolled and fine) and a commercial available gluten free porridge-mix were analyzed using a Rapid Visco Analyzer (RVA) as described in ICC Standard 162. For all experiments the same dry matter was used. The behavior of the commercial available gluten free porridge mixture was quite different compared to traditional porridges. After correlating the starch content with the end viscosity of the RVA measurements the six most promising flakes - buckwheat, red millet, lupine, amaranth, millet and teff - were analyzed using a rheometer. An individual profile was created to record the viscosity of the mixtures during mixing, heating and cooling trying to simulate the typical porridge cooking procedure. Within this procedure the sample mixture was mixed at 20°C for 10 s with 160 rpm, the temperature was linearly increased up to 95°C within 4 min, maintained at 95°C for 2 min and decreased to 60°C linearly within 5 minutes and maintained at 60°C for 5 minutes. The viscosity was characterized with rotational measurements at 60 rpm for the heating and maintaining step. The viscosity of the cooling and maintaining step was characterized with oscillatory measurements at 1 % deformation and a constant radial frequency of 10 rad/s. Finally the rheometer measurements were repeated with mixtures of flakes that resembled to oat flakes. In this contribution we will present that a similar rheological behavior to that of oat porridge can be obtained with mixtures of different pseudo cereals and cereals.

### Impact of preshearing conditions on rheology of non-colloidal suspensions

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Dense non-colloidal suspensions exhibit a complex rheological behavior depending on various parameters such as the solids volume fraction, the particle size distribution, the shapes of the particles, and the preshearing conditions. In an earlier experimental study [1], it was found that lignite-water slurries having a high solids volume fraction (higher than 0.4) show a typical thixotropic loop during the first cycle of a cyclic shear measurement, while in consecutive runs, the extent of the loop is reduced and an abrupt change in the slope is present. This flow-induced phenomenon was attributed to shear banding. In this talk, we will discuss the impact of the preshear stress level on the rheology of these slurries. The experiments were performed using parallel-plates and Couette geometries. We found that increasing the magnitude of the applied preshear stress increases the yield stress and moves the onset of shear banding to higher shear rates. The storage and loss moduli were only found to be quantitatively affected by the preshearing conditions. In addition, we also performed some creep measurements to investigate the temporal behavior of both unimodal and biomodal suspensions and it was found that a power-law relationship could be used to describe the initial strain-time relationship.

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### Extensional rheological characterization of food suspensions for optimizing spraying and 3D printing

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Food industry and governmental institutions provide substantial research grants for investigating and developing novel printing technologies like 3D printing with food suspensions [1]. In 3D printing technology, developers prefer to use non-toxic and eatable samples for their trial runs [2]. Typical products for 3D printing include confectionary, pasta and pizza dishes as well as personalized nutrition meals for special target groups like elderly persons, pregnant women, sportspersons and astronauts with individual concentrations of essential nutrients. For the confectionary and chocolate industry, 3D printing offers the capability to generate specific shapes and designs which are not available with traditional processing techniques like molding and spraying [3]. In 3D printing as well as in spraying, the material is forced through a narrow opening like a nozzle or a die. This causes shear deformation as well as extensional deformation of the processed material. Thus, for a comprehensive understanding of the entire process, rheological investigations in shear as well as in extension are mandatory. The shear behavior of chocolate melts is well known and has been objective of several studies worldwide. However, the extensional behavior of these materials has not been extensively studied yet. This work describes the extensional behavior of chocolate melts investigated by using capillary break-up experiments. The gained information can then be used to optimize the 3D printing processes.

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#### Development of a new rheological method in the rennet and lactic coagulation

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Texture of gel during the coagulation process is an important characteristic of cheese and yoghurt [1]. The rheology is a technique widely used to investigate this coagulation process [2]. The experiment more widely used is the time sweep oscillation test. In this test, the storage modulus G' and the loss modulus G" are recorded during the coagulation process [3]. In the most part of the published papers, the time sweep is made at a fixed strain, which, in some cases, is obtained in a previous strain sweep test with a coagulated sample, and in other cases, it is simply obtained through the reading of other authors. There are two important aims when a determined strain is chosen. One of them is that the strain has to stay, during the coagulation experiment, within the linear viscoelastic region in order to prevent the breaking of the gel. The other is that, the strain (stress) has to be higher than the minimum strain (stress) that the instrument is able to develop (strain or stress depends on the instrument, strain control or stress control). In general, the change in G<sup> $\prime$ </sup> is about 3 or 5 decades during the lactic, enzymatic or mixed coagulation. So, if in a coagulation experiment, the strain is constant, at least one of the two conditions previously mentioned does no't probably fit. So, in this work, we propose a new method to perform all kind of coagulation experiments changing the strain amplitude depending on the applied oscillatory stress. A new method to perform rennet and lactic coagulation experiments was performed to improve the oscillatory data. This method allows to measure the low viscous material at low times with a high strain and the high viscous material at high times with a low strain.

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### Temperature ramp rheological method to measure five kinds of mozzarella cheese

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The texture and rheological methods are widely used in the characterization of mozzarella cheese [1-3]. Nevertheless, there are few articles that provide rheological properties in dependence of temperature. Obviously, it is very interesting to characterize the melting properties of different kinds of mozzarella cheese during a temperature ramp in the widest rage of temperature. The temperature control, the normal force control and the continuous oscillation (direct strain oscillation) of the AR-G2 and the AR-2000 allow performing this kind of measurements accurately. With this method, five kinds of commercial mozzarella cheese have been measured. We describe the rheological properties and a comparison between them. The method developed is suitable for the characterization of the melting process of the greases of a mozzarella cheese. The oscillatory measurements within the linear viscoelastic range provide accurate results. The normal force control allows keeping the contact between the plates and the cheese. This measurement allows characterizing the melting process of cheeses.

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### Determination of flow stability of dietary food fibers by using powder rheometer

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Dietary fibers are commonly used in food industry due to their functional properties. Many of animal and vegetable originated foods contain different proportion of dietary fiber within their nature. Some of cereals such as wheat, rice, oat, barley; some fruit and vegetables like apple, orange, fig, apricots, pea, carrot, broccoli and cabbage are the food source of dietary fibers. Commercially, food fibers are obtained from these food materials. In recent years, the production and consumption of food which contain fibers are increasing day by day since their functionality. Powder form of these fibers affects the physicochemical properties of foodstuff which is added in. Thus, it gets importance in the production, storage and transportation processes especially in terms of flowability of the powder. In this study, we evaluated the powder rheology of some selected and commonly used food fibers. For this purpose, a powder rheometer attached to texture analyzer was used and flow characteristics of inulin, oligofructose, carrot, apple, lemon and pea fibers were examined. Dietary fibers were subjected to cohesion, caking and speed dependency tests. Cohesion index values are used to get information about powder classification with flow characteristics. Fibers were categorized as free flowing, easy flowing and cohesive according to cohesion indexes. Inulin and oligofructose belonged to free flowing category with cohesion index value of 8.98  $\pm$  0.92 and 9.04  $\pm$  0.59; lemon and pea fibers were easy flowing with 13.52  $\pm$  0.58 and 13.37  $\pm$  0.66; carrot and apple fibers fall into cohesive class with 14.24  $\pm$  0.14 and 13.52  $\pm$  0.58, respectively. To evaluate the caking properties of fibers caking test was performed. There was a significant difference (p < 0.05) between the mean cake strength of fibers. Powder flow speed dependency test gives information about the resistancy of flow and subjection to break downing of powders during test. Flow stability of fibers was changed between 0.92 and 1.16 and indicated the unchanged flow properties.

### Viscosity and powder rheology of a traditional powdered dessert: Incir Uyutmasi

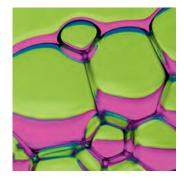
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The milk has a very important role in human nutrition. In order to get more benefit from milk various dairy production technologies has been improved to increase the shelf-life and to get some different functionality. In recent years, the trends in consumption of natural and healthy foods directed the food sector to produce natural foods or desserts. Incir Uyutmasi, which consumed in Anatolia and Middle Asia is a one of the dairy based traditional desserts. To prepare this dessert, the figs are sliced into smaller pieces and mixed with boiled milk, after homogenization process the mixture is waited at 40°C for nearly 1 hour. After fermentation dairy dessert are stored at 4°C for 5 hours. In this study, to evaluate the rheological characteristics, effect of fig concentration (0.100,0.150, 0.200, 0.225, 0.300 % (g/L)) on the viscosity of samples was determined. Results indicated that, the viscosity of samples was changed between 163 - 3393 mPas and viscosity was increased by increasing fig concentration. The optimization of Incir Uyutmasi dessert in powdered form was also investigated. In this respect, powder rheological analysis was applied by a powder rheometer attached with a texture analyzer. To determine flow characteristics of powder samples, three critical tests (cohesion, powder flow speed dependency (PFSD) and caking tests) were performed. The cohesion index of sample was 10.06  $\pm$  0.9 which indicates the free flowing powder. PFSD test gives information about the flow properties of powders at different speeds. Flow stability of the powder ingredients of dessert was calculated as 1.08  $\pm$  0.00 and this close value to 1 indicates there is no change within the powder during test. As is known, caking is undesirable process condition in transportation and storage periods since caking results in large agglomerates and causes problems. Indication to caking was tested and mean cake strength which was the average force to cut the cake in g was determined as 75.14  $\pm$  2.4. This slightly low mean cake strength was supported by low cohesion index for free flowing powder in this study. Since causes of caking can be dependent strongly with cohesion, low cohesion indicates little caking formation as in the case.







## Emulsions and Interfaces

### Rheology, microstructure and particle size of oil/water/cellulose emulsions used as fat replacers

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The reduction and/or elimination of saturated and trans-fatty acids from diets constitutes a difficult task for food manufacturers. Fats with low percentages of saturated fatty acid, such as olive oil or sunflower oil are liquid at ambient temperature and cannot accomplish the structural functions associated to solid fats. One way to confer semi solid structure to liquid oils without generating trans-fatty acids or increasing the saturated fatty acids is to incorporate them in a cellulose ether emulsion. An emulsion composed of sunflower oil, water and a cellulose ether makes possible the total replacement of conventional fat in a short dough recipe while obtaining good sensory acceptability. The aim of the present work is to investigate the thermo-rheological properties of the oil/water cellulose emulsions. Three different cellulose types (F4M, A4M and MX) were used and the thermal reversibility properties of the vegetable oil cellulose ether emulsions were studied by small amplitude oscillatory shear, optical microscopy and particle size analysis. Differences in the viscoelastic properties were observed among the different cellulose emulsions at 20 and 80°C after thermal gelation. At 20°C the highest viscoelasticity was shown by the MX emulsion. The emulsion gelation temperature decreased with cellulose ether methoxyl content. Emulsion MX showed the lowest thermal stability. Heating the MX cellulose emulsion caused syneresis, fat flocculation and the appearance of particle size polydispersity, indicating lower thermal stability and lower thermal reversibility. The different viscoelastic properties and thermal stabilities obtained by varying the type of cellulose increase the number of possible food applications where the emulsions could be employed as suitable fat replacers.

## Rheological properties of O/W/O emulsion containing resveratrol and milk protein

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Resveratrol is a natural polyphenolic compound found in peanut butter, red wine, and grape juice. This compound has aroused widespread interest due to biological effects associated with health benefits. Most of its biological activities are attributed to the trans-isomer and in solution; transresveratrol converts to its cis-isomers under exposure to light. This component exhibits often low bioavailability as its absorption may be kinetically limited by low rates of dissolution and capacity limited by poor solubility. For this reason a double emulsion system (O/W/O) that nowadays is considered as a highly attractive option in both pharmaceutical and nutraceutical application will be used. Emulsion templating method will be used. In this regards, we will use a three-step procedure: Preparation of single emulsion, preparation of double emulsion (O/W/O), and gelation reaction with calcium chloride. To characterize the physicochemical properties measurements, zeta potential, rheology, and TEM will be performed.

#### Emulsifying and stabilizing properties of functionalized orange pulp fibers

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The emulsifying and stabilizing properties of dried orange pulp fibers will be described. This plant cell wall material demonstrates interesting surface active properties, which are believed to originate from the intrinsic proteins ( $\sim 8$  %) and pectins ( $\sim 35$  %). It also contains a high amount of cellulose (i245%), which enables the fibers to act as thickener. The emulsifying properties of the dried native fibers will be compared to the ones of their functionalized counterparts. Functionalization will be obtained through high pressure homogenization of the fibers at 700 bar prior drying. Viscoelastic properties of the different emulsions will be studied in relationship to their long term stability. c\* of the emulsions at same fiber:water ratio will be compared to the critical concentration of the fiber suspensions in absence of oil in order to study the impact of oil on fiber confinement. CLSM imaging will be used to study the impact of processing on fiber morphology and how it impacts the microstructure of the obtained emulsions. A comparison will be made between functionalized orange pulp fibers and their non-activated counterparts.

#### Characterization of olive oil emulsions formed by catastrophic phase inversion using bacterial cellulose and whey protein isolate

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This work was focused on the use of catastrophic phase inversion (CPI) technique for the formation of emulsions in the presence of green ingredients, such as bacterial cellulose (BC) and whey protein isolate (WPI). Emulsions were prepared at pH 3.8 with an oil to water ratio of 10:90. The aqueous phase, containing BC and WPI at various concentrations or a mixture of both, was titrated via a burette into the oil phase under constant stirring (500 rpm) at ambient temperature  $(25^{\circ}C)$  with a flow rate of 10 drops/min. The produced emulsions were investigated by measuring the oil droplet size, their stability over storage (7 days, 4°C) using the multiple light scattering theory (MLS) and their structure by optical microscopy. According to our findings, phase inversion happened at the 55 - 70 % of the added water phase. When BC or WPI on their own were used, droplet size decreased with increased concentration. On the contrary, their mixture had no significant effect on droplet size. Furthermore, the produced emulsions presented instability phenomena. Creaming phenomena occurred as soon as the emulsions were prepared. Soon after, all emulsions presented sedimentation instability. As WPI or BC concentration was increased, the serum level was lessened and the sediment was increased. The mixture showed similar instability behavior. In some cases, microscopy images indicated flocculated droplets. In order to have a better understanding of our findings, flow curves (i.e. viscosity versus shear rate) of the biopolymer solutions used in the aqueous phase were constructed. According to those, WPI solutions, at all studied concentrations, were Newtonian, whereas BC exhibited shear thinning behavior. Moreover, BC solutions were more viscous than the WPI ones. All BC-WPI mixtures exhibited shear thinning behavior and shared similar viscosities with the solutions containing only BC; thus, showing that BC has the dominant part in their behavior. In all cases, viscosity increased with concentration. Taking into consideration all our findings, it seems reasonable that the emulsification process played an important role. As a low energy method was used, it is possible that the energy offered to the system was not sufficient.

## The use of green biopolymers in the formation of double emulsions

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Multiple emulsions have great potential at pharmaceuticals, cosmetics and food as encapsulation systems. They consist of a dispersed phase which contains smaller droplets similar to the continuous phase. The aim of this study was to investigate the utilization of two green biopolymers, namely bacterial cellulose (BC) and whey protein isolate (WPI), coming from renewable resources and waste streams, respectively, in the formation of water-in-oil-in-water emulsions. Initially, primary water-in-oil (W1/O) emulsions were prepared using olive oil and an aqueous phase of increasing concentrations of polyglycerol polyricinoleate (PGPR). Afterwards, aqueous BC or BC-WPI solutions (W2) were used to form secondary water-in-oil-in-water (W1/O/W2) emulsions. In addition, in a selected PGPR concentration, increasing amounts of sodium chloride were added in the internal aqueous phase (W1) in order to improve emulsion stability of the final double emulsion. The external phase was a standard aqueous BC-WPI solution (W2). The emulsification of both stages was performed with a rotor-stator CAT X120 device at 20°C and at pH 3.6. Produced emulsions were characterized in terms of microstructure, droplet size, stability and viscosity. According to our results, the droplet size was highly dependent on the concentration of the emulsifier or the stabilizer used, as increased PGPR or BC concentration resulted in smaller droplets. The combination of BC and WPI, led to more decreased droplet size compared to BC alone. Additionally, an increase in NaCl concentration was followed by increased droplet size. Stability assessment during the emulsion storage showed instability phenomena, mainly flocculation. However, the addition of NaCl in the concentration range of 4 - 5 %wt of the total double emulsion limited the instability rate. Based on these results, the more stable emulsions were selected and their viscosity over shear rate was measured. The constructed flow curves showed that all emulsions containing sodium chloride exhibited shear thinning behavior and their viscosity depended on the salt concentration. In the absence of salt, the emulsions were slightly off the Newtonian behavior. According to literature, shear thinning is associated with aggregated emulsion droplets. In good agreement with this suggestion, the optical microscope images of the studied emulsions showed that they consist of flocculated droplets.

#### Incorporation of epigallocatechin-3-gallate (EGCG) in emulsions containing bacterial cellulose

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Epigallocatechin-3-gallate (EGCG) is considered the most significant tea catechin because of its high free radical scavenging activity and its ability to prevent carcinogenesis. Thus, adding EGCG to food products could potentially benefit the consumers. However, EGCG has low bioavailability which makes it difficult to be incorporated in food products. When EGCG is modified structurally, its lipophilicity is improved and thus its application in lipophilic media is expanded. The lipophilicity of EGCG could be enhanced by esterification of hydrophilic EGCG with fatty acids, followed by purification. By this method, the EGCG incorporation rate in emulsions could be enhanced. Emulsions are commonly used delivery systems for antioxidant components. However, not much research has been done on the use of new polymers as thickeners in these emulsions. Bacterial Cellulose (BC) is a polysaccharide produced from Komagataeibacter sucrofermentans, not widely used in the food industry. Hence, the objective of the present study is to investigate the effect of the BC addition on the properties of WPI-coated emulsions and the oxidative stability of the emulsions when adding EGCG (0 - 0.5 mg/mL). O/w emulsions containing olive oil, WPI (2-5% wt) and BC (0 - 1 %wt) were prepared by ultrasounds. Emulsion characterization was carried out in terms of droplet size, stability and rheological behavior. At lower BC concentrations (0.5 -0.7 %wt), extensive aggregation led to unstable emulsions due to bridging flocculation. However, at higher concentrations (1 %wt BC), stable emulsions were obtained due to steric stabilization or network formation of BC fibrils. The viscosity profile of the BC emulsions was shear thinning. In the most stable emulsions, EGCG hydrophilized (H) and lipophilized (L) was added. Samples were stored at 25°C for 30 days and analyzed for conjugated dienes (CD) and p-anisidine value (p-AnV) at predetermined time intervals. As far as the oxidative stability is concerned, by the addition of EGCG, the emulsions showed lowered CD values, indicating that EGCG had delayed the oil oxidation. Furthermore, the primary and secondary oxidation products showed dependency on the EGCG structure (lipophilized or not). Results indicated that both lipophilized and hydrophilized EGCG could behave as an antioxidant in order to improve emulsions oxidative stability.

## Rheological investigations on foaming properties of gas-saturated O/W emulsions

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The production of foams like ice cream, confectionary mousses or bakery products is widespread in the food industry. Their popularity is based on the textural and rheological properties giving a light and creamy mouthfeel. The incorporation of gas into a fluid and the subsequent stabilization of bubbles are crucial to generate foams with targeted overrun, bubble morphology and texture. Previous investigations on different food materials indicate that in situ foaming by gas evaporation on pressure release leads to finer foam structures compared to whipping or shaking processes. For controlled foam generation it is necessary to investigate the complex process of (i) gas dissolution, (ii) bubble nucleation and growth and (iii) foam stabilization as many changes on a physical, chemical and colloidal level occur at the same time. For systematic analysis of the foaming process a versatile O/W emulsion with a shear thinning power-law flow behavior was chosen. The amount of dissolved gas and the kinetics of dissolution were determined at selected temperatures and pressures using a magnetic suspension balance. Carbon dioxide and nitrogen were used as blowing agents, wherein carbon dioxide shows a higher solubility and shorter equilibrium times. Since generation and stability of aerated systems are primarily affected by their rheology, a new setup has been developed combining a high-pressure vessel with a rheometer. This makes it possible to measure the viscosity of gas-saturated liquids depending on shear rate, temperature and pressure. With increasing amount of dissolved blowing agent the viscosity of the emulsion decreases. The next critical step in the foaming process is the generation of high bubble nucleation rates in order to achieve fine foam structures. Number and size of bubbles mainly depend on viscosity, amount and type of gas and pressure drop rate. The influence of these parameters on viscosity changes during bubble nucleation and growth were studied as well as the overrun, cell density and foam morphology. The outcome of this work helps to understand the relationships between relevant parameters in order to optimize such foaming processes and to tailor foam structures with specific properties.

## Effect of pectin on the interfacial rheology of $\beta$ -casein layers at the air/water and oil/water interfaces

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A large number of food foams and emulsions is stabilized by proteins. Polysaccharides are also added to the systems aiming at increase the viscosity of dispersed phase which, in turn, results in a stabilizing effects. A number of polysaccharides can also have interfacial activity and, therefore, can modify the behavior of proteins adsorbed at the interface. Aim of this work is the investigation of pectin behavior in mixture with  $\beta$ -casein, a protein guite common in food multiphasic systems. The study was carried out focussing on interfacial rheological properties and absorption kinetics of pure  $\beta$ -casein, pure pectin and their mixtures both at the air-water (A/W), and olive oil-water interfaces. Two pectins were used, with the aim of evaluating the effect of this characteristic on interfacial properties. Moreover, olive oil was used both in a purified (Op/W samples) and nonpurified (O/W) form, being the last one closer to the real commercial systems. The concentration of the pure samples was changed in the range  $10^{-4}$  - 1.5 g/l and 1 - 15 g/l for  $\beta$ -casein and pectins respectively, while for binary mixture a constant protein concentration (0.1 g/I) and different protein/pectin ratios were used during the tests. The samples were characterized in two different kinematic conditions, shear and dilatational, with the use of a magnetic rod Interfacial Shear Rheometer (ISR) and a pendant drop tensiometer. Dilatational experimental results evidenced that the adsorption and the interfacial dilatational properties of  $\beta$ -casein/pectin adsorbed layers are mainly affected by the presence of  $\beta$ -casein molecules, as also suggested by the application of a rheological model, commonly adopted for the description of bulk properties of mixed systems. Dynamic dilatational data exhibit a solid-like behavior for the interfacial films similar to 3D weaklystructured systems. Shear tests showed an increase of interfacial shear properties as a function of aging time and concentration at all interfaces, mainly at A/W and Op/W interfaces. Moreover, interfacial shear rheology showed that the addition of pectin improves the mechanical properties of interfacial films, mainly in presence of low-methoxyl pectin.

## Preparation of water-in-water emulsions based on gelatin/pectin solutions for food systems

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Protein-polysaccharide systems recently have been brought as a novel and promising area for investigations and precise studies of key factors responsible for interactions that occur in the system. Water-in-water emulsions can be essential for substitution of oil-in-water emulsions in developing new food textures with improved physicochemical properties as well as for the novel encapsulation systems. This work was dedicated to the development of water-in-water emulsion basing on the ability of protein to demonstrate segregative phase separation in polysaccharide medium [1]. Mixtures of gelatin/high methoxy (HM) pectin and gelatin/low methoxy (LM) pectin were investigated under certain conditions. A visual phase separation was not observed in gelatin/LM pectin mixtures irrespective of concentrations and pH of the solutions. Thus, phase separation was exhibited only for gelatin/HM pectin system at pH = 7.0. Characterization of each phase after preparing a sample and centrifuging indicated the formation of two biopolymer-rich phases: gelatin-enriched lower phase (W1) and a pectin-enriched upper phase (W2) that specifically corresponded to thermodynamic incompatibility of polysaccharide and protein. Considering a wide range of gelatin and pectin concentrations a phase diagram of particular biopolymer solutions was determined [2]. From the general observation of working solutions the phase diagram was consisted of two distinct regions referring to homogeneous phase and the one separated into two different phases. Water-in-water emulsions (W1/W2 or W2/W1) with different compositions were prepared by blending incompatible upper and lower phases together. The microstructure of obtained emulsions was observed and characterized by using optical microscopy in combination with rheological methods. The gelation temperature and viscous/elastic parameters were determined by studying the rheological behavior of the mixtures at constant HM pectin concentrations of 3 % and varying concentrations of gelatin.

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#### Impact of structural factors on the fat release from emulsions during digestion in vitro and in vivo

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Behavior of emulsions in the gastrointestinal tract depends on their initial structural properties. With properly modified initial emulsion properties it is possible to slow down the release of fat in the digestive tract and the absorption of lipolysis products. In vitro digestion systems of emulsions are still in the development stage, which is why it is difficult to compare results of similar studies between different scientific groups. In addition, many of these results are not verified in the human digestive or in vitro systems with natural human digestive juices. The aim of this study was to evaluate the stability of one layer stabilized (whey proteins) and double layer stabilized (whey proteins and carboxymethylcellulose) W/O emulsions (40 % fat) under digestion conditions, using in vitro digestion systems with natural (human) digestive juices. Results of this study were compared with the results obtained using in vivo model. The in vitro emulsion digestion assay was performed in two steps: intestinal and duodenal. The samples were taken every 30 minutes of digestion. The degradation of protein and fat, rheological properties of emulsions and the oil droplet size distribution during digestion were evaluated. The in vivo method was performed with adult male Wistar rats fed by emulsions for two weeks. Triglicerides and free fatty acid content in the blood and fecal after the first and second week of the experiment was determined. The hydrolysis of fat in double layer stabilized emulsions, under simulated in vitro conditions of the stomach and small intestine, was lower than that of the one layer stabilized emulsion. At the end of process, the oil droplets of the double layer stabilized emulsion remained small ( $\sim$  4  $\mu$ m), covered with a  $\beta$ -lactoglobulin surface layer, and evenly distributed. The values of the consistency index were determined to be higher (110.09  $\pm$  11.15 mPas<sup>n</sup>). These results suggest that the second layer formed on the oil droplet by carboxymethylcellulose, prevented the lipase from reaching the droplet surface, thus slowing down the digestion of fat. For this reason, the smallest amount of free fatty acids was determined in this emulsion (1.94  $\pm$  0.09 %). A lower amount of triglycerides (0.28  $\pm$ 0.04 mmol/L) and a higher amount of fat (5.83  $\pm$  0.16 %) were determined, respectively, in the blood and feces of rats fed with double layer stabilized emulsions for two weeks, in comparison with data obtained when rats were fed an emulsion stabilized by whey protein alone.

#### Application of response surface methodology and central composite rotatable design to the analysis of egg white protein-pectin electrostatic interaction in a high sugar content system on foaming capacity and foam rheological properties

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In aerated confectionery, foams are produced by aeration of a mixture of sugar syrup and proteins such as egg white protein (EW). Polysaccharides are also used due to their thickening and gelling properties. At pH 3.0, the pH is bellow the EW pl (4.5) and above the pectin pKa(2.9 - 3.5). Thus, negative charge of pectin may interact with positive charge of protein leading to electrostatic complexes formation. The aim of this study was to evaluate the effect of electrostatic interaction between egg white protein (EW) and low-methoxyl pectin in a high sugar content system (80 wt% total solid) on the foaming capacity (density and overrun) and foam rheological properties. Central composite rotatable design was carried out to evaluate the effect of total biopolymer concentration and EW:pectin ratio on apparent viscosity of sugar/EW/pectin mixture before whipping at 10 1/s, foaming capacity for fresh foam and rheological properties (elastic modulus G', viscous modulus G" and tan  $\delta = G'/G''$  at 1 Hz) for fresh foam and foam aged for 24 h at pH 3.0. Apparent viscosity of sugar/EW/pectin mixture before whipping increased with increasing biopolymer concentration. EW:pectin ratio had no significant effect (p > 0.1) on this response. At biopolymer concentration between 3.0 and 4.0 wt% and EW:pectin ratio from 40:1 to 63:1, the mixtures showed high foaming capacity (low density and high overrun value), and the foams, elastic (high value of G') and solid behavior (low value of  $\delta$ ). On the other hand, increasing relative pectin concentration (EW:pectin ratio < 35:1) the foaming capacity decreased and the foam became less elastic (low value of G') and solid (high value of  $\delta$ ), within the studied range. Possibly, EW:pectin ratio lower than 35:1 leads to formation of insoluble complexes between the biopolymers resulting in reduced protein availability, which may hinder the incorporation of air. The effect of electrostatic interaction between EW and pectin on foaming capacity and rheological properties in a high sugar content system depend on EW:pectin ratio which are related to electrostatic complexes formation at pH 3.0.

#### Influence of pH on foaming and rheological properties of aerated high sugar system with egg white protein and hydroxypropylmethylcellulose

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Aerated confectioneries (marshmallow and nougat) are produced using high boiled syrup and surface active agent such as protein which can be combined with polysaccharides. Egg white protein (EW) is used as surface-active ingredient in aerated food and hydroxypropylmethylcellulose (HPMC) is a polysaccharide that behaves as a surfactant. Thermodynamic incompatibility of proteins and polysaccharides in solution and the effect of sucrose on the thermodynamic properties (protein hydrophilicity and surface activity) depend on the pH. The objective of this work was to study the influence of pH on the apparent viscosity of sugar/EW/HPMC mixture before whipping, foaming capacity (density and overrun) and foam rheological properties (elastic modulus G', viscous modulus G" and tan  $\delta = G''/G'$  at 1 Hz) of sugar/EW/HPMC mixtures. Experiments were carried out with 5 %wt total biopolymer concentration, 14:1 EW:HPMC ratio, 80 wt% total solid (high sugar content) at pH 3.0, 4.5, and 6.0. Bubble size distributions of foams aged for 24 h were measured by analyzing the confocal scanning laser microscope images using Image J software in order to evaluate the foam stability. pH did not affect the apparent viscosity of sugar/EW/HPMC mixture before whipping. At pH 3.0, foam presented the highest foaming capacity (low density and high overrun value), elastic (high value of G') and solid (low value of tan  $\delta$ ) behavior without drainage. At pH 4.5, foam presented lower foaming capacity and monodisperse bubble size distribution, leading to better stability (concerning disproportionation and coalescence) than foam at pH 3.0. At pH 6.0, foam showed the lowest foaming capacity, the highest instability (widest bubble size distribution) and viscous behavior (high value of tan  $\delta$ ). Thus, HPMC could be considered to increase the stability of aerated confectionery at pH close to pl of EW.

## The effect of ultrasound on the rheological properties of emulsions

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Emulsions are heterogeneous mixtures in which two insoluble liquids are dispersed in each other and thermodynamically unstable systems. In the food industry, emulsion based transport systems are used for many different purposes such as protecting the bioactive components from environmental conditions, increasing the stability and the bioavailability of the prepared emulsion, masking unpleasant odors and taste. In recent years, increase in the cost of heat treatment at high temperature and the loss of nutritional value have made an increasing trends towards alternative new technologies. The ultrasound application which has a potential to improve the structural properties of a product is a new research area. With these applications, the water solubility of bioactive components can be increased; the particle size of the dispersed phase may decrease. The purpose of the current study is two-fold. First, to determine some physicochemical properties (color, emulsion stability, emulsion capacity, creaming index) of model food emulsion prepared with ultrasonic homogenization. Second to show the effect of ultrasound application on the rheological properties of the prepared emulsions. It was found that ultrasound application significantly affected the rheological parameters of the samples. The samples showed Ostwald-de Waele flow behavior.  $R^2$  of the control sample was 0.998 and  $R^2 = 0.995$  for the sample with ultrasonic treatment. The effects of ultrasound on the apparent viscosity values at 50 1/s were satisfactorily modeled by a modified power-law model. The consistency index K, flow behavior index n and apparent viscosity  $n_{50}$  of the control samples was 311.5  $\pm$  37.2 mPas, 0.49  $\pm$  0.01, 27.2  $\pm$  0.59 mPas and for the emulsion with ultrasonic treatment was 63.08  $\pm$  6.56 mPas , 0.702  $\pm$  0.02 , 27.97  $\pm$  1.42 mPas, respectively.

## Better foam stability via interplay between $\beta$ -lactoglobulin and HM-pectin

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Proteins can stabilise foam and emulsion interfaces and especially proteins from bovine milk has been studied with particular focus on foaming of  $\beta$ -lactoglobulin (BLG), the major whey protein. The recent studies point the beneficial effects of heat treatment, acidic pH and the presence of polysaccharides on protein-foam stability. The obtained improvement of foam stability via the complex formation between the protein and the proper polysaccharide are extremely exiting. The aim of the current study was to investigate the effects of severe heat treatment of BLG, acidic pH and the presence of anionic high-methoxyl (HM)-pectin on foaming and to pursue the possible relations between the foam stability and the air-water interface viscoelasticity. Measurements were conducted at a protein:pectin ratio of 10:1. In dilute protein systems, heat-treated (90°C, 30 minutes) BLG molecules were able to lower the surface tension at air-water interface at a much faster rate compared to the native BLG while the equilibrium surface tension stayed at a higher value in heattreated case. Foam stability was considerably improved at pH 5.0 in the presence of pectin due to the complex formation between BLG and pectin as evidenced by zeta-potential measurements. Interfacial shear rheology by using a stress-controlled rheometer (DHR-2, TA Instruments) equipped with a Du Noüy ring geometry was performed at air-water interface particularly at the protein concentration relevant to foaming experiments (0.5 %). Results indicated an effect of HM-pectin on the interfacial shear modulus. The yield strain was significantly larger in the presence of pectin as compared with the same system without pectin. Resolving the true relation between rheological properties of statically formed interfacial films and the stability of actual foams would assist both the understanding of those highly complex interfacial phenomena and lead the way to easier prediction of long term stability of food formulations.

## Soft tribology of oil-in-water emulsions: Whey protein versus silica nanoparticle stabilization

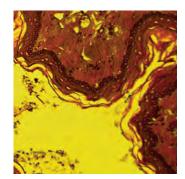
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Tribological behaviour of oil-in-water emulsions was studied in a soft contact of PDMS elastomers used as model oral surfaces. Dependence of friction coefficient on the entrainment speed was followed for a sunflower oil-in-deionized water system stabilised either by whey protein isolate (WPI) or by a corresponding amount of hydrophobised silica nanoparticles by a Mini Traction Machine. We have observed significant differences between the two types of emulsions that were prepared with a similar droplet size. The lubrication behaviour of the particle stabilized emulsions was similar to that of a pure oil phase. This indicates low stability of emulsion droplets under deformation that cause spreading of droplets at the hydrophobic contact area already at high entrainment speeds/lubricious film thickness leading to good lubrication. On the other hand, lubrication of WPI stabilised emulsions was determined by the aqueous phase at the high entrainment speeds and the WPI emulsions were less lubricious than these stabilised by the silica particles. At lower entrainment speeds/film thickness, the friction coefficient of WPI emulsions decreased due to breakage/spreading of emulsion droplets at the contact area and the friction coefficient for WPI emulsions became lower than that for particle stabilised emulsions. The observed tribological differences between the two types of emulsions were related to differences between the WPI and silica nanoparticles in: i) ability to alter wetting properties of PDMS, ii) surface activity at air-water and oil-water interfaces, iii) surface rheology at air-water and oil-water interfaces.







## Cheese, Chocolate, and Health

#### Rheological and sensory properties of instant hot chocolate: Application different multi-criteria decision techniques to determine optimum fat composition

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Physical and sensory properties of instant food products are strongly affected by the ingredients. In particular those physical and sensory properties are highly associated with fat content of the product. Moreover the fat composition of the ingredients such as milk powder and cocoa powder could have significant direct effect to improve physical and sensory properties and acceptance of the products. However there is no experimental study to approve this hypothesis and therefore this study aims to investigate the effect of the fat composition of the main ingredients of hot chocolate. The fat content effects of different levels of fat containing cocoa powder and milk powder was used and rheological and sensory properties of the instant hot chocolate were studied for improvement of fluidity properties of hot chocolate beverage. The wettability, solubility, bulk density, soluble solids, pH, color values, rheological and sensory properties of the samples were determined with 9 samples. The brix, pH, L\*, a\* and b\* values changed between 10.34 - 10.94, 7.4 - 8.2, 4.1 - 15.3, 4.2 - 12.9 and 2.6 - 13.9, respectively. The apparent viscosities of the hot chocolate samples were ranging between 0.015 and 0.025 Pa.s. Samples and the findings of this study showed that, fat content of the milk powder changes the viscosity and the consistency rating rheologically. Lower fat content had a lowered effect on the viscosity. Multi-criteria decision techniques (AHP, SAW, ELECTRE and TOPSIS) were applied to obtain optimum fat content of samples on the sensorial scores. According to the results S2 sample was mostly preferred for Multi-criteria decision techniques. Results demonstrated that fat content of hot chocolate beverages changed the sensory preference and fatty samples were the most favorite hot chocolate in all products.

## Rheo-kinetics of the enzymatic degradation process of thickened fluids for dysphagia patients

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Knowledge of the evolution of the rheological behavior of thickened fluids during the swallowing process is of crucial interest for the safety of dysphagia patients. It is apparent that further studies are needed to have a better understanding of this behavior, aiming to develop safer products for dysphagia management. In this sense, the goals of this research were to characterize the timedependent viscosity-decay that occurs in different thickened fluids upon in-vitro mixing with salivary alpha-amylase, to model the rheo-kinetics of the process, and to assess their potential effects on the swallowing process of dysphagia patients. With this aim, different thickener powders, starch-based and gum-based, were used to prepare a range of thickened fluids according to the stages defined by the American Dietetic Association. An ad-hoc helical mixing geometry fitted to a rotational rheometer was used to measure the viscosity decay of the thickened fluids after alpha-amylase addition. Thus, a small amount of salivary alpha-amylase (selected according to a previously reported protocol) was added and the evolution of torque with respect to the elapsed time (at a constant agitation speed and temperature) was monitored throughout the experiment. The results obtained demonstrate that the rheo-kinetics of this degradation process depends on the type and concentration of thickener, salivary alpha-amylase concentration and mixing intensity. More specifically, starch-based thickened fluids show a dramatic decrease in viscosity as compared to gum-based thickened fluids, which may compromise the safety and comfort of dysphagia patients during swallowing.

#### Influence of alpha-amylase addition on the elongational behaviour of thickened fluids for dysphagia management

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Thickened fluids prepared from powder thickeners are part of the dietary management of dysphagia patients. The aim behind the use of these thickened fluids is to reduce the flow rate at which the bolus moves through pharynx and, hence, to reduce the risk of aspiration. However, it is now acknowledged that extensional rheology may also play an important role in the development of dysphagia-oriented products, since the elongational properties of thickened fluids may affect the characteristics of the swallowing process. Traditional powder thickeners were based on starch. Nevertheless, it is well-known that salivary  $\alpha$ -amylase is responsible for the enzymatic breakdown of starch and other carbohydrate sources found in food and beverages. Consequently, starch-based thickened fluids may undergo a dramatic decrease in their viscosity, and viscoelasticity, upon mixing with saliva in the mouth. Previous studies carried out by some of the authors were focused on the influence of salivary alpha-amylase on the viscous behavior of thickened fluids for dysphagia patients. On the contrary, the goal of this work was to investigate the effect of salivary alpha-amylase on the extensional proprieties of starch-based and gum-based food thickeners used in dysphagia management. Elongational flow behavior and the full filament profile were monitored using a high-speed camera attached to a capillary break-up extensional rheometer (CaBER). Aqueous dispersions of commercially available dysphagia-oriented thickeners (prepared according to the stages defined by the American Dietetic Association) were tested, as well as their blends with salivary  $\alpha$ -amylase (prepared according to previously reported protocols). The filament lifetime and the filament shape were both qualitatively and quantitatively compared. The experimental results obtained provide a clear view of the influence of salivary  $\alpha$ -amylase on the elongational properties of different thickeners, differentiating between gum-based and starch-based thickened samples. Thus, the results demonstrate a dramatic decrease in filament breakup time of starch-based thickeners in the presence of  $\alpha$ -amylase, while gum-based thickeners show a much longer filament breakup time, regardless of the presence of  $\alpha$ -amylase. As a consequence, capillary break-up rheometry may be considered a guick method to further understand the elongational properties of thickened products in the presence of salivary alpha-amylase, and may contribute to a deeper knowledge of the complex rheological behavior of these thickened fluids during the swallowing process.

#### Effect of saliva (mouth digestion) and in vitro stomach digestion on the rheological properties of vegetable purees made with hydrocolloids to enhance satiety

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Food manufacturers have shown increased interest in seeking alternatives to enhance the effects of foods on satiety and subsequent food intake. The addition of hydrocolloids to food products has shown a great efficacy in increasing the feeling of fullness and reducing energy intake. The aim of this study is firstly to develop foods that provide satiety studying the effects of the addition of different types of hydrocolloids in the reformulation of pureed vegetables to get a food with high satiating capacity. Four hydrocolloids (starch, carrageenan, guar gum and hydroxypropylmethylcellulose) have been used to prepare purees. After that, the effect of the saliva and the digestive enzymes on the rheological properties of purees was studied to evaluate the structural destruction of the samples as a function of the hydrocolloid used. Starch puree was the only sample that was degraded by the action of amylase and little differences in the rheological properties on the other samples with digestive enzymes were found. Lastly, the acceptability of the four purees was evaluated by sensory analysis, where the degree of liking of appearance, flavor, consistency, overall acceptability and the expectation of satiety were evaluated.

#### Intragastric rheological properties and their relationships with the satiating capacity of cheese pies with glucomannan

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Konjac glucomannan (KGM) is consistently associated with creating a sense of fullness while slowing down physiological processes associated with the digestion of foods and absorption of nutrients. It is difficult to formulate food with KGM because it develops very high viscosity in aqueous solution. In the present study, cheese pies containing increasing amounts of KGM were prepared in such a way that the gum was not fully hydrated, with the double propose of achieving formulations with high doses of KGM and delaying the development of the gum's rheological properties until it reaches the gastric tract. The pies, the gum alone and a commercial product based on KGM were then submitted to oral plus gastric in vitro digestion and their rheological properties were measured and compared. The viscoelastic properties increased as the KGM content of the pies rose. In addition, the digested pies with KGM were more effective at forming solid-like structures than both the digested gum alone and commercial KGM-based product at the same KGM concentration. The instrumental texture measurements of the pies indicated that higher KGM levels produced harder and more cohesive pies. These texture attributes would potentially enhance their expected satiating capacity since oral processing would also play a role in the elicitation of these expectations.

## The structural properties of puffed breakfest cereals with plant based fibre additives

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Obesity is a major health issue in the developed and, increasingly, in the developing world; indeed obesity is considered the most serious health issue facing the developed world, formally recognized as such by the World Health Organization. Supportive environments and communities are fundamental in shaping people's choices, limit energy intake from total fats and sugars; increase consumption of fruit and vegetables, as well as legumes, whole grains and nuts and regular physical activity the easiest choice (accessible, available and affordable), and therefore preventing obesity. Puffed breakfast cereals from plant based fiber are one of possibilities to developed products with heightened dietary fiber, bioactive compounds and reduced starch content.

The aim of this study was to evaluate the structural properties of Puffed breakfast cereals with hydrolyzed rye bran, apple and pumpkin marc additives. Experiments were done at the Latvia University of Agriculture and Riga Technical University. Samples of puffed breakfast cereals were prepared from wheat, rye, rice flour, rye bran, hydrolyzed rye bran and apple and pumpkin marc. For puffed breakfast cereals structure analyses were used texture TA.XT Plus, Stable Microsystems. For microstructure research Leica DM3000 Microscope system with Leica Application Suite program was used. Starch and proteins are important elements for structural properties of extruded products. Generally for extruded products maize, rice, wheat flour, sometime wheat or rye bran could be used. Enzymatically treated rye bran is ingredient with high nutrition value. During the enzymatic hydrolysis the degradation of cell walls were observed by scanning microscope. Microscopy of the bran samples showed that degradation of cell walls was initiated in the pericarp layer, as well in the starch/ protein matrix [1], it could be influence structural properties of extruded products. Apple and pumpkin marcs are important resources of biological active compounds and dietary fiber. The preliminary results of the present research show the tendency of smaller changes in structural properties for samples with additives, comparing with control sample from wheat and rice flour.

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#### Specific and unspecific adhesion of gut bacteria

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Bacteria adhesion layers are highly complex biological assemblies composed of bacterial cells embedded in an extracellular polymeric matrix. Such biofilms are ubiquitous and, on one hand may cause severe environmental and health problems but on the other hand protect and support the gut flora during digestion. Therefore it is intriguing to understand the influence factors on the formation and the destruction of bacteria biofilms [1, 2]. In this contribution, we study the initial kinetics of bacterial attachment as well as the transient layer formation of model bacteria at both the air/water and oil/water interface through interfacial rheology and tensiometry. Electrophoretic mobility measurements and bacterial adhesion to hydrocarbons (BATH) tests were performed to characterize selected bacteria. To validate interfacial rheology and tensiometry measurements, we monitored biofilm formation utilizing both confocal laser scanning microscopy and light microscopy. Using this combination of techniques, we were able to observe the interfacial tension and elasticity development over time, from the first bacterial attachment up to biofilm formation. Interfacial rheology proved to be a valuable tool for studying biofilms as the influence of temperature, media type, bacterial strain, pH and surfactant concentration could be observed successfully during biofilm formation [1, 3].

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- [2] Wu C, Lim JY, Fuller GG, Cegelski L: Biophysical Journal 103 (2012) 464-471.
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## Is physical strength related to oral processing of food structure?

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The study aims to explain how the physical properties of food are perceived differently depending on the human physical status (oral strengths). To characterize the physical properties, fracture mechanics of 14 commonly consumed food products (fruits, vegetables and dairy origin) were analyzed using a penetration test (Texture Analyzer, Stable Micro Systems, Godalming, UK). Among the different products studied, 6 selected products (carrot, banana, mozzarella, potato, Babybel cheddar, hard cheddar) were given to ten young participants (< 25 y.o.). Physical measurements taken included tongue pressure, (Iowa Oral Performance Instrument, Medical LLC, Redmond, Washington, USA) and bite force (flexible force transducer Tekscan, South Boston, Massachusetts, USA). Participants ranked the food products by the level of difficulty perceived. Additionally, the number of chews and the time at swallow were video-recorded for each participant. Ranking showed that harder products (maximum break point) were related with difficulty perceived (except for the carrot). Other food breaking characteristics (number of peaks and gradient of the penetration curves) showed correlation with mastication time and chewing cycles. Individuals with higher bite force chew less, and those with higher tongue pressure kept the food for shorter duration in mouth. However, no relationship could be established between the bite force and food perception difficulty. Additionally, the relationship of structure to individual's oral strength was rather food-specific. These correlations (bite force/ tongue pressure vs. number of chews/ time at mouth) were observed just in the case of the soft cheeses (mozzarella and baby cheddar), which might be attributed to the soft and cohesive structure being oral-strength dependent, and requires specific oral muscles. In conclusion, relationship of food oral processing to oral strength is driven to a large extent by type of food consumed in healthy young individuals, with the strength being correlated to the mastication time needed to create a swallowable bolus.

## Lipase-induced structural modification of anhydrous milk fat

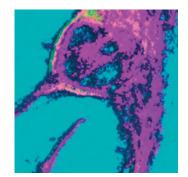
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The majority of the palmitic acid (C16:0) content of human milk is located at the sn-2 (middle) position of the triacylglycerol molecule. This position enhances digestibility and has important implications for infant health. In order to maximize the nutritional quality of infant milk formula lipid ingredients should approximate human milk fat in terms of both composition (total fatty acid content) and structure (stereo-specific distribution of fatty acids). Anhydrous milk fat (AMF) offers considerable potential for use as a human milk fat substitute (HMFS) as it rich in C16:0. In contrast with human milk fat, however, the majority of the C16:0 is located at the sn-1 and sn-3 (outer) triacylglycerol positions. 'Humanisation' of the lipid-structure of AMF through enzymatic action may provide a means of correcting this imbalance. The purpose of this study was to explore the potential of AMF as substrate for lipase-induced, triacylglycerol modification, with emphasis on increasing the proportion of palmitic acid (C16:0) at the sn-2 position. The interesterification behavior of ten commercial lipases was compared through determination of fatty acid methyl esters (FAMEs) and pancreatic lipase-mediated, sn-2 positional analysis. Changes in the proportion of C16:0, at the sn-2 position, were relatively minor with a maximum increase of 9.5 %w/w. More extensive changes at the sn-2 position were observed for fatty acid types other than C16:0. Fractionation of AMF, by controlled cooling, provided a means of increasing the total C16:0 content of the stearin (solid) fraction. Subsequent hydrolysis further increased the amount of C16:0 at sn-2 to 13.24 g/100 g (an 18.2 %w/w increase compared with the AMF control). This study demonstrates that the lipid-structure of AMF can be modified to more closely resemble that of human milk fat. Addition of other oils as both compositional adjuncts and acyl donors appears necessary, however, in order to fully exploit the potential of AMF for use as an ingredient in HMFS.







## Colloidal Dispersions

## Rheological interactions of natural micro-particle food polymers in different temperatures

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Hydrocolloids (gums) are polymeric substances which are soluble or dispersible in water and are used as thickening and gelling agents. Micro and nano-particles are widely used in food industry especially in area of food additives due to their physicochemical and functional properties. In this research, some of the polymers that are used in food industry as stabilizing and thickening agents such as guar gum, xantan gum, carboxymethylcellulose (CMC), pectin and carrageenan were investigated rheologically after particle size reduction. A biopolymer particle size may alter physicochemical properties (optical, rheological and stability), encapsulation characteristics and functional characteristics of foods such as behavior in the gastrointestinal tract. In this study, particle sizes of gums before and after milling were analyzed with Scanning Electron Microscopy (SEM) and rheological properties of gums were determined at four different temperatures 25, 40, 60, and 80°C. Ball milling was performed with a total of 20 minutes and consequently 25 - 72 % size reduction was obtained from different hydrocolloids. For the average particle sizes of pectin before and after size reduction were between 1.89 -122.04 and 1.35 - 22.11  $\mu$ m, respectively. When the rheological properties were examined, it was observed that the consistency coefficient K of 1 % solution of size reduced gums was decreased, whereas flow behavior indexes n were increased with size reduction. Additionally, the viscosity of the polymer solutions was decreased with increasing temperature analyzed by the method of temperature sweep test.

#### Structure properties and crystallization kinetics of diverse palm oils for nougat spread production with small amounts of high melting triglycerides and different particle size distributions of sugar

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Fat crystal networks play an important role in food production. Whereas certain hardness but rapid melting properties are desired for chocolate, good spreadability and a tender mouthfeel are desired for nougat spreads leading to different recipes. Hence network formation properties of palmoils where measured by oscillation rheometry within the linear-viscoelastic-Range (LVE-Range) and coupled with crystallization kinetics by applying the avrami kinetic via isothermal DSC measurements. Three different origins of palmoil where used as first series of measurements for examining effects of different triglyceride profiles. Network formation properties of palmoil can be attributed to tripalmitin. The higher contents of tripalmitin, the higher G' and yield points. Higher tripalmitin content equally leads to higher rate constants (constant of crystallization speed) and avrami exponents which are associated with better crystallization properties and enhanced network formation. All three palmoils can be distinguished by this explanation. Furthermore palmoil with additions of 5, 10, and 20 % palmstearin (source of tripalmitin) where measured. The utilized palmstearin consisted of 21 % tripalmitin. Enhancing tripalmitin by this procedure led to a distinctive increase of G', yield point and an extension of the rate constant) and the avrami exponent in comparison with unmodified palm oil. This displays the dominant role of tripalmitin upon network formation in palmoil and shows pathways of specifically adjusting network formation properties. Last not least two different sugars with the same specific surface area  $S_v$  where mixed with palmoil utilizing 5 % of fine particles  $(S_v = 3.6 * 10^6 \text{ 1/m})$  and 50 % of coarse particles  $(S_v = 0.36 * 10^6 \text{ 1/m})$ . Particle presence in general led to extensions of the rate constant and the avrami exponents compared to unmodified palmoil. G' of both samples is manifold higher then of unmodified palmoil, with 50 % coarse particles displaying the highest G' but with no significant difference of the yield point compared with 5 % fine particles. Screening off the fine particle fraction from the coarse collective led to significant drops of G' and the yield point although amounts of sugar remained equal. Hence the particle size distribution seems to influence network formation of triglycerides, with fine particles displaying better reinforcement character of the crystal lattice.

# Effect of structural parameters on rheological behaviour of apple cells dispersions: A comparison of real systems and model systems.

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Being able to control the texture allows to generate innovative products and to deal with consumer expectations or physical issues, that's why many studies aim at understanding the structure/properties relationship of vegetable based products. Particle content and size are identified as major parameters on vegetable suspensions rheology. The objective of this work is to confirm the structural parameters that control the rheological behavior of vegetable cells dispersions, and to determine the impact of the continuous phase. Two different kinds of apple cells dispersions were prepared. On one hand, a large range of reconstructed-purees varying in particle content and particle size was prepared. A single batch of puree was grounded to obtain three different particle size distributions. Then, a separation-reconstruction step was realized to formulate suspensions in their own aqueous phase varying in particle content. Twelve levels of particle content were reached from twenty time diluted to twice concentrated. On the other hand, model dispersions of apple cells were prepared. To do so, the same batch of apple puree was grounded, wet sieved and dried to obtain dehydrated size controlled apple particles. These particles were dispersed in a controlled aqueous medium to reach the same particle contents as those reached for the reconstructed-purees. Structural parameters are described using laser granulometry and optic microscopy. Rheological behavior was investigated using a large gap coaxial cylinder. This study confirms that solid content and particle size have a major impact on rheological behavior of apple cells dispersions, while the continuous phase seems to have a major impact only in diluted domains. Rheological parameters (apparent viscosity, yield stress, storage modulus) increase with the particle size and the solid content. Different rheological behavior regimes were obtained depending on the concentration of insoluble solids. At low particle concentration (diluted domain), specific viscosity increases linearly depending on the solid content with no noticeable effect of particle size. At higher concentrations, particle morphology and size impact rheological behavior. Specific viscosity becomes more dependent on solid content and two different behaviors are noticeable. Just beyond the diluted domain, a power law relationship between the storage modulus and the solid content appears, corresponding to the establishment of a weak network domain with measurable elastic properties. By increasing the concentration of insoluble solids, the entrance in a highly packed domain is observed.

### Characterisation of protein aggregates in milk protein concentrate

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Pre-heat treatment of proteins is widely used in dairy processing to improve the stability of milk products or ingredients. During heat treatment, soluble aggregates of caseins and whey proteins are formed. A method of purification was developed to analyze the change in the properties of these aggregates as a function of the pre-heat treatment conditions. A reconstituted solution of milk protein concentrate (MPC 80) at 10 % protein was heated at 90°C for 25 minutes (pH 7.2) with or without calcium (2.5 mM). The aggregates were purified by centrifugation and Fast Protein Liquid Chromatography (FPLC). The aggregates were mostly composed of  $\beta$ -lactoglobulin and  $\beta$ -casein at different ratio. The ratio  $\beta$ -lactoglobulin/ $\beta$ -casein and the aggregate sizes were positively correlated to the addition of calcium. Aggregates with smaller particle size had a lower ratio of  $\beta$ -Lactoglobulin/ $\beta$ -casein.

#### Characterisation of the flowability of the instant pudding and baking powder by using powder rheometer

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Powder characterization is one of the important issues in powder handling. With powder rheology detailed flow properties of a powder or powder mixture can be characterized. The characterization of flowing rheological properties in food powders -pudding, baking powders and components [sugar, corn starch, skim milk powder (SMP), cocoa, carrageenan, sodium bicarbonate, citric acid, sodium acid pyrophosphate (SAPP)] - was aimed based on the analysis by powder rheometer attached with texture analyzer. Three tests were applied to determine flow characteristics of the food powders. These were cohesion, caking and powder flow speed dependency (PFSD) tests. Cohesion Test was used to determine the cohesion coefficient and cohesion index. The cohesion coefficient (g.mm) is the negative area under the force/distance curve. Cohesion index is a unit-less value and ratio of cohesion coefficient/sample weight. According to cohesion index values powders were categorized as: free flowing - sugar, (10.42), corn starch (9.61), SMP (9.68), sodium bicarbonate (9.86) and citric acid (9.71); easy flowing – carrageenan (13.98), SAPP (14.71) and baking powder (12.20); cohesive – pudding (14.38); extremely cohesive-cocoa (20.29). Caking is the tendency of powder to form large agglomerates during storage and transportation. In the caking test, cake height ratios were recorded. Increasing cake height ratio indicates the powder has a high tendency to form cake, and unchanging cake ratio means little or no tendency to cake. The highest rise in the cake height ratio between the first and the last cycles were shown by cocoa and SAPP as 0.269 and 0.310, respectively. Powder flow properties may change with increasing or decreasing flow speeds. Flow stability values which gives important information about the flow resistance of powder and provides an indication of susceptibility of the product to attrition were recorded. Flow stability values of corn starch (1.00), carrageenan (0.99), sodium bicarbonate (1.00) and SAPP (1.01) were closed to 1 and meant no changes for powder breakdown during the test, but it was greater than 1 for sugar (1.11), cocoa (1.14), SMP (1.04) and citric acid (1.08), and less than 1 for pudding (0.97) and baking powder (0.94) samples. Thus, their susceptibility to attrition must be taken into account in the process.

## Composition, morphology and pasting properties of orchis anatolica tuber gum

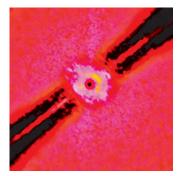
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Orchis anatolica (O. anatolica) tuber is commonly used in the production of Salep or O. anatolica tuber gum (OaG) as a hydrocolloid ingredient. The aim of this study was to investigate the composition (starch, glucomannan, protein, ash, moisture and fat), morphology and pasting properties of OaG. Morphological analysis and pasting properties were carried out by scanning electron microscopy, light microscopy and rotational rheometry. OaG was composed mainly of starch (35.5 %) and glucomannan (18.5 %). The OaG ingredient powder had irregular shaped particles with smooth surfaces. After pasting treatment, the initial and final viscosity values of the OaG dispersions at a concentration of 0.5 % OaG were 33.7  $\pm$  0.24 to 34.3  $\pm$  0.45 mPas whereas the corresponding values at a concentration of 2.5 % OaG were 1193  $\pm$  92.0 and 1437  $\pm$  83.3 mPas, respectively. O. anatolica tuber gum, a complex biopolymer, can provide interesting and unique functionality to the food industry in the development of novel food structures.







## Rheological Modelling and Numerical Methods

#### Modelling the growth of bubbles in a range of non-isothermal generalised Newtonian fluids

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The dynamics of bubble growth and collapse in response to external changes of temperature and pressure is of great importance to many parts of industry, including polymeric foam blowing and the processing of bubbly foods. The objective of any industrial study would be to be able to tailor the bubble microstructure created by a process by altering the process parameters in predictable ways. In many cases the fluid will exhibit a complex rheology, which will affect the manner in which it will be deformed as it is forced out of the way of a growing (or shrinking) bubble. Hence the models used to predict bubble dynamics must be able to account for these effects, instead of assuming Newtonian behavior. The problem is usually approached in one of two ways, where a single bubble is modeled in either a finite or infinite medium of surrounding fluid. While a greater range of fluids have been studied in an infinite medium, the problem with this approach is that a final equilibrium cannot be reached if there is dissolved gas in the fluid. A model has therefore been derived which accounts for the diffusion- and temperature-controlled bubble growth and shrinkage in a finite medium of any generalized Newtonian fluid. This is achieved by identifying the differential equations which govern conservation (in the bubble) and diffusion (in the fluid) of mass, energy, and momentum, and then solving them by discretizing the fluid space and time stepping from the initial conditions toward a solution. The model was validated in the Newtonian case against results from an existing model, and then tested using power law and Carreau-Yasuda fluid models. It was found that the predominant difference of any generalized Newtonian fluid model is determined by the extent of viscous thinning exhibited by the fluid, which manifests most when the bubble is small and fast growing. The constitutive equation does not affect the size of the final equilibrium unless it includes a yield stress. Preliminary work has been done to allow the model to accept Bingham and Herschel-Bulkley fluid models to investigate this further.

## Fractional equations for the firmness, rubberiness, and springiness of food gels

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Constitutive models for soft solids that relate the stress to the deformation history have the potential to be used in a structure-texture engineering context, but successful examples are scarce. In the present work we define equations for the firmness F, springiness S, and rubberiness R, of semisoft food gels that exhibit broad power-law stress relaxation over a wide range of timescales. The equations contain only two material properties, which have their origin in the food micro-structure: a fractional exponent, which quantifies the frequency and temporal response and secondly a scale factor or "quasi-property", which sets the magnitude of the stress in the material. Together they form a constitutive element, known as the 'springpot' or Scott Blair element. This model correctly predicts the time- and interdependency of the firmness, springiness and rubberiness of emulsionfilled hydrocolloidal gels such as full- and reduced-fat semi-soft cheeses. Firmness and rubberiness are time-dependent properties, whereas springiness is not. The magnitude of the firmness and springiness are inversely related through the fractional constitutive model. Our equations for F, S and R also correctly predict the effect of changing the magnitude or time-scale of the stress loading on the material even in the case of irreversible flow events, when cheese progressively transitions from a solid to a liquid. Finally we show how our FSR-equations can be used in a texture engineering context; they guide product reformulation studies and allow for extrapolation of a firmness measurement to practical situations where the gel is subjected to prolonged creep loading.

## Particle-filled meat protein gels: Investigating the role of particle size and protein/filler interactions

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Finely comminuted meat protein products such as frankfurters and bologna can generally be regarded as myofibrillar protein gels containing a homogeneously distributed particulate fat phase. The purpose of this investigation was to elucidate how different filler properties influence the large deformation mechanical properties of such products. The role of filler/matrix interactions was investigated by using fillers with contrasting surface chemistries: hydrophobic rice bran wax particles and hydrophillic glass beads. Additionally, various particle size ranges (< 100  $\mu$ m to > 1000  $\mu$ m) were used to determine the influence of filler size. All particle types and sizes were incorporated into the gel system over a broad concentration range (0 - 0.5 volume fraction filler  $\phi_f$ ). RBX particles were found to be strongly bound to the gel matrix, whereas glass beads were only weakly bound to the continuous phase, as observed by cryo-SEM. The Young's modulus as a function of  $\phi_f$  was strongly influenced by particle size, where a reinforcing effect was seen for all but the largest glass beads (> 1000  $\mu$ m diameter). Here we have compared two well established models first presented by van der Poel and Kerner, respectively. We have found that the exact solution to the modified van der Poel model best describes the behavior of the modulus for the tightly bound wax-filled gels at all size ranges if the volume fraction filler is replaced by an 'effective' volume fraction  $\phi_{eff}$ , where  $\phi_{eff} = \alpha \phi_f$ . This  $\phi_{eff}$  was found to increase with decreasing particle size and may account for inhomogeneous particle distribution and the increase in surface area due to the non-spherical nature of the chopped wax particles. In contrast, the behavior of the weakly bound glass-filled gels were best described by a modified form of the Kerner equation which introduces a maximum packing fraction to account for particle crowding  $\phi_{max}$ .  $\phi_{max}$  was also found to be inversely correlated to particle size range, and approached the theoretical limit of close packed uniform spheres with decreasing particle size. This work gives insight into the influence of matrix/filler interactions, as well as filler size on the large deformation properties of comminuted meat products.

#### Computational fluid dynamics for pasta extrusion

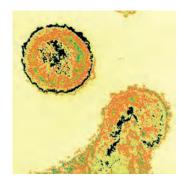
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Pasta properties and pasta production efficiency are related to the fluid flow through the pasta press. The raw materials are mixed and kneaded to form the fresh dough, that finally flows through the crosshead and the die. To offer high quality pasta products and high efficiency production computational fluid dynamics (CFD) is used to understand and optimize the pasta extrusion process, in particular through the crosshead and the die. To this end we have characterized and modeled fresh pasta dough with a moisture content of 28 %, which is highly viscous, shear-thinning and has a low heat transfer coefficient. Although pasta dough is viscoelastic, the Reynolds number of typically  $10^{-5}$  is too low for it to affect the flow, e.g. die swell could not be observed. Moreover, the process exhibits wall slip behavior when extruded through PEEK or TPFE dies. Shear viscosity and wall slip were modeled as power-laws fitted to capillary rheometer measurements, that were obtained with dies made of steel or PEEK, and with varying diameter-to-length ratios. Additionally, an Arrhenius correction was used to account for temperature effects. Using ANSYS CFX, the material model was successfully validated against capillary rheometer data, prior to be used in a simulation of a lab-sized industrial pasta press. The CFD results showed good agreement with experimental observations, and eventually allowed to understand temperature and extrusion velocity variations.







## Semi-Solid Foods

## Investigation of rheology of semi-hard cheeses using fluorescence spectroscopy and chemometrics

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The purpose of this study was to evaluate the feasibility of fluorescence spectroscopy to investigate the rheological properties of semi-hard cheese with 20, 45, and 55 % fat in dry matter (FDM) contents. Rheological parameters as storage modulus G', loss modulus G'' and shear stress were determined in the three different regions. Yield stress, stress and LVE (Linear Viscoelastic) regions were tested at 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70°C. Tryptophan, vitamin A emission and vitamin A excitation spectra were recorded at the same temperature ranges (25 - 70°C). For each cheese sample, the data sets containing fluorescence spectra, rheological data and chemical parameters were evaluated using principal component analysis (PCA), partial least squares (PLS) regression and partial least Squares discriminant analysis (PLSDA). High correlations were observed between the rheological parameters, chemical parameters and fluorescence values of cheese samples. Thus, it was concluded that fluorescence spectroscopy in combination with chemometrics has a potential as a fast, accurate method for the analysis of rheological parameters and chemical composition of cheese.

## Influence of chemical and biochemical parameters on texture of Appenzeller cheese

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Texture properties of Appenzeller cheese are very important quality parameters. Appenzeller cheese makers association request that optimal texture quality is characterized by a soft structure that lasts over at least three months. During winter production, texture related problems were sporadically monitored. In comparison to the sensory assessment of cheese texture, the uniaxial compression test yields absolute values and thus facilitates the discrimination of optimal and inaccurate texture properties of cheeses. The water content is known as "the softener" in cheese, but additional multivariate factors such as fat and calcium content significantly influence the texture of cheese. Moreover, the status of the calcium-protein-framework is important. In the present study 10 Appenzeller cheese loafs with optimal sensorial texture aged for 120 days and 10 loafs with dry and firm texture of the same age were selected and further analyzed with a uniaxial compression test. All cheeses had been produced in the same week during winter (January) in different cheese factories and ripened for four month in commercial cheese aging facilities. In addition to texture analysis, 18 chemical parameters and 6 biochemical parameters were analyzed. In a first step, a t-test between the two groups (optimal and inaccurate quality) was performed with rheological data (uniaxial compression test, stress at 33 % strain), chemical and biochemical parameters. All parameters with significant differences were in a second step integrated into a multivariate general linear model (GLM) with backward elimination in order to identify the most important parameters influencing texture. Three key factors (water and calcium content and the ratio of water soluble nitrogen to total nitrogen (WLN/TN)) were found to influence the texture properties of Appenzeller cheese.

## Effect of storage conditions on quality characteristics of dietary biscuits made with currant paste

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Short-dough biscuit is a well-known food product, consisting of three main ingredients (flour, sugar and fat). Its high sugar content is against the trend of a healthy lifestyle and therefore its substitution would be desirable. The black Corinth Raisin (currants) is a dried fruit cultivated in Greece, with high nutritional value, rich in dietary fibers, minerals, vitamins, poly-phenolic antioxidants and other phyto-nutrients. Due to its high content in fructose (> 35 %wt), currants are classified among the foodstuffs of medium glycemic index and can be consumed even from specific parts of population like diabetics. In this research the effect of currant-paste incorporation on quality characteristics of biscuits was studied over a three month storage period. Three types of biscuits were produced by substituting sugars with currant paste at 0 (control), 50 and, 100 % level. Biscuit samples were packaged in laminate and polypropylene bags and stored at 25, 35, and 45°C (accelerated shelf life test) for three months. Physical, mechanical and sensory properties of the biscuits were evaluated at predetermined storage time intervals. More specifically the properties examined were dough's apparent biaxial extensional viscosity, spread factor (SF), porosity, apparent density, surface color, breaking strength, moisture content, water activity, sorption characteristics and peroxide value (PV). Besides, a sensory analysis of biscuits was carried out by 10 trained panelists. Sensory attributes included appearance, color, taste, texture and overall acceptance. During storage, PV and moisture content increased while breaking strength of biscuit samples decreased, depending on the packaging material. Adding currant-paste in biscuits leads to products with remarkable nutritional value and highly accepted sensory attributes. In conclusion, the incorporation of currant-paste in biscuits is a promising alternative in the field of bakery products.

## Effect of carob flour particle size on the gelling properties of rice flour

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The demand for high nutritional and taste quality gluten-free products has considerably increased in recent years due to an increase in diagnosed celiac patients. Gluten-free products lack many important nutrients, since are commonly made from rice flour or other refined cereal starches. A possibility for increasing the nutritional value of gluten-free products could be carob flour, due to its nutritional characteristics. The challenge is to understand how the gluten-free flours behave under food processing conditions and mainly baking since gels are formed and their final structure and texture properties are strongly dependent on processing temperature/time, cooling rates and flour composition and are known to affect the texture of finished products. Analysis of pasting behavior and viscoelastic properties is a useful method to determine the properties of these systems. The purpose of this study is to evaluate the effect of carob flour amount and particle size on the gel-forming kinetics during thermal processing and stability of carob flour-rice flour systems. Gels (6 %wt) were prepared using rice flour (Rf) and four carob flour fractions (Cf) at several ratios Rf/Cf = 95/5, 90/10, and 85/15. Carob flour fractions were named as Cfa ( $d_{43} = 292.74 \ \mu m$ ), Cfb  $(d_{43} = 216.98 \ \mu m)$ , Cfc  $(d_{43} = 166.84 \ \mu m)$  and Cfd  $(d_{43} = 139.31 \ \mu m)$ . Differences in chemical composition among carob fractions were observed, with protein content being higher in finer fractions, unlike dietary fibre content, which is higher in coarser fractions. Gels made with carob fractions richer in dietary fibres (namely Cfa and Cfb) reported higher values of peak viscosity compared to control sample (only rice flour) and gels with Cfc and Cfd additions. The addition of all carob fractions leaded in gels that did not exhibit breakdown (the ability of the gels to withstand heating at high temperature and shear stress) compared to control sample. The increase of viscosity during cooling or setback (related to the crystallization of starch), was increased for Cfc and Cfd addition (richer in proteins) compared to control samples, since it could be affected by the reorganization of the denatured protein.

## Rheological investigation of starch based edible coatings for ready-to-eat potato production

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Edible coatings are interesting solutions to improve the shelf life of different vegetal systems; the product is coated with a very thin layer of edible materials able to build a barrier against moisture loss and gas exchange. Among the different potential agents, used for edible coatings, starch is widely used because it is already present in many vegetables and it is able to avoid potential alterations of the original taste of coated products. Anyway the coating properties are strongly dependent on starch characteristics and on adopted formulation. In the present work potato starch was extracted from different local potato cultivars and it was used to prepare edible coating for ready-to eat potato slice production. A preliminary physical characterization of extracted starch was carried out by determining rheological properties and contact angle of starch solution, with the aim of investigating potential differences among the different starch sources. Moreover, the results of this preliminary investigation were used trying to optimize the edible coating formulation adopted to improve the shelf-life of portioned potato slices. Experimental results evidenced the positive effects of selected coating formulation on potato shelf life.

#### Relationships among small-strain rheological behavior, large-strain rheological behavior, and structure of different mashed potato preparations

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Mashed potato products are a popular side dish in the United States. Texture is a major driver of consumer liking of mashed potatoes and other potato products. Therefore, texture is important quality attribute of potato products. However, training and maintaining a sensory panel to evaluate textural attributes can be expensive and time-consuming. As an alternative, food textural attributes can be inferred through rheological evaluation, reducing testing time and cost. The objective of this study was to determine the rheological behaviors of different formulations of mashed potatoes (made with whole potatoes, instant potato flakes, pure potato starch, or freeze dried potatoes). Smalland large-strain rheometry, including large amplitude oscillatory shear, and confocal microscopy imaging were performed to evaluate potato mechanical behavior and structural contributions to that behavior. Several relationships were found between mashed potato rheological behavior and structural damage to the potato starch granules. Whole and freeze-dried potato samples exhibited a greater extent of elastic behavior than the potato starch and instant potato samples during smallstrain rheological evaluation. In large-strain rheological testing, the instant potatoes and potato starch exhibited reduced nonlinear behavior and increased viscous-type behavior as compared to the whole potato and freeze-dried samples. These results were likely due to the difference in the amount of starch granule damage among the samples. Instant potatoes and potato starch samples showed a greater degree of starch granule breakage than freeze-dried and whole potato samples. Whole potato samples showed little starch granule damage. While freeze-dried samples did not show extensive starch granule breakage, microfractures were present on the surface of their starch granules. It was hypothesized that these microfractures allowed amylose strands to leach from the granules without destroying their structural integrity. The free amylose formed a gel, creating a weak network that reduced the extent of viscous behavior. Based on these results, mashed potato rheological behavior (and therefore texture), as well as the rheological behavior and texture of other starch-based food products, can be manipulated by controlling the extent of starch damage and the amount of free amylose.

## Physicochemical and viscosity of honey from Côte d'Ivoire

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The rheological properties of honey are important parameters that influence its organoleptic quality and also affect a number of technological operations (honey heating, mixing, filtering, hydraulic transport and bottling). In Côte d'Ivoire, honey is produced from wild bees as well as beekeeping. It is used for nutritional and medical purposes. More attention is paid to beekeeping to transform honey into an economic alternative. Taking into account quality parameters is essential for achieving this goal. The present study aims at determining the physicochemical characteristics and viscosity of honey produced in Côte d'Ivoire. Different honey samples were bought on the market for determination of moisture content, specific gravity and soluble sugars content. The viscosity was also measured at 50°C. Results showed that the mean values of moisture content, specific gravity and soluble sugars were 18.9%, 1.5, and 81.2% Brix, respectively. These parameters fall within the international standard for honey (Codex Stan 12-1981). The viscosity at 50°C was 6.9 Pas, suggesting high viscosity upon heating. This property could be benefit for technological applications. Further investigations will focused on thermal properties and flow behavior of the product.

#### Factors affecting shelf life and quality attributes of currant-paste

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The black Corinth raisin (currants) is a high nutrient variety of raisins, cultivated in Greece, which is recognized for its potential effects on health. Since currants contain occasionally undesirable microscopic seeds, the development of a new product, such as a seedless paste, would result to currants' upgrading. Shelf life of currant-paste is mainly determined by its sensory characteristics that tend to degrade during storage as a consequence of sugar crystallization and hardening of currant-paste. The aim of this research was the study of the factors affecting the shelf-life of currant-paste stored in three different temperatures (15, 25, and  $35^{\circ}$ C) for a prolonged storage period (1 and 3 days, 1, 2, 3, 4, and 6 weeks, 2 months) and the effect of plasticizers addition such as glycerol and water incorporated in various concentrations (0, 5, 10, 15 % water; 5, 10, 15 % glycerol; 15 % water and glycerol). All samples of currant-paste were evaluated for their physicochemical (moisture content and water activity) and mechanical properties (hardness, cohesiveness etc.) using texture analysis profile (TPA). Additionally, a sensory analysis was conducted by 13 trained panelists who carried out hedonic and intensity sensory tests of characteristics, along with microbial growth measurements of total viable, yeast and mold count. Statistical analysis was also performed using SPSS version 20.0 software. Although moisture content remained fairly constant for all samples, an increase of water activity during storage was observed. The hardness of all currant-paste samples exhibited a strong positive correlation with the storage time period. In contrast, the increase of storage temperature and the incorporated amount of plasticizers exhibited a negative effect on currant-paste hardness. Textural measurements were directly correlated to sensory evaluation and hardness was proved to be the crucial factor for currant-paste's acceptability. Microbial growth didn't seem to mainly affect the shelf life of currant-paste, excluding the samples with high moisture content. In conclusion, the development of seedless currant-paste production process seems to be a highly promising technique for upgrading the value of currants with undesirable quality characteristics.

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