international symposium on food rheology and structure

ISFRS 2019

ABSTRACT BOOK OF THE 8TH INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

JUNE 17 - 20 2019
ZURICH
SWITZERLAND

EDITORS:
PETER FISCHER
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Eidgenössische Technische Hochschule Zürich
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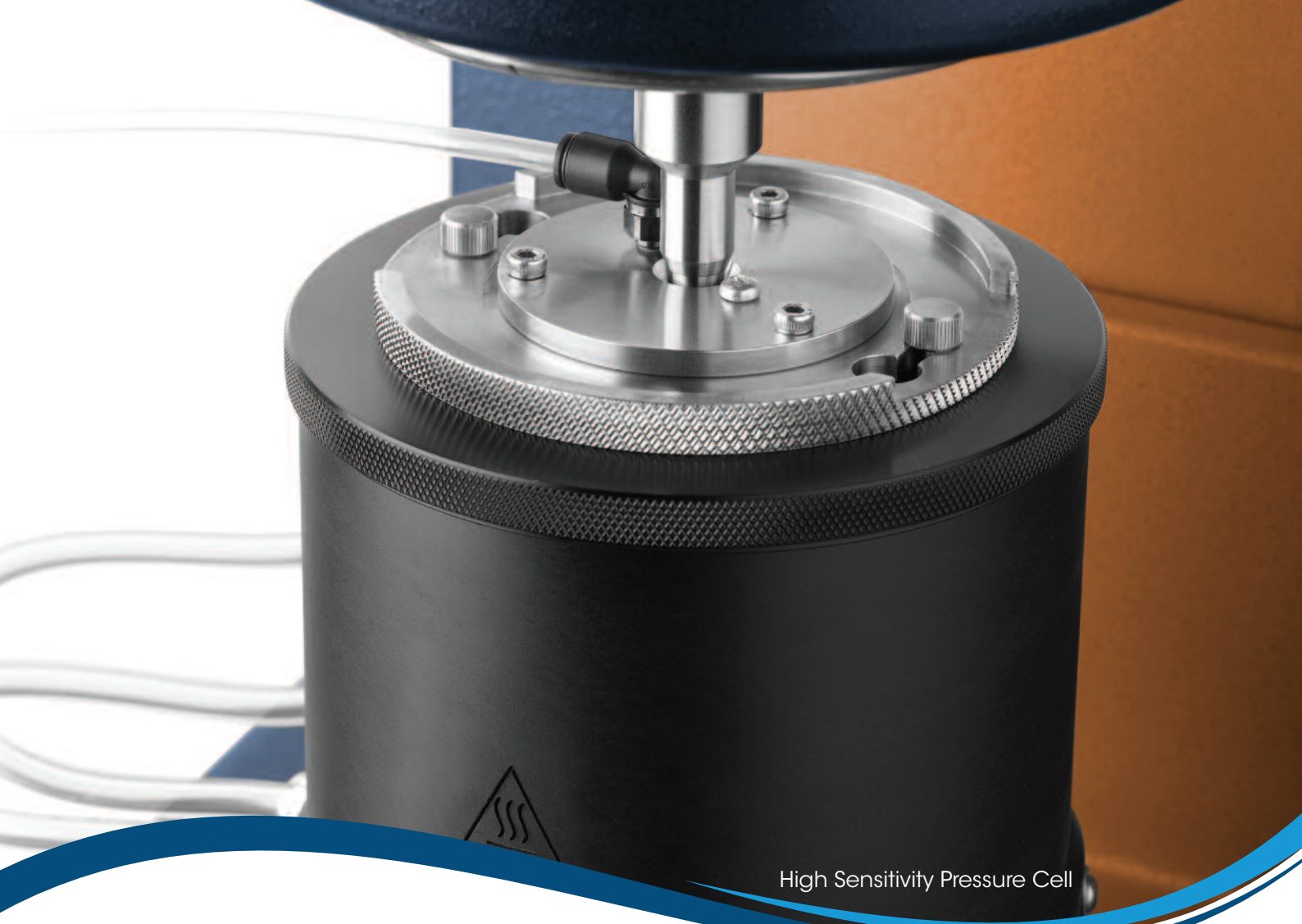
Laboratory of Food Process Engineering
Institute of Food, Nutrition and Health



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Abstract Book of the 8th International Symposium on Food Rheology and Structure

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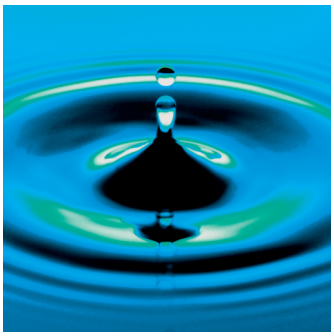

Opening Lecture

Perception of food structure during oral processing: How material properties translate into texture perception

E. Allen Foegeding

Department of Food, Bioprocessing, and Nutritional Sciences, North Carolina State University, Raleigh, NC 27695, United States

The senses of sight, sound, smell, taste, and touch are used to various degrees in the evaluation of food quality. While color, aroma, and taste can be explained at the molecular length scale, touch or texture derives from evaluation of physical properties that are related to structures across meso and macro length scales. Moreover, it is often the temporal changes in physical properties that produce the textural cognition. The science of food texture has underpinnings from a range of disciplines. Food structure starts with the chemical properties that produce interactions among molecules producing structural elements. Physical properties of food structures are determined using methods and approaches from rheology, fracture mechanics, tribology, and pressure sensitive adhesion. The biology of texture is diverse, from the basic understanding of feeding in mammals, to health aspect associated with dentistry, and finally with the physiology and psychology of sensory evaluation of texture. For decades, there has been a desire to have a method or methods that predict the physical properties sensed by physiological elements during sensory evaluation of texture. The relatively recent approach of using structural transformations during oral processing as a framework for understanding textural perception has produced a renaissance in the science of food texture. Moreover, the current desire to increase plant-based foods in the diet, and the coinciding development of plant-based forms of traditional animal-based foods, has generated an immediate need to understand how food structures generate specific textural properties. This talk covers historic and current understanding of texture perception and measurement and concludes with a look towards the future

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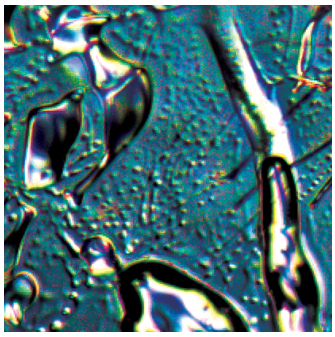
Symposium BS



Biopolymer Solutions and Gels

Readdressing theoretical approaches for modelling food protein gels as particle-filled soft solids

Andrew J. Gravelle, Reed A. Nicholson, Shai Barbut, and Alejandro G. Marangoni

Department of Food Science, University of Guelph, Guelph, Ontario N1G 2W1, Canada

Various fat-containing foods such as dairy products (cheese, processed cheese products, yoghurt, etc.) and emulsified meats (e.g. bologna and frankfurters) consist of a dispersed fat phase embedded in a protein matrix. In such systems, the fat droplets often play major functional roles, influencing the structural, rheological, and sensory properties of the food product. These foods can be described as particle-filled soft materials, and well established particle reinforcement theories are often used to describe their rheological and mechanical behavior. However, it is generally recognized that existing models provide an incomplete description of food systems, as they do not consider aspects such as filler size, clustering, or imperfect interfacial adhesion. In this work, we propose an empirically-derived model which accurately describes the impact of incorporating model fillers on the elastic modulus of a particle-filled food protein gel; heat-set whey protein isolate (WPI) gels filled with glass microspheres of distinct size ranges. The proposed model provided excellent fits of experimental data (R^2=0.97), and more accurately followed the observed trends in reinforcement than established theories. Increasing filler size and associated polydispersity resulted in a reduction in the observed reinforcement. This effect was attributed to an improved filler packing efficiency, which was explicitly expressed in the model through a maximum packing fraction term. Increasing the ionic strength of the WPI gels via addition of NaCl caused a decrease in the extent of filler-matrix interactions. We further demonstrate the empirical model could be adapted to incorporate imperfect interfacial adhesion by combining contributions of bound and unbound fillers using a weighted average approach. We are presently exploring methods of deriving the proposed model through a more rigorous mathematical approach which could be used to optimize the contribution of the fat phase in (for example) reduced fat emulsion-filled gels.

Mixtures of xanthan gum with locust bean gum, guar gum and konjac glucomannan and their molecular interactions in cold gelled systems

Christine Schreiber[1], Marta Ghebremedhin[1], Birgitta Zielbauer[1], Natalie Dietz[2], and Thomas A. Vilgis[1]

[1]Max Planck Institute for Polymer Research, Mainz 55128, Germany; [2]Application Technology, Jungbunzlauer Ladenburg GmbH, Ladenburg 68526, Germany

Xanthan gum (XG), locust bean gum (LBG), guar gum (GG) and konjac glucomannan (KGM) are common ingredients used to optimize the viscoelastic properties of food, body and personal care products. When XG is mixed with LBG, GG or KGM a synergistic effect is observed in the thickening properties of these mixtures. Synergism between hydrocolloids is assumed to be primarily due to hydrogen bonding, flexibility of the polymer backbone and the charge density of the polymers. However, the exact molecular mechanisms underlying the physical origin of the synergism are hitherto not understood. Understanding the physical basis of the synergistic interaction helps to control the textural and rheological properties of hydrocolloid gels more precisely. To understand the synergy during gelation it is necessary to investigate the xanthan-hydrocolloid solutions in the non-heated state because it sets and controls the preferred initial conditions on the given interactions by chain stiffness, charge and polarity under different concentrations. In this study we investigated the interactions of XG with LBG, GG and KGM on a molecular level using visual observation, rheology and AFM measurements. It was observed that the solutions separate after a certain time which indicates that the mixed state does not correspond to the equilibrium of the blends. The rheology measurements of elastic and loss moduli showed the highest synergism in the mixtures of XG-KGM followed by XG-LBG and XG-GG. The ratios for the highest synergism also varied between the mixtures. For XG-GG there is no specific maximum, for XG-LBG and for XG-KGM it is around 20:80. From the AFM micrographs it was observed that XG-KGM gave most homogenous mixtures, whereas XG-LBG and XG-GG showed strong phase separation. Based on our experimental results and the characteristics of the molecules such as molecular size, shape and side chains we propose molecular models to explain the physical interactions in these systems which are supported by atomic force microscopy.

Microstructure influence on rheology of high acyl gellan and maltodextrin mixed gels

Kelsey M. Kanyuck, Tom Mills, Ian T. Norton, and Abigail B. Norton

School of Chemical Engineering, University of Birmingham, Birmingham, United Kingdom

Mixed biopolymer gels allow for the development of novel properties that neither polymer alone could create. Applications in the food industry include creation of novel texture, mimicking of traditional foods with improved nutritional profiles (low and no-fat), and controlled release functionality. For the first time, this work examined a mixed-gel of high acyl gellan (native) and maltodextrin (potato DE2) and demonstrated a range of physical properties with a proposed interpenetrating network. Material properties of quiescently set composite gels were examined by categorization of the network type and allocation of the contributions of each polymer. Characterization was conducted through bulk fracture, small deformation rheology, DSC, proton relaxation NMR, and microscopy. Independently, high acyl gellan formed a highly deformable elastic gel at 0.5 % and higher, while maltodextrin was firm and brittle at 30 % or higher and paste-like below 30 %. At concentrations which did not form a gel, solutions remained clear without indication of phase separation. By adding maltodextrin (from 0 to 35 %) at a constant 0.75 % gellan, the gel true strain at fracture decreased from 0.52 to 0.25 while the true stress increased from 35 to 143 kPa. Most mixed gel analysis has been based on phase separation models, but this mixture does not fit these models and instead an interpenetrating network is suggested. It is hypothesized that gellan forms a network first and maltodextrin aggregates within the pores and restricts movement and flexibility. At concentrations below gelation of one polymer, aggregates still influenced junction zone size or flexibility and modification of water partitioning within the system. In combination with rheological measurements, microscopy and DSC will be discussed to detail the influence of polymer microstructure on mixed-gels and support the proposal of an interpenetrating network.

A novel scheme to model non-Fickian diffusion in heterogeneous food hydrogels

Leonard M. Sagis

Physics and Physical Chemistry of Foods, Wageningen University and Research, Wageningen 6708 WG, The Netherlands

Diffusion in food gels is often non-Fickian, as a result of interactions of diffusing components with the matrix, and swelling. For heterogeneous gels local variations of affinity with the matrix are difficult to capture with existing models, particularly when the length scales of the heterogeneities are much smaller than the macroscopic scale. We present a scheme to derive models for the mass flux in such systems, in which interactions with the matrix are captured through combinations of basic elements describing sorption and diffusion, similar to how viscoelasticity is described through combinations of springs and dashpots. We have applied this scheme to digestion of oil in alginate gels by lipase. In recent experiments we found a markedly different scaling for the propagation of lipase into the gel, than the well-known scaling for Fick diffusion. A sharp front was observed which moved through the gel linear in time. In homogeneous gels this is typical for Case II diffusion, and associated with swelling. However, our gels did not swell. To explain our observation we need to consider the motion of lipase through the gel. When it enters the gel, it starts to diffuse through its pores, which can be described by Fick's law. When the molecule encounters an oil droplet, it adsorbs at the o/w interface, and digests the oil. During the adsorption phase the enzyme is in fact "reversibly stored" in a subsystem of the gel. When we describe this process by a sorption element in series with a Fickian diffusion element, we obtain a Maxwell-type equation for the mass flux, and the mass balance for lipase takes the form of a Telegraph equation, predicting front-like propagation of lipase, linear with time. We extend this scheme to other heterogeneous gels, and derive equations for the mass flux, equivalent to the Kelvin Voigt, Jeffreys and Burger model. For gels which swell, we show how the sorption-diffusion scheme can be coupled to spring-dashpot schemes, giving equations which describe propagation of swelling fronts.

Acid gelation of enzymatically cross-linked caseinates: Relationship between molecular characteristics, rheology and gel microstructure

Norbert Raak[1], Raffaele Andrea Abbate[2], Susanne Boye[2], Albena Lederer[2], Harald Rohm[1], and Doris Jaros[1]

[1]Chair of Food Engineering, Technische Universität Dresden, Dresden, Germany;
[2]Leibniz-Institut für Polymerforschung Dresden e.V., Dresden, Germany

Microbial transglutaminase (mTGase) catalyses the formation of covalent isopeptide bonds between Gln and Lys residues of proteins and thereby affects techno-functional properties. In this study, structure-function-interrelations of cross-linked caseins are elucidated by molecular characterisation, rheological studies and evaluation of gel microstructure. Sodium caseinate (NaCn), acid casein in 0.1 M phosphate buffer (Cn-PB) and calcium caseinate (CaCn) served as model substrates with different ionic milieus. Using field flow fractionation, we demonstrated that casein molecules in Cn-PB associate to elongated particles with Rg~16nm, which are internally cross-linked and transformed into more compact and spherical structures during incubation with mTGase [1]. Dissociating SEC-MALS showed that casein polymer size increased according to the particular effects of ions on casein association in the order NaCn < Cn-PB < CaCn, and that size and molar mass of casein polymers in one system remained constant after 3h incubation [2, 3]. mTGase incubation for 24h increased the number of isopeptide bonds but decreased the flexibility of the cross-linked casein particles, leading to reduced gel stiffness in the presence of ions [3]. Increasing the NaCn concentration in the range 10 - 250 g/kg resulted in a liquid-to-solid transition at a critical concentration, which decreased with prior cross-linking for 3h but increased again with incubation for 24 h [4]. This is in agreement with the tan d of acid caseinate gels at 27 g/kg showing a minimum at 3 h incubation [3]. Microstructure parameters of the gels obtained from forced syneresis experiments and diffusing wave spectroscopy were related to both G' and tan d, and additional evaluation by CLSM is under progress.

[1] Abbate et al.: Food Hydrocoll. (2019) doi:10.1016/j.foodhyd.2019.01.043
[2] Raak et al.: Food Biosci. (2019) doi:10.1016/j.fbio.2019.01.016
[3] Raak et al.: Food Hydrocoll. 86 (2019) 43-49.
[4] Raak et al.: Manuscript in preparation

Using low frequency 1H-NMR and digital microscopy to describe yogurt gel structure and serum entrapment

Audrey Gilbert[1], Laurie-Eve Rioux[1], Daniel St-Gelais[2], and Sylvie L. Turgeon[1]

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Investigating yogurt's water retention is a challenge. In dairy gels, this is generally measured by centrifugation (induced syneresis). Low-frequency NMR (1H-LF-NMR) is a non-destructive technique to potentially better understand syneresis phenomena. This study aimed to understand the link between yogurt microstructure and syneresis using 1H-LF-NMR. Experimental dairy protein solutions (micellar casein, whey protein, and reconstituted milk), experimental yogurt from commercial pasteurized skim milk, and commercial stirred yogurts were analyzed. After a mathematical transformation of the signal, hydrogen atoms pools were differentiated according to their mobility. Each hydrogen pool stood for a type of water mobility in the matrices characterized by a relaxation time (T2(i)), and a signal intensity (I2(i)). Yogurt water retention was assessed by induced syneresis (centrifugation) and structure was characterized using digital microscopy. During induced syneresis measurement, it is not possible to differentiate spontaneous syneresis from the serum that was expelled during centrifugation. Low frequency NMR detected four different water mobility groups in matrices. Among these, there was a signal from the serum, and another one only found in yogurts that came from separated serum (spontaneous syneresis). In yogurts, serum mobility is reduced with the increase of protein content or the increase with the protein network density. In commercial yogurt without gelatin, having a dense and homogeneous network, induced syneresis and serum mobility were low. In experimental yogurts, for which serum was separating spontaneously, induced syneresis was higher than 50 % agreeing with spontaneous syneresis detected by 1H-LF-NMR. This study showed that 1H-LF-NMR associated with digital microscopy efficiently assesses and describes yogurts water retention and spontaneous syneresis without gel destruction.

Exploring local diffusion in heterogeneous food structures

Niklas Lorén[1], John P. van Duynhoven[2], and Magnus Röding[1]

[1]RISE Agrifood and Bioscience, Product Design and Perception, Göteborg 402 29, Sweden;
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Food microstructures are hierarchical, heterogeneous, multiscale, and multiphase. This makes interpretation of relationships between microstructure and food properties challenging. Flow and diffusion are vital for many food properties such as water management in pasta and pastry products and oil migration induced fat bloom in chocolate. Local mass transport properties sum up to macroscopic properties. Hence, versatile measurement techniques for local mass transport are essential to understand structure - mass transport relationships at different length scales in the structure. In this talk, relations between structure and mass transport in heterogeneous foods will be discussed. In addition, an overview of confocal microscopy-based techniques for determination of local diffusion properties will be given. The use of Fluorescence Recovery After Photobleaching (FRAP) [1] and Raster Image Correlation Spectroscopy (RICS) to reveal local diffusion properties at the micrometer level in foods will be demonstrated. A new powerful numerical model based on spectral methods for analysis of FRAP data will be introduced1. The model covers pure diffusion and reaction-diffusion with immobile binding sites. The model was validated by comparison to stochastic simulations of particle dynamics and it was found to be highly accurate. Functionalized nanoparticles and Nuclear Magnetic Resonance (NMR), FRAP and RICS were used to probe the structural heterogeneity in Na+/K+ induced k-carrageenan gels [2]. Results revealing a fast and a slow diffusion Component will be shown.

[1] Röding M, Lacroix L, Krona A, Gebäck T, Lorén N: A highly accurate pixel-based numerical FRAP model based on spectral methods, Submitted (2019).
[2] de Kort DW, Schuster E, Hoeben FJM, Barnes R, Emondts M, Janssen HM, Lorén N, Han S, van As H, van Duynhoven JPM: Heterogeneity of network structures and water dynamics in k-carrageenan gels probed by nanoparticle diffusometry, Langmuir 34 (2018) 11110-11120.

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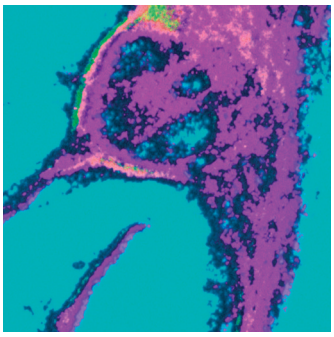

Colloidal Dispersions

The structure and rheology of some dietary fiber suspensions

Eva M. Tornberg

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The structure and rheology of some dietary fibre (DF) suspensions has been investigated, where the origin of the vegetable DF has been tomato CB and HB, apple, carrot, potato, parsnip and yacon. The DF constitutes of the cell wall components building up a network of insoluble cellulose fibrils and hemicelluloses and pectins, where the two latter can be partly soluble. Yacon root fruit also contains another interesting fiber, namely fructo oligosaccharides. For the fibers like carrot and potato pulp containing high amounts of insoluble pectin the morphology of the cells is cell clusters even after homogenisation, whereas tomato suspensions having larger, single cells originally are easily degraded to cell fragments by homogenisation. Apple cells originally also having larger, single cells are though not so easily degraded. Usually bimodal area-based particle size distributions (PSD) are observed for fibre suspensions in general, where the smaller particles are between 1 to 100 μm and the larger between 100 to 1000 μm. As most of the particles belong to the larger particles (>100μm) they constitute the network and the larger the area of this network the larger the elasticity, G'. This seems to hold for all the DF suspensions except for yacon. The elasticity, G', of the DF network is dependent on the conc. of insoluble particles (WIS), where parsnip does not give rise to the concentrated region until a WIS of about 2.5%, whereas tomato CB and HB, apple, potato pulp, carrot and yacon all give rise to the concentrated region below 1% WIS.. Conclusively, the pectin-rich vegetable fibers seem to glue the insoluble particles to each other, probably through pectin adhering to the insoluble fiber and forming a gel. The capacity of this network of insoluble fibers to give rise to a high elastic modulus is dependent on the amount of WIS, the area of the large particles and in the concentrated region also on the hardness of the particles.

Determining the viscoelastic and solubility properties of soy protein isolate solutions

Timothy D. O'Flynn[1], Noel A. McCarthy[2], and James A. O'Mahony[1]

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For 2019 the projected global soybean crop production yield is expected to be 369 million tonnes, with the U.S., Brazil and Argentina producing 122, 120 and 39 million tonnes, respectively. In recent years it is becoming an increasingly popular choice as an alternative protein source to dairy, due to increasing costs associated with animal protein sources and a cultural shift in consumer attitudes towards more vegetarian diets. Soy protein is currently used in follow-on infant formulas, high protein beverages and sports nutritional powders, as it is a good source of essential amino acids. However, soy protein isolate (SPI) powder is extremely poorly soluble in water, an attribute which is essential for its incorporation in to more complex nutritional systems. The aim of this study was to improve the functional properties of SPI for subsequent use in nutritional formulations. A commercially available SPI powder was obtained and the solubility determined at pH 2, 6.7 and 9.0 at 20°C. Adjusting the pH of SPI dispersions from pH 6.7 to 9.0 improved protein solubility but increased the viscosity of the samples at 20°C, presumably due to increased electronegative charge and greater voluminosity of the proteins. Readjusting the pH to 6.7 resulted in a return of the original insolubility properties of the protein. Heat treatment (80°C x 5 min) of SPI dispersions (10 % w/w) resulted in an increase in viscosity at pH 6.7; however, viscosity decreased in pH 9.0 samples with increasing temperature and remained low during cooling back to 20°C, although the system still maintained an elastic-like behaviour (i.e., G' > G"). In conclusion the solubility of SPI can be improved through pH changes, and viscoelastic properties controlled and manipulated via temperature.

Effect of particle size on optical properties and viscoelasticity of nano-microstructured cellulose based suspensions

Rene Machuca, Josefina Ortega, Francisca Palacios, Daniella Sotella, Javier Enrione, and Paulo Diaz-Calderon

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Cellulose structured at nano and microscale are novel materials with wide and interesting applications in food science and technology (e.g. packaging, coatings, emulsions), however their use in commercial products are still limited. The goal of this work was to analyse the role of particle size on optical properties, viscosity and viscoelasticity of suspensions of cellulose nanocrystals (CNC) and bacterial cellulose microfibrils (BCMF). CNC (100nm lenght) were purchased on Process Development Center UMaine (USA), whereas BCMF (300 microns length) were kindly provided by Vuelo-Pharma (Brazil). Suspensions of CNC and BCMF in distilled water were prepared at 0.16, 0.52 and 0.90 %w/v. CNC-BCMF suspensions were analysed in terms of their optical properties (absorbance-transmittance between 400 - 700 nm), viscosity and viscoelasticity (apparent viscosity by flow sweep and G', G'' and tan delta by frequency sweep). All measurements were carried out considering at least five replications. BCMF showed absorbance higher than 0.75 in all the tested range, whereas CNC showed values close to 0.15 or even lower in the same range. Transmittance was higher than 75 % in CNC suspensions, but lower than 10 % in BCMF. BCMF also showed higher viscosity than CNC suspensions, tested by both viscometry and flow sweep (e.g. 5 - 7 cP in CNC, > 100cP in BCMF). Both CNC-BCMF suspensions showed to be non-Newtonian. The latter was consistent with the viscoelasticy, where BCMF showed values of both G' and G'' were 2 - 4 log magnitudes higher than CNC suspensions. BCMF was showing G' values independent of the frequency suggesting formation of stiff gels, which was not observed in CNC suspensions. This behavior is coherent with the behavior of tan delta which was constant and < 1 in all BCMF samples, but strongly dependent of angular frequency in CNC suspensions. Therefore, particle size would be one of the key factors which define the performance of cellulosic materials structured at nano and microscale.

Hemp globulin and casein: Colloidal frenemies

Simon M. Loveday[1], Chih-Chieh Chuang[2], Skelte G. Anema[3], and Teresa F. Wegrzyn[2]

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Hemp globulins are salt-soluble, heat-labile oligomeric proteins that are present at high levels in hemp seed cake, i.e. the waste material from hemp oil extraction. Hemp protein is a potential food source, but its poor solubility and tendency to aggregate on heating limit food applications. We have exploited the chaperone-like properties of caseins (sodium caseinate, SC) to solubilise hemp globulins (HG) in colloidal complexes. Complexes were formed by heating or pH-cycling mixtures of HG and SC, and they were characterised with dynamic light scattering, TEM, SDS-PAGE and a novel high-throughput fluorescence method. SC inhibited heat-aggregation of HG in a concentration-dependent way, and we hypothesise that SC caused a shift from diffusion-limited cluster aggregation to reaction-limited cluster aggregation. When mixtures of HG and SC were cycled between pH 7 and pH 12, monodisperse complexes ~100nm in diameter were formed, and the proportion of colloidally stable HG rose from 3% to >85%. Complexes disintegrated in urea solution, which suggests that hydrogen-bonding was crucial to their integrity, and we believe that caseins on the surface of complexes provided electro-steric stabilisation. Plant proteins and dairy proteins are often framed as enemies; we have shown that when it comes to colloidal stability, they can be frenemies.

Using pea-derived maltodextrins for nutraceutical formulation

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Nutraceutical food ingredients market is continuously growing, although technological issues remain unsolved. Indeed, most of them are labile to heat and oxygen, insoluble in water and have a poor bioavailability in the gastrointestinal (GI) tract. To overcome those issues, solubility improvement has been investigated through particularly the formation of inclusion complexes using starch-derived products such as amylose or cyclodextrins. As pea is naturally rich in amylose, we investigated the potentiality of pea-derived maltodextrins (MD) as solubilization enhancers. The study was separated in two parts: set-up of a model study to define the methodology and the potential structure obtained followed by its extension to various nutraceutical food ingredients to validate the model and explore their bioavailability and stability. The model study was conducted to investigate the behavior of a non-soluble molecule in an aqueous solution of MD. 1-naphthol was selected as host-molecule for its low water solubility and physicochemical features especially related to amylose complex. Solubilization was performed in an aqueous solution and supernatants were characterized by analytical tools: vibrational spectroscopy, liquid and solid NMR, XRD and microscopy. MD achieved in fully solubilizing 1-naphthol in aqueous solution. NMR and vibrational spectroscopy experiments pointed out specific interactions between 1-naphthol and MD. XRD and solid-state NMR didn't reveal typical amylose-complex fingerprint, meaning that different mechanisms enabled 1-Naphthol solubilization. Microscopy analyses showed small organized domains forming coiling-like layered microstructure. Our model study was extended to several molecules often used as nutraceuticals. The same analytical strategy was applied to characterize the types of interaction between MD and targeted molecules. Bioavailability and stability of each system were assessed in in vitro GI conditions. A correlation between in vitro results and structural analysis was finally proposed.

Structuring lipids through enzymatic glycerolysis

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Hydrogenation, interesterification, and fractionation are processes commonly used to improve the functionality of edible oils by increasing the saturated fat content, thereby altering the crystallization behaviour of the lipids. Furthermore, monoacylglycerols (MAGs) and diacylglycerols (DAGs) have also been known to affect the crystallization properties. The purpose of this research is to use glycerolysis to produce partial glycerols to directly structure liquid oils without affecting the fatty acid composition. Glycerolysis reactions were performed at 65°C for various lengths of time in the presence of glycerol using *Candida antarctica* lipase B as the catalyst. Differential scanning calorimetry demonstrated a 20°C increase in the onset of crystallization of both cottonseed and peanut oils following glycerolysis. Pulsed nuclear magnetic resonance showed that the solid fat content of cottonseed and peanut oils increased from 8 to 27 % and 3 to 28 %, respectively, at 5°C. Additionally, at 5°C the cottonseed oil only begins to crystallize after approximately 4 h, while this process begins immediately, after the oil has undergone glycerolysis. The structured cottonseed oil represents a viable alternative to palm oil and hydrogenated vegetable oils for applications in margarine as well as shortenings. When incorporated into peanut butter, the peanut oil glycerolysis product decreased the oil loss to less than 6 % of that of peanut butter containing unaltered peanut oil. The textural properties of the peanut butter can be tailored to meet the demands of consumers by changing the conditions of the glycerolysis reaction to obtain various partial glycerol compositions. This research demonstrates that glycerolysis can be used to enhance the functionality of edible oils for their use in numerous food applications.

Rheological and structural characterization of dairy desserts with resistant starches under oral conditions

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Starch is one of the most widely used thickeners in the food industry because it gives smoothness, texture and prevents syneresis. Among starches, the use of resistant starches (RS) is increasing, and its addition to a semi-solid system might modify the food structure resulting in perceptible mouthfeel changes. The objectives of the present work were 1) to analyze the effect of adding two different RS (type II and type III) at different levels (1.5 and 3 %) and in mixture with other thickeners (xantham gum, carboxymethyl cellulose and kappa-carrageenan) on the microstructural and rheological properties of custards before and under in vitro oral digestion and 2) to characterize the sensory properties of the custards For that, a combination of several techniques was carried out, such as particle size distributions, optical microscopy, rheology and sensory. First, flow properties (shear rates of 1 - 200 1/s for 60s , 35°C), and viscoelastic properties (amplitude and frequency sweep) of desserts samples were measured. Structural in vitro breakage of the samples were determined using a Starch Pasting Cell adapted to a controlled stress rheometer, at 10 1/s shear rate for 120 s at 35°C and adding artificial saliva. Sensory differences in creaminess, grittiness and consistency were evaluated using ranking tests by 60 consumers per session (three different sessions). Results show preservation of the RS granules after in-vitro oral digestion for both RS used, although particle size differences were observed between granules (RS type II bigger than RS type III). In combination with a thickener, structure of custards prepared with xantham gum and RS (type II and III) were preserved better (less viscosity decay). Samples prepared with any of the RS and carboxymethyl cellulose or kappa-carrageenan had high initial viscosity, but higher viscosity decay, this provided higher perceived consistency and grittiness.

MONDAY AFTERNOON

Symposium KA

Keynote Lecture A

Fibrillar structures in mixed systems

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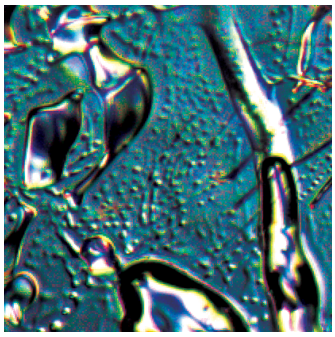
Three mixed systems were studied. The first consisted of protein based semi-flexible fibrils with oil droplets. It was observed that above a minimum concentration of fibrils depletion flocculation occurred. Above a certain concentration of fibrils, the emulusion was stabilised, and small oil droplet aggregates with single droplets were observed. The aggregate size is independent of the fibril concentration. The droplet aggregation was reversible upon dilution with an equally concentrated fibril solution and a solution without fibrils. No emulsion droplet network nor fibril network was observed. The viscosity of the emulsion was similar to that of the fibril solution. The second system consisted of monodisperse polystyrene latex particles with protein fibrils. Similar observations to the emulsion systems were gathered, and in addition within a certain concentration regime also bridging flocculation occurred. The third systems consisted of protein fibrils and Bacterial Cellulose (BC) microfibrils with oil droplets. When only BC microfibrils are added to an emulsion, oil droplet creaming is slowed down, and eventually arrested above a certain concentration of BC microfibrils. Adding both protein fibrils and BC microfibrils to an emulsion may lead to an antagonistic effect: at low concentration of protein fibrils the destabilising effect of the protein fibrils can be counteracted by the stabilising effect by the BC microfibrils.

international symposium on food rheology and structure

ISFRS 2019

8TH INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

Symposium BS



Biopolymer Solutions and Gels

The influence of suspension rheology and micromechanics on sensory grittiness

Heather M. Shewan[1], Jason R. Stokes[1], and Heather E. Smyth[2]

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The discovery of unexpected sensations associated with the presence of particles, such as grittiness or powderiness, arising from oral processing of a soft food product can impact liking and repeat purchase. It is known that for hard particles such as unswollen starch or undissolved sugar particles, sensory grittiness is a function of particle size, shape, roughness and size distribution. However, the origin of gritty/powdery/chalky etc. sensations in many commercial soft food products is not obvious since they predominantly contain soft microscopic particles. Little quantitative information is known on the inter-relationship between particle softness, phase volume and matrix phase viscosity or rheological profile on sensory grittiness and particle detectability. Existing research in this area uses highly complex food systems where the degree of particle swelling, softness and aggregation are unknown and particles are subject to degradation in-mouth, which prevents development of coherent relationships between defining parameters. With a view to address these short-comings, in this work we specifically aim to quantify the influence of suspension micromechanics on particle detectability in fluid and semi-fluid foods using model microgel particles. Agar microgels with tuneable mechanical properties are used as an additive to hydrocolloid matrix phases with a range of viscosity from Newtonian to yield stress fluids to form a mixture of samples of varying rheology and particle concentration. Sensory evaluation by a trained panel confirms the hypothesis that grittiness perception decreases with decreasing particle modulus for particles with a maximum particle diameter Dmax ca. 100 micron. A new rheometer-based technique is used to capture suspension micromechanics, and this is shown to provide unprecedented ability to define relevant material properties governing in-mouth response that capture the synergistic effects of size, softness, phase volume and matrix phase viscosity.

Do rheology and oral tribology relate to sensory texture perception? A case study on hydrogels

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Characterisation of oral processing using rheology as a tool has produced relationships between instrumental measures (e.g. bulk viscosity) and sensory perception (e.g. perceived thickness) in fatty products. This study aimed to characterise the relationship between instrumental properties of non-fat hydrogels and their sensory perception. The fracture properties of kappa-carrageenan (kC) hydrogels and mixed gels containing different ratios of kC and locust bean gum (LBG), sodium alginate (NaA) or with 300 or 1000 µm calcium alginate beads (CaA) at 1 - 4 wt% total biopolymer concentration, were evaluated with a compression test at 22°C. The hydrogel boli were examined after simulated oral processing by flow curves (plate-plate geometry) and friction coefficient µ, using polydimethylsiloxane (PDMS) ball-on-disc set-up with pre-adsorbed artificial salivary film at 37°C. Nine sensory attributes were identified with a panel (n = 11) that could be related to either the chewing or lubrication aspects of oral processing. Results demonstrated that the gel fracture properties and bolus viscosity were directly correlated to the chewing-related sensory attributes, such as firm, elastic and chewy, as well as inversely correlated to the lubrication-related attributes pasty and melting ($p < 0.05$). On the other hand, µ at orally relevant speeds (50 mm/s) correlated to the lubrication-related attributes slippery and salivating and inversely correlated to pasty for the gel bolus fluid where the large bolus fragments were filtered out. Theoretical 'scaling' of µ against the product of entrainment speed and bolus viscosity showed good agreement of observed data with the Stribeck master curve in the mixed regime. Thus, our study demonstrated a unique relationship between rheology, tribology and sensory perception in aqueous hydrogels, which can open new horizons in designing foods with tailored orally perceived texture attributes.

[1] Krop EM et al.: Food Hydrocolloids 88 (2019).

Protein-based emulsion gels for edible oil structuring

Ina Nephomnyshy and Maya Davidovich-Pinhas

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Oleogelation has recently gained popularity as strategy for transforming liquid oil into soft solid-like structured gels to obtain the functionality of fats in food products. The gel-like structure is achieved by oil entrapment within 3D network created by self-assembling molecules. Among food grade oleogelators, proteins offer the greatest potential as network-forming structurants since they are widely available, relatively cheap and are considered healthy ingredient in modern diet. However, utilization of protein as an oil structuring agent raise a great challenge due to its chemical composition. The current research focus on the characterization of protein stabilized emulsion gel fabricated using a direct procedure at moderate temperature feasible for large-scale production. Micrsocopy images indicated on the formation of a composite system where the oil droplets are dispersed within continuous protein network composed of particulate protein aggregates. The emulsion gel stability at different protein and oil content was evaluated using thermal analysis. Mechanical analysis demonstrated a positive relation between the protein content and gel hardness while a reverse relation was observed with respect to the oil content and the hardness. Oscillatory temperature sweep analysis demonstrated the sol-gel transition of the emulsion gel from 4 to 90°C with storage modulus (G') higher than loss modulus (G"). In addition, thermo-reversible behavior was observed during two cooling/heating cycles. Frequency sweep analysis at different temperatures confirmed the formation and breaking of hydrogen bonds between the particulate protein aggregates. The emulsion gel also exhibited excellent thixotropic behaviour with 100% recovery after high shear treatment. The current research demonstrate a thorough structure-function study providing a broad understanding of the gel building blocks. Such understanding can be used to further develop protein based oleogel systems with desirable characteristics.

Primary, secondary, tertiary and quaternary structure levels in linear polysaccharides: From random coil, to single helix to supramolecular assembly

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Polysaccharides are ubiquitous in nature and represent an essential class of biopolymers with multiple levels of conformation and structural hierarchy. However, a standardized structural nomenclature, as in the case of proteins, is still lacking due to uncertainty on their hierarchical organization. In this work we use carrageenans as model polysaccharides to demonstrate that several structural levels exist and can be unambiguously resolved by statistical analysis on high resolution AFM images, supported by spectroscopic, X-ray scattering and rheological techniques. In direct analogy with proteins, we identify primary, secondary, tertiary and quaternary structures. The structure-property relationship induced by monovalent ions for κ-, ι- and the non-gelling control λ-carrageenan is established from the single chain regime to the occurrence of hydrogels at higher concentrations. For κ-carrageenan in the presence of potassium, a disorder-order transition from random coil to single helix is first observed (secondary structure), followed by intrachain supercoiling events (tertiary structure) and macroscopic anisotropic domains which are parts of a network (quaternary structure) with tunable elasticity up to ~10^3 Pa. In contrast, κ-carrageenan in the presence of sodium only produces changes in secondary structure without supercoiling events, prior to formation of gels, highlighting the ion-specificity of the process. Loosely intertwined single helices are observed for ι-carrageenan in the presence of sodium and potassium chloride, providing an elastic mesh with many junction zones, while λ-carrageenan does not undergo any structural change. A generality of the observed behavior may be inferred by extending these observations to a distinct class of polysaccharides, the weak carboxylic polyelectrolyte Gellan gum. These results advance our understanding of ion-specific structural changes of polysaccharides and the physical mechanisms responsible for their gelation.

Differences in the microstructure and rheological properties of acid gels from goat, sheep and cow milk

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Acid milk gel is the basis of the most popular semi-solid dairy products such as yoghurt. It is formed with a three dimensional milk protein network structure. Although it is known that there are differences in the composition of milk proteins and primary structures of individual caseins and whey proteins in cow, sheep and goat milk, little is known how such differences might influence the microstructure of acid milk gels produced and consequent rheological properties. In this study, the physicochemical properties including rheology and microstructure of acid gels made from goat, sheep and cow milk were investigated and systematically compared. Confocal laser microscopy and scanning electron microscopy were used to investigate the microstructure of the acid gels while dynamic oscillatory low-strain time and temperature sweep rheology was used to examine the rheological properties and acid gelation process. The results showed that goat yoghurt required the longest fermentation and gelation time but exhibited the lowest storage modulus at the end of the fermentation. Goat yoghurt had the weakest protein network with the most porous structure, fewest crosslinks between protein strands and the lowest gel firmness. While cow and sheep yoghurts had a similar fermentation time, sheep yoghurt had a more compact and denser protein network. Cooling resulted in an increase in the storage modulus with the biggest magnitude observed in sheep yoghurt and the smallest in goat yoghurt. The results obtained here provide a valuable insight into the differences in the microstructure and rheological properties of the yoghurts as affected by the different milk types. The results also demonstrate a link between the structure and rheological and functional properties of yoghurts, suggesting that rheology and microstructure could be used to engineer food product properties.

Tackling the question of specific interactions in a complex blend of proteins: Gluten

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Natural protein sources often display a huge complexity, being composed of a blend of polypeptides of various molecular weight, pHi and charge density. Gluten, the proteins extracted from wheat flour is one of such. Gluten is widely used for its viscoelastic properties as an improver of cereal products (bread, pastry, etc.). It is composed of two classes of proteins, named gliadin and glutenin, similar in their amounts in glutamine (30%) and proline (10%). The more than 25 different polypeptides belonging to the gliadin class are hard to fractionate into individual components because of high redundancy in the primary sequences. Glutenin are in the form of polymers made from several distinct polypeptides concatenated through interchain disulfide bonds. While it is well established that gliadin confers viscosity to gluten whereas glutenin polymers are at the origin of its elastic resistance, the interactions existing between both classes of gluten protein remain unknown. We previously showed by SLS and multi-angle DLS that gluten proteins suspended in ethanol/water, a theta solvent, includes large proteins assemblies displaying an internal dynamic. To get a better insight of the composition of those assemblies, we combined biochemical and physicochemical approaches. On the one hand, gluten proteins suspended in ethanol/water were fractionated by Asymmetrical-Flow-Field-Flow Fractionation coupled to UV, SLS and QELS detectors. On the other hand, gluten proteins were partitioned by liquid-phase decomposition in respect with temperature. Protein composition of partitioned phases and eluting fractions recovered from A4F were characterized by size-exclusion chromatography. A specific interaction between omega-gliadin and glutenin polymers was evidenced. This result strengthens a very recent study that demonstrates a significant positive correlation between glutenin polymeric proteins, the ?-gliadin fraction, and dough properties.

Large deformation, fracture and lubrication properties of emulsion-filled gellan gum gels

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This work investigates the large deformation, fracture and lubrication properties of emulsion-filled gels, which are prepared from a low-acyl gellan gum, soybean oil and tween®80. The use of low-acyl, as opposed to high-acyl gellan gum to prepare the gels, is more challenging as it is more susceptible to emulsion instability due to its lower gelling temperature. The effect of the oil droplets on the properties was examined using uniaxial compression and tribological experiments. At concentrations between 0.2-1.5% w/w, the stress-strain response of emulsion-free gels was hyperelastic with strain rate independent. In contrast, the fracture behavior was strain rate dependent and the rate dependency was more pronounced at low gel concentration and strain rate. Such characteristic is similar to starch and gelatin gels with helical structure. For the emulsion-filled gels, the presence of oil droplets, for volume fractions $\varphi = 0.1\text{-}0.3$, slightly improved the gel stiffness, thus indicating that the oil droplets behave as active fillers. The fracture properties were considerably affected by the inclusion of oil droplets but its dependency on the strain rate is suppressed compared to the emulsion-free gel. Furthermore, both fracture stress and fracture strain decreased with increasing oil content but were approximately constant at $\varphi > 0.2$. Such feature is driven by stress concentration phenomena due to the presence of structural inhomogeneities as well as inclusions in the gel network. Microstructure analysis also confirmed the aggregation of oil droplets, resulting in irregular particle structures. Furthermore, the emulsion-filled gels showed a decrease in the measured coefficient of friction compared to the emulsion-free gels. Therefore, this study has shown that the presence of oil droplets and its interaction with the gel matrix can significantly affect the textural properties of emulsion-filled gels, and understanding their effects will lead to the better food texture design and optimisation.

Symposium KB

Keynote Lecture B

Numerical and experimental investigation of bread dough kneading in a 3D spiral kneader

Laila Abu-Farah, Thomas B. Goudoulas, and Natalie Germann

TUM, Freising, Germany

Kneading is a key step in the production of bread. The intent of this process is to develop the gluten network and to bring air into the dough. In this work, numerical simulations were conducted in an industrial spiral kneader to obtain information about the flow kinematics and the material properties inside the mass and on the dough surface during kneading. The White-Metzner model along with the Bird-Carreau viscosity function was used to describe the dough rheology. Unstructured tetrahedral grids were generated using ICEM CFD 17.1. Transient 3D simulations were carried out using OpenFOAM v.4.0 in combination with the RheoTool package v.2.0 for viscoelastic flows [1]. The governing equations were discretized using finite volumes and subsequently solved using the free-surface solver rheoInterFoam. Our method was successfully validated by simulating the rod climbing benchmark problem in a cylindrical bowl [2]. The flow patterns, the rheological properties, and the distribution of air bubbles in the dough were analyzed for different rotational speeds and compared with laboratory kneading experiments. Local information about the microstructure development was obtained using the previously implemented damage function calculated in terms of the eigenvalues of the Cauchy strain tensor [3]. The waves at the dough/air interface could be successfully predicted. Pockets of different sizes were formed, extended, and finally broken in the shaft region. Tangential and spiral flows were the dominant flow patterns. Microstructure development was greatest at locations where extension was strong. Industrial kneading geometries and processes may be optimized further by making use of these types of simulations.

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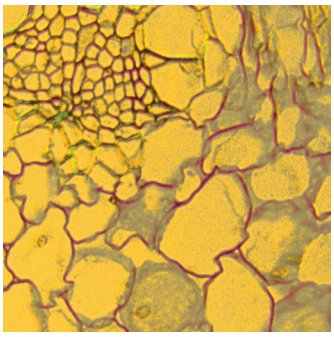
Symposium DG



Dough

Exploring the effect of arabinoxylans on the rheology of blended wheat flour-rye flour doughs via treatment with xylanases

Yannick Meeus[1], Frederik Janssen[2], Arno Wouters[2], Jan Delcour[2], and Paula Moldenaers[1]

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The production of multigrain breads appears to be a useful strategy to obtain high quality breads both in terms of baking performance and nutritional value. The recipe for a multigrain bread typically contains wheat flour (WF) to assure bread making performance, and one or more non-wheat cereals which improve the nutritional profile. While the importance of the WF gluten network for WF dough rheology is already well known, the role of the non-gluten components in multigrain doughs is still largely unknown. In this study we examine the influence of the water-extractable (WE) and water-unextractable (WU) arabinoxylans (AX) fraction on the rheology of blended WF-rye flour (RF) doughs. To this end, the doughs were treated with a xylanase with specificity for hydrolyzing the WEAX or WUAX. Gas chromatography was used to assess hydrolysis of the AX. Uniaxial extensional measurements characterized the non-linear extensional behavior whereas small-amplitude oscillatory shear (SAOS) measurements were used to study the rheology in the linear shear regime. Our results show that hydrolysis of the WEAX, as manifested by a decrease in the average degree of polymerization of the WEAX, did not affect the extensional rheology of the doughs. Hydrolysis of the WUAX mainly impacted the extensional viscosity extrapolated from the linear viscoelastic envelop. The influence of AX on the extensional viscosity at maximum strain was limited. Even though the strain hardening index for the doughs increased with high-dose xylanase treatment, the volume of the corresponding breads was unaltered after bread making trials. The SAOS measurements displayed an increase of the phase angle upon addition of xylanase to the dough recipes, especially at high angular frequencies, indicating that the effect of AX on linear dough rheology was not merely an effect of water.

Characterising the microstructure of deep-fried battered and breaded coatings to understand crispness

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The quality of deep-fried battered and breaded coatings is recognised by a crisp and dry outer coating contrasting a moist and tender core. Substrates are coated with a batter layer, which is essentially a wheat flour mixture then coated with a panko breadcrumb layer. During the deep-fat frying process, battered and breaded coatings submerge into hot oil, where temperatures exceed 100oC, this results in surface drying, shrinkage and roughness. Simultaneously, moisture deep within the core migrates towards the surface and releases via explosive evaporation. This results in voids that create a porous structure, as well as entry points for oil and moisture exchange. Deep-fat frying is a simultaneous process of heat and mass transfer. As a textural attribute, crispness perception has been shown to be a combination of tactile and auditory components and depends on macroscopic and microscopic features within the food. X-ray MicroCT was employed to characterise the internal morphology of battered and breaded coatings of eight breadcrumb sizes: 4.0, 2.8, 2.0, 1.4, 1.0, 0.71, 0.5, 0.355 mm. Coatings with a larger breadcrumb size showed higher porosity (70.4 % ± 7.5), wide pore size distribution (0.009 - 1.815 mm) and narrow structural thickness (0.009 - 0.797 mm). Coatings with a larger breadcrumb size also showed high compression force (45.5 N ± 8.6), sound pressure levels (75.1 db ± 4.6) and sound peaks (86.2 ± 29.2). These variables were inversely proportional with smaller breadcrumb sizes. These internal characteristics dictate the amount of moisture lost and oil penetration, all of which were studied using confocal microscopy. A combination of physical and mechanical properties will affect the sensorial perception of crispness. The use of force-deformation coupled with acoustic emissions as well as internal morphology characterisation allows for a thorough understanding of how the structure of food materials relates to texture perception.

Hydrogen-bond interactions as quantitative descriptors of food structuring mechanisms during cereal-based food processing

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Wageningen Food & Biobased Research, Wageningen University, Wageningen, The Netherlands

The structure and texture of bakery products is largely controlled by biopolymers hydration and structural arrangements as well as their melting transitions (i.e. starch gelatinization, protein denaturation) during processing. Solutes such as sugars, polyols and soluble fibres present in the water phase can strongly influence these processes. Sugars in sweet bakery products like biscuits and cakes exert important functionality in relation to structure and texture as they largely affect starch gelatinization and protein denaturation. Which makes a substantial reduction in sugar difficult to achieve. In bread, the enrichment in fibre is also technologically hampered by the detrimental effects on gluten network. The mechanisms by which sugars, polyols and soluble fibre interact with food biopolymers is not fully understood. In this lecture we demonstrate how the volumetric density of hydrogen bonds can quantitatively describe the melting transition of starch and proteins in water solutions containing different solutes, i.e. sugars, polyols, soluble fibres and mixtures thereof. Taking as example a bread application, we then show how the volumetric density of hydrogen bonds can be used to describe the functionality of different type of soluble fibres. Insights are provided on the influence of the effective number of hydrogen bonding sites of the fibres on gluten structure, dough rheology and thermo-mechanical behaviour during baking. Overall, this lecture shows how key structuring processes during preparation of bakery products can be largely related to physical parameters descriptive of hydrogen bond interactions.

Impact of endogenous wheat lipids on bread quality, linear and non-linear extensional rheology of dough and air/water interfacial properties of dough liquor

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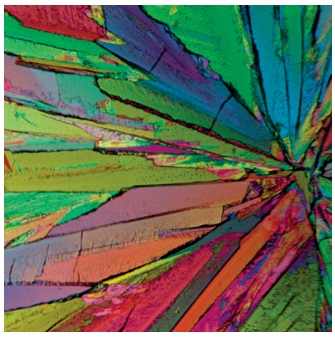
Proper gas cell stabilization during dough fermentation is essential to obtain high quality bread. Despite the low level of wheat endogenous lipids in flour, they are assumed to impart an (i) indirect and (ii) direct effect on gas cell stabilization in bread by (i) strengthening the gluten network and (ii) adsorbing at gas cell air/water interfaces. Wheat flour lipids are divided in those occurring in starch (and not functional in dough) and those freely present. The latter are a heterogeneous mixture of triacylglycerols, free fatty acids, and glyco- and phospholipids. Sroan and MacRitchie (2009) observed that nonpolar and polar wheat flour lipids each have a different impact on bread volume. Because no effect of adding nonpolar and polar wheat flour lipids on dough rheology was observed in this study, the conclusion was drawn that lipids determine bread volume by adsorbing at gas cell interfaces. However, the relative standard errors of their rheological measurements were very high and gas cell stabilization by adsorption of surface-active constituents was not experimentally investigated. The latter is typically done by studying the air/water interfacial properties of dough liquor (DL), the supernatant obtained by ultracentrifugation. Thus, the mechanism by which endogenous wheat flour lipids impact bread volume is still not completely understood. In the present study, breads were made with wheat flour to which total, polar, and nonpolar flour lipid fractions were each added in levels of 50 and 100% (relative to the total lipid level of the native flour) on top of the already present lipids. Total flour lipid extracts obtained by extraction with chloroform/methanol (1:1 v/v) (Ryckebosch et al. 2012) were separated into nonpolar and polar lipid fractions with solid phase extraction (Ryckebosch et al. 2012). Changes in bread loaf volume were related to the linear and non-linear extensional rheology of dough (Meerts et al. 2016) and to the air/water interfacial properties of DL constituents.

Symposium IP



Influence of Processing on Structure and Rheology

Materials science approach for continuous encapsulation and structuring with protein-carbohydrate matrices

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Encapsulation requires entrapment of sensitive bioactives with structure-forming food components to enhance protection and delivery. In the present study, an encapsulation matrix was developed using whey proteins and sugars for controlled hardening and vitrification of wall materials. A concentrated mix of WPI (hydrated separately or combined) and sucrose in water as gelling and glass-forming ingredients, respectively, was used to form stable particles; product characteristics were compared for protein rehydration. Using a peristaltic pump, liquid flowed continuously through silicon tubing and formed droplets. Rapid solidification of protein occurred when droplets were submerged in heated, stirred oil; drops were then harvested for vacuum oven drying. Feeds were characterized by viscosity and flow tests to gather densities, flow rates, surface tensions, drop weights, and estimated droplet sizes. Glass transitions and other changes in state were recorded using differential scanning calorimetry. Dried drops were characterized for porosity, hardness, diameter, water activity, and solids content. Microstructures were analyzed with optical, confocal scanning laser, and scanning electron microscopy. Analysis determined that drops containing 40% WPI and 10% sucrose by weight possessed optimum structuring qualities. The continuous process was adapted to encapsulate anthocyanins from blueberry concentrate; changes in feed and drop characteristics were evaluated and compared to those without berries. Solid structures formed after undergoing heat-induced protein gelation, drop expansion prior to network collapse with water loss, and sucrose glass formation during drying. This study provides insight into systematic, materials-based preparation and use of high solids-concentrated dispersions, demonstrating an alternative encapsulation technology.

Heterogeneous high concentrated phase separated food systems

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Food products such as pasta, bread and plant-based meat substitutes can be considered as highly concentrated biopolymer systems of discrete and immiscible phases constituted by proteins and/or starch and fibers. Protein functionality and the compatibility among the ingredients play an important role when designing food products to obtain desired textures. High concentrated phase separated systems are less explored scientifically in comparison with hydrated mixed starch and protein gel systems. Here, we show the possibilities for creating innovative food products by designing high concentrated phase separated assembled in discrete layered structures on different length scale. High concentrated phase separated food model systems in micrometer scale were made through co-extrusion of pellets based on different cereal mixtures. The degree of phase separation, i.e. distribution, size and form of the cereal materials was controlled by extrusion parameters and material compositions. Food systems of layered structures in millimeter scale were made by combining two extruders using a feeding block. The most important factor for controlling the distribution of different phases in a system was found to be the material compatibility in combination with the material composition in the interfaces. Two cereal mixtures were highly compatibility at the specific composition resulting in complete mixing of the materials. Purer continuous phases with high concentrations of either protein or starch were overly incompatible causing repellency between materials and the extrudates fell apart. Hence, designing high concentrated phase separated food systems in micrometer scale required moderately material compatibility, i.e. one bicontinuous mixture of protein and starch and one continuous phase of either protein or starch. Incompatible cereal fractions caused adhesion problems, however by partly mixing one of the materials with the other changing the phase volumes, good adhesion was achieved.

Influence of kinetic and shear rate on whey protein aggregates structure: A small-angle x-ray scattering and fluorescent microscopy study

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Whey proteins are of interest because of their nutritional and functional properties in food application. Heat-induced aggregation coupled with process conditions of whey proteins gives them new functional properties that can be used to impart specific structural and physical properties of food products. Aggregation process needs to be well understood and controlled to design specific functional whey protein aggregates. Most of previous studies has focused on the role of the physicochemical conditions on the structure and size of protein aggregates, whereas process parameters haven't been clearly investigated. Role of process parameters, i.e. shear, heat treatment and time on the size and structure of protein aggregates will be investigated. A continuous process of aggregation at small-scale (<1 mm) was developed to have laminar flow conditions for various shear rates and a fine control of the thermal history. Aggregation is no longer limited by heat transfers. Thermal and flow conditions can be controlled independently. This small-scale continuous process allows us to vary the residence time and to establish the kinetics of aggregation and the shear rate up to 500s-1. The role of several process parameters on the kinetics and structure of whey protein aggregates were investigated by small angle X-ray scattering (SAXS) techniques for given physicochemical conditions. Structure of whey protein aggregates larger than few micrometres are also investigated by quantitative fluorescent microscopy. We show that the kinetics leads to the formation of new aggregates and not to their enlargement. Then, we show that the flow process has a large impact on the size and structure of the aggregates: the size of the aggregates is increased by a factor 3 when comparing the ones obtained under static conditions and the ones obtained under flow, whereas their internal structure remains unchanged. The shear rate leads to an increase of the size of the aggregates without increasing their density.

Symposium PL

Plenary Lectures

Design of yield-stress fluids

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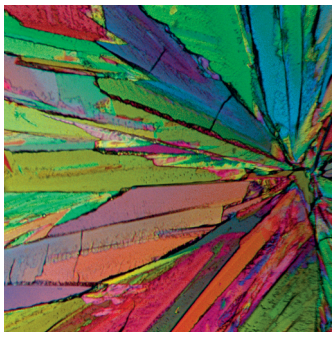
Yield-stress fluids can be engineered from a diverse range of microstructures including polymeric gels, colloidal glasses, emulsions, pastes, foams, and more. This talk will discuss our research on design thinking for yield-stress fluids [1], addressing the rheology-to-structure inverse problem of the many ways to achieve a yield stress [2] and the important secondary property of extensibility in real materials including food products [3]. In addition to food and confectionary products, our application-motivated research includes direct-write 3D printing [4], high performance concrete [5], and fire suppression with sprayable gels [6].

[1] A.Z. Nelson, K.S. Schweizer, B.M. Rauzan, R.G. Nuzzo, J. Vermant, R.H. Ewoldt: Designing and transforming yield-stress fluids, submitted, 2019
[2] A.Z. Nelson, R.H. Ewoldt: Design of yield-stress fluids: A rheology-to-structure inverse problem, Soft Matter 13 (2017) 7578-7594.
[3] A.Z. Nelson, R.E. Bras, J. Liu, R.H. Ewoldt: Extending yield-stress fluid paradigms, J. Rheol. 62 (2018) 357-369.
[4] B.M. Rauzan, A.Z. Nelson, S.E. Lehman, R.H. Ewoldt, R.G. Nuzzo: Particle-free emulsions for 3D printing elastomers, Adv. Funct. Mater. 28 (2018) 1707032.
[5] J.A. Koch, D. Castaneda, R.H. Ewoldt, D. Lange: Vibration of fresh concrete understood through the paradigm of granular physics, Cement Concrete Res. 115 (2019) 31-42.
[6] B.C. Blackwell, M.E. Deetjen, J.E. Gaudio, R.H. Ewoldt: Sticking and splashing in yield-stress fluid drop impacts on coated surfaces, Phys. Fluids 27 (2015) 043101.

Symposium IP



Influence of Processing on Structure and Rheology

Rheological study on the interactions between oleosomes and co-extracted materials during aqueous extraction

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Oleosomes (or oil bodies) are micro sized (0.2 to 2 μm) oil storage organelles in seeds, which are composed by a core of triacylglycerols (TAGs) surrounded by a monolayer of phospholipids and proteins. The partially hydrophilic nature of their interfacial proteins, allows their extraction by aqueous solvents to form a natural oil in water emulsion. Co-extracted seed material, like storage proteins interact with oleosomes along the extraction process and affect the rheological properties of the obtained natural emulsion. Therefore, understanding how to adjust the interactions between extraneous proteins and oleosomes and the effect of these interactions on the rheological properties of the obtained emulsion, is essential, in order to enable the use of oleosomes as building blocks in food materials. In this study, the rheological properties of a multi-component natural emulsion obtained from rapeseeds were investigated as affected by the aqueous extraction process. The results showed that it was possible to coalesce the oleosomes by reducing the interfacial charge. Additionally, it was possible to customize the aggregation of the oleosomes which seemed to be caused by the London dispersion forces between oleosomes interfacial proteins and co-extracted materials. The presence of enlarged single oil droplets leaded to comparable rheological properties between the obtained natural multi-component emulsion from aqueous extraction and commercial emulsions (i.e. mayonnaise). This work provide new insights into the development of natural oil droplets as a food ingredient.

Kinetics of heat-induced denaturation of whey proteins and characterization of protein aggregates in model infant formulas

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In 2018, about 60 % of world's newborns received cow milk-based infant formulas (IF) instead of human milk (UNICEF). The process of manufacturing IF involves heat treatments altering the physicochemical properties of milk components, especially whey proteins (WP), and so the rheological properties of IF. The objective of the study was to investigate the impacts of thermal treatments on the denaturation of WP of IF, particularly for those mimicking the protein profile of human milk, and to characterize the heat-induced protein structures. Three model IF were produced with a caseins:WP ratio of 40:60 at 1.3 and 5.5 % of total proteins, i.e. the protein contents at which are applied heat treatments during the manufacture of liquid or powder IF, respectively. Skimmed milk was mixed with a WP isolate, a mix of WP isolate and purified lactoferrin or a mix of both purified lactoferrin and a-lactalbumin in proportion similar to that in human milk. The kinetic of heat-induced denaturation of each WP was investigated between 67.5 and 80°C by RP-HPLC. The heat-induced protein structures were studied by dynamic light scattering, electrophoresis and asymmetric flow field flow fractionation coupled with MALLS. The results revealed that the extent of denaturation of WP depended on the protein content and the nature of the WP within the IF. IF at 5.5 % of proteins and containing ß-lactoglobulin gelled for longer heating time at 80°C. At similar rate of total WP denaturation at 67.5 and 80°C, the protein composition of the heat-induced aggregates changed between formulas, protein concentrations and heating temperatures but disulfide bonds were the main intermolecular links. The aggregates were larger (fractal shape dfapp = 2.1) in formulas at 5.5 % proteins whereas they were of spherical shape (dfapp = 2.9) in formulas at 1.3% proteins. These results provide reliable data on the protein structures formed during the heat treatments of IF. The impact on digestibility will be subsequently investigated.

The impact of hydrocolloids on the microstructure and function of cream cheese

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The hydrocolloids Locust bean gum and Guar gum are often added to dairy and other food products to alter chemico-physical properties, including water holding capacity and flow properties under shear. In cream cheese, these gums are known to alter product texture, prevent whey separation during storage and improve both product spreadability and mouth-feel. Yet few studies have systematically examined the effect of these gums either individually and synergistically on the structure and function of double fat cream cheese, a product of economic importance to Australia. In this study, we examined the effect of gum concentration, together with processing variables, including temperature and shear, on the microstructure and rheological properties of double cream cheese. We employed both confocal and cryo scanning electron microscopies to observe differences arising in the corpuscular structure as a function of ingredient type and processing variables. Our findings illustrate how formulation can be used to increase the stability of cream cheese structure and to tailor product properties. They also demonstrate the influence of processing on product properties which has implications for the pumping and handling of products during manufacture. The study is part of a broader program that aims to improve our understanding of formulation, structure and the impact of processing variables on semi-soft, fresh acid curd cheeses, which will also assist in future ingredient substitution and the development of 'clean label' products.

Dynamic structural breakdown behaviour of a model Maasdam-style cheese under tensile deformation as studied using confocal scanning laser microscopy

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Knowledge of the breakdown behaviour of cheese is important for gaining insights into texture perception, flavour and nutrient release, as well as the origin of undesirable texture defects within the cheese matrix such as slits and cracks. In this novel study, changes in the microstructure of model Maasdam-style cheeses were observed in situ during tensile deformation by placing a microtensile stage directly under a confocal scanning laser microscope (CSLM), and recording force/displacement data simultaneously. A small indentation (called a notch) was made at a centre point on the test specimens, and growth of the notch was observed under tensile deformation using CSLM. Widening of the notch, stretching of the protein network near the leading point of the notch, detachment of fat globules, and their subsequent release from the cheese matrix, as well as fracturing of the cheeses, partly along curd granule junctions, were all observed during tensile deformation. Moreover, an inherent micro-defect was observed at a curd granule junction within the cheese matrix and this micro-defect fractured along the curd granule junction under tensile deformation, suggesting that the micro-defects present within the cheese matrix could be a key underlying factor in the formation of undesirable slits or cracks. Further work showed that the fracture behaviour of cheese was altered by changing ripening temperature, using different coagulant types, or by inhibition of residual chymosin. Such approaches could be applied to designing cheeses with specific texture profiles or for designing optimal cheese textures to withstand increased gas pressure during ripening in eye-type cheeses, which may help to prevent the formation of undesirable splits and cracks. Overall, this study demonstrated the potential of in situ imaging of cheese microstructure for developing a greater understanding of the breakdown behaviour of cheese matrices.

Comprehensive pulsed electric field system analysis for microalgae processing

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Pulsed electric field (PEF) is an emerging nonthermal technique with promising applications in microalgae biorefinery concepts. In this work, the flow field in continuous PEF processing and its influencing factors were analyzed and energy input distributions in PEF treatment chambers were investigated. The results were obtained using an interdisciplinary approach that combined multiphysics simulations with ultrasonic Doppler velocity profiling (UVP) and rheological measurements of Arthrospira platensis suspensions as a case study for applications in the biobased industry. UVP enabled non-invasive validation of multiphysics simulations. A. platensis suspensions follow a non-Newtonian, shear-thinning behavior, and measurement data could be fitted with rheological functions, which were used as an input for fluid dynamics simulations [1]. Within the present work, a comprehensive system characterization was achieved that will facilitate research in the field of PEF processing.

[1] Leandro Buchmann, Robin Bloch, Alexander Mathys: Comprehensive pulsed electric field (PEF) system analysis for microalgae processing, Bioresource Technology 265 (2018) 268-274.

Influence of mold materials on the gloss of chocolate bars

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The gloss of a chocolate bar surface is one of the main quality parameters appreciated by the consumers, despite texture and taste. The chocolate should exhibit a high and uniform gloss. It can be expected that the chocolate gloss is influenced both by the processing, especially appropriate pre-crystallization, and by the characteristics of the mold material surface which is in direct contact with the chocolate during solidification. So far, the correlation between surface properties of the mold material in microscopic scale and the resulting chocolate gloss is not really understood and deviations in gloss, like local matte areas, are still found on chocolate bars after de-molding. Therefore, different materials were investigated with respect to the gloss of the chocolate molded at these surfaces. Materials and chocolate were characterized with respect to their microstructure by atomic force microscopy (AFM) and their surface energies. AFM analyses covered topography, including roughness, and adhesion forces. With respect to chocolate gloss, a distinct influence of the surface roughness could be detected. Only very flat surfaces with a low roughness of less than few micrometers can produce high gloss chocolates. Additionally, the surface energy also affects the gloss indicating that the interactions of the liquid chocolate during molding and the mold material surface are relevant. These correlations were implemented in a model enabling the prediction of chocolate gloss from mold material properties.

Properties of fresh milk protein ingredients as a consequence of frozen storage

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Milk protein ingredients (MPIs) are known for their favorable nutritional and functional properties when applied in food products, including dairy products. In order to study the effect of processing on the performance of MPIs, it would be an advantage to have model systems of e.g. milk, frozen down. We will present from a study where we have investigated the effect of freezing on fresh casein and whey protein concentrates produced from skim milk by the use of microfiltration (MF) and ultrafiltration (UF). The different properties (e.g. particle size distribution, hydrophobicity, rheological behavior) of fresh, unfrozen protein samples (cold stored at 4°C) were compared with samples subjected to frozen storage (- 80°C). Our results indicate that there were no significant differences between the particle size distribution and hydrophobicity for the frozen samples compared to cold stored samples. However, the viscosities of all the frozen casein concentrate samples at different concentrations (18, 5, 4, 3, and 2 %) were significantly lower than the unfrozen ones. A gelation test using chemical acidification GDL (Glucono Delta-Lactone) showed that frozen and unfrozen samples had the same trend in measured moduli. Nevertheless, both G' (storage modulus) and G" (loss modulus) values of frozen samples were lower than for the unfrozen in the initial stages of gelation and higher when the equilibrium was reached. Results from gelation using rennet exhibited similar gelation behaviors. This study provides novel possibilities for the use of frozen milk protein concentrates for model systems which emulate milk and which can be designed with e.g. controlled variation in casein/whey protein ratio in order to elucidate MPIs functionalities in dairy systems.

Water redistribution determined by time domain NMR explains rheological properties of dense fibrous protein blends at high temperature

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Pea protein isolate (PPI) can be combined with wheat gluten (WG) into materials with a fibrous morphology, using shear induced structuring at specific process conditions. The rheological properties largely determine the structure formation process in this phase-separated blend. Therefore, the objective of this study was to understand the rheological properties of the protein phases at process conditions relevant for structure formation. Firstly, the viscoelastic properties of PPI and WG were determined as function of the water content and processing temperature (100-140 °C). Similar properties are measured for the protein blends consisting of those proteins at different ratios and different overall dry matter content. It was found that the apparent complex modulus of WG is always larger than PPI at same dry matter content and the modulus decreased with increasing temperature and decreasing dry matter content. Subsequently, the water content in each protein phase was measured with time domain nuclear magnetic resonance. It turns out that water does not distribute homogenously over the phases. More water is absorbed by the PPI phase relative to the WG phase. Combining the information on water distribution from time domain nuclear magnetic resonance and the viscoelastic properties of PPI and WG from rheology enabled accurate predictions of the rheological properties of dense fibrous PPI-WG-blends.

Inline capillary rheometry and die entry flow simulation of high moisture extruded meat analogues

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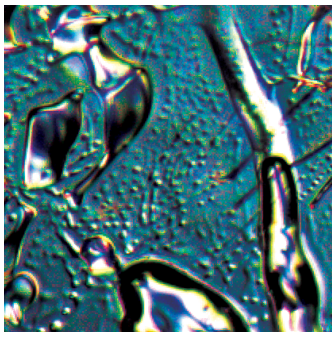
High moisture extrusion (HME) is a process fostering the production of meat analogues from various protein sources. The formation of fibrous, meat-like structures is based on the thermomechanical energy input in the extruder and the subsequent alignment of proteins in a cooling die. Even if the HME-technology is available on a production scale, only little is known about the mechanisms behind the structure formation in the cooling section. This results in time-consuming product developments and laborious determination of optimal process settings. The aim of this study was to better understand and predict protein structuring behavior by characterization of the material properties and numerical simulation of the die entry flow. Rheological data of protein-water mixtures from soy and pumpkin protein were obtained inline at the extruder end plate using capillaries with different length/diameter ratio. The total throughput was varied from 5 to 70 kg/h covering apparent shear rates in the range of 0.1 to 100 s-1. Shear viscosities were calculated from measured pressure drop differences and corrected for entry effects. A rheological and thermodynamic model was developed describing the die entry flow behavior and temperature conditions during extrusion. The rheological analysis was the basis for subsequent 3D CFD flow calculations supporting R&D of meat analogues and the optimized design of HME cooling dies.

Symposium BS



Biopolymer Solutions and Gels

Properties of nanomaterials from maize starches modified with stearic acid

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Amylose lipid complexes (ALC) are formed when fatty acid methylene tail enters the hydrophobic core of amylose helical structure from starch. This research determines the isolation of ALC using a 'green' chemistry principles. Normal and Maize starch was pasted with stearic acid at 91°C for 120 minutes. The paste was then hydrolysed with thermostable alpha amylase and washed with distilled water to remove the soluble dextrins. Wide angle X-ray scattering showed the residues to be amylose lipid complexes (ALC) and Differential scanning calorimetry showed they were of type II. Atomic force microscopy and electron microscopy showed ALC to be about 2.5 - 7 nm. The yield of nanomaterials obtained were about 28 % from normal maize starch and 50 % from high amylose maize starch. The isolation was possible as alpha amylase did not hydrolyse the alpha 1-4 glycosidic bond as the latter is burried inside the complex. Thus the glycosidic bond is not available for hydrolysis. The nano materials in aqueous medium are non gelling as determined by rheological methods and can be used as fat replacers in margarine. The simple pasting followed by enzyme hydrolysis shows potential to isolate ALC as nanomaterials and can be considered as 'clean label' to be used in food system. Nanomaterials in foods have limited use as most are from non-edible source. ALC produced here are edible.

Quantitative analysis on viscous behaviour of concentrated biopolymer solutions related to morphology development during drying

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The functional properties of spray-dried powders are influenced by particle morphology created during the drying process. Morphology development is initiated during drying by increasing solute concentration at the surface of the droplet leading to skin formation (Vehring et al., 2007). The rheological properties of the skin are believed to affect morphology development as the skin impacts the mechanical deformation of the shrinking droplet (Sugiyama et al., 2006). The availability of rheological data of biopolymer solutions at high solids concentrations that are relevant to skin formation is limited. Therefore, the aim of this study was to analyse viscosity of biopolymer formulations at high solids concentration that are relevant to particle morphology development during spray drying. Maltodextrin, whey protein and their mixtures were studied as model system. Firstly, the viscosities of the prepared solutions were assessed experimentally as function of increasing solids concentration. Next, the viscosity data of maltodextrins was modelled by combining Spurlin-Martin-Tennent's (SMT) and Williams, Landel and Ferry (WLF) theories. The viscosity of whey protein solutions was modelled with Krieger-Dougherty. Viscous behaviour of mixed maltodextrin-whey protein solutions was described by Krieger-Dougherty, where the viscosity of maltodextrin solution around the hard spheres was based on the SMT/WLF theory. Finally, single droplet drying experiments with the model systems were carried out to study the influence of medium composition, droplet size and initial solids concentration on morphology development. When drying at high initial solids, morphology development of mixed formulations could be explained from measured and modelled rheological data. This suggests that the analysis of rheological properties at high solids concentrations is useful to explain morphology development. In future, this research may contribute to improved guidelines to steer functional behaviour of powders.

Rheological, tribological and phase-separating properties of concentrated acid gel suspensions in the presence of polymers and at defined particle size distributions

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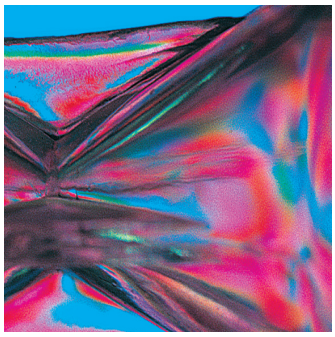
Concentrated fermented dairy products, e.g. fresh cheeses, can be considered as acid microgel suspensions. Fat-reduced formulations and long holding time at high temperature during production bear the risk of inducing textural and stability defects, therefore hydrocolloids are added frequently. The aim of our study was to investigate the effects of added polymers and of the particle size distribution (PSD) on the rheology, tribology and stability of concentrated acid microgel suspensions. Commercial low-fat fresh cheese was blended with aqueous solutions of guar gum or polyethylene glycol (PEG) to adjust serum viscosity to 2.8 mPa·s and protein content to 10 %. The serum from fresh cheese obtained by centrifugation served as reference (1.5 mPa·s). PEG solutions were also incorporated in fresh cheeses with defined PSD that were regulated by thermostatting the samples to 6, 50 or 65°C for 5 h. Rheological and tribological (flow curves, frequency sweeps, Stribeck curves), and phase-separating properties (photo-analytical centrifuge) of the mixtures showed a strong dependency on the type of the incorporated thickener. A high level of cross-linking in the serum phase as observed with guar gum resulted in higher yield stress (11 Pa) and decreased sedimentation velocity (25 µm/s) compared to the reference (2 Pa and 49 µm/s, respectively). Mixtures with PEG showed an increase in yield stress with increasing particle sizes and generally lower friction coefficients (0.31) than samples containing guar gum and the reference (0.36 - 0.38). From the results we conclude that, besides the increase of serum viscosity, specific interactions of the thickeners with protein particles improve texture and stability of concentrated acid microgel suspensions. The IGF Project 18318 BR of the FEI was supported via AiF within the programme for promoting the Industrial Collective Research (IGF) of the German Ministry of Economic Affairs and Energy (BMWi), based on a resolution of the German Parliament.

Symposium RM



Rheological Methods

Charactering acid-induced casein gels wear by creep-recovery and wear-recovery behaviors

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Hydrogels (e.g food gels, artificial cartilage, and contact lenses) are soft solid materials that have applications in a variety of industries. Their wear behaviors are important as wear and deformation rates are connected to durability. However, little is known about hydrogel wear compared to hydrogel rheological behaviors. Moreover, measuring soft material wear rates is relatively imprecise. Hence, the objective of study was to develop a new method to determine hydrogel wear through a combination of creep-recovery and wear-recovery measurements. Casein hydrogels of different concentrations (8 - 15 %) and pH (2.3 - 4.8), and tested under different normal forces (0.3 - 0.5 N). Both creep and wear behaviors were measured using identical testing geometry. Casein hydrogel wear-recovery behaviors were fit to an exponential model. The extent of recovery in both creep and wear tests decreased with higher normal force and pH, and lower casein concentration. Lower pH and higher casein concentration resulted in less wear. However, higher normal force promoted wear. Higher casein concentration resulted in less irreversible deformation, while higher normal force and lower pH increased irreversible deformation. The R2 values of model fits to casein gel wear-retention behaviors were > 0.98, indicating good fit. The method developed in this study can be used to measure hydrogel wear with a greater degree of precision and accuracy. Additionally, the information of how pH, normal force, and casein concentration impact casein hydrogel wear behaviors may be used by food manufacturers to tune gel strengths for specific applications.

Environmental scanning electron microscopy as a novel tool to characterise in real-time the hydration of milk protein concentrates

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The physical state of milk protein concentrate (MPC) powder particles during hydration is not well defined. Nanoparticle size plays a crucial role in the structural, flow and compositional properties of milk powders especially so when determining their subsequent hydration profiles. Particle size is an extremely useful indicator of quality and performance, particularly in the reconstitution of milk powders. Mainstream research has investigated milk powder hydration using bulk density methods. Using advanced bioimaging technologies the real-time hydration of milk powders can be observed at the level of the individual particle. Currently, there are no studies that have examined milk powder particles hydration using environmental scanning electron microscopy (ESEM). The aim of this study was to characterise the real-time hydration of MPC particles using ESEM. Low-(MPC40 and 50), medium-(MPC65 and 75) and high-(MPC85) protein content MPC powders manufactured by Teagasc, Moorepark, Fermoy were stored at 54 and 88 % relative humidity (RH) at 4 ºC. The hydration of the MPC powders were investigated under ESEM to examine the effects of storage temperature and RH. MPC40 and MPC50 which were stored at 54 and 88 % RH exhibited a weakening of the external framework leading to their subsequent collapse and in some cases full dissolution. MPC65 and MPC75 stored at 54 % RH revealed large breaks on the external skin of powder particles and MPC85 displayed little dissolution. MPC65, MPC75, and MPC85 stored at 88 % RH showed a graded disruption of the external skin of the powder particles. This study validates the use of ESEM as a novel tool to record dynamic in-situ hydration of milk powder particles.

Optical characterization methods of dairy products

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Following the seminal work of Loiseleux [1], we have investigated the ability of a whey protein, namely beta-lactoglobulin to texturize water-in-oil emulsions. Aggregates of beta-lactoglulin were produced by applying a thermal treatment. Model triglyceride-in-water emulsions were then formulated in presence of a mixture of native and clustered beta-lactoglobulin. Depending on the native-to cluster mass ratio, different behaviors were observed: liquids, instant gels, delayed gels. The kinetic evolution of the systems was monitored using different techniques: (i) Turbiscan® to follow the creaming process, (ii) Rheoscan ®, and (iii) oscillatory rheology to follow the gelation process. Overall, it is shown that one and the same component, i.e. a protein can be used to both stabilize and texturize systems of interest to the food industry. This approach paves the way for the formulation of food products with a limited range of ingredients.

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Microrheology as a tool for the gel-point determination in food industry

Danila Gaudino, Mathias Reufer, and Andreas C. Voelker

LS Instruments, Fribourg, Switzerland

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. Diffusing Wave Spectroscopy (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 artefacts like non-linear behaviour, shear banding, and thixotropy can be avoided. This significantly simplifies studies on aging and gelling behaviour of shear sensitive products. We present applications of DWS-microrheology on common food products such as gelatine and yogurt. In particular, while the determination of the gel point is a complex topic in science, we show a practical way based on an empirical approach to measure it by means of microrheology. Additional rheological properties, such as the elastic modulus G' and viscous modulus G'' can be also acquired in few minutes and over a huge frequency range, otherwise inaccessible through the classical rheometric techniques.

Effect of in situ relative humidity in the measurement of rheological properties of food products

Carlos A. Gracia Fernández

TA Instruments Waters Division, New Castle, DE 19720, United States

The DHR-RH Accessory is a new environmental system for the Discovery Hybrid Rheometer that enables accurate control of sample temperature and relative humidity. The DHR-RH Accessory employs a similar control methodology and common technology to the popular DMA-RH accessory. Optimized for the rheometer, the DHR-RH Accessory provides stable, reliable control of temperature and humidity over the entire operating range and successfully prevents condensation, a common occurrence in controlled-humidity environments which makes accurate control of relative humidity impossible. In this talk, we will present data on several food products where the control of the influence of the relative humidity is crucial for an accurate rheological measurement

Estimation of pressure field in shear thinning fluid flows based on ultrasound velocity profiler applied to vortex shedding flows

Neetu Tiwari, Yuji Tasaka, and Yuichi Murai

Faculty of Engineering, Hokkaido University, Sapporo, Japan

Pressure field estimation in non-Newtonian flows are very important for industrial and biological processes but literature is scarce in this field [1]. So, we developed a novel pressure estimation algorithm for shear thinning fluid based on UVP (Ultrasonic Velocity Profiler) measurements. In the algorithm, equation of continuity and momentum equation are involved along with a rheological model fitted using flow curve produced by rheometer measurements. The algorithm is applied to study the vortex flow in downstream of a cylinder. The CMC (Carboxy Methyl Cellulose) of weight concentration of 0.1% in water was used, which has shear thinning property. The measurement data are filtered by POD (Proper Orthogonal Decomposition) and FFT (Fast Fourier Transform) low pass filter. The 2-D (two-dimensional) velocity field is reconstructed with one component measured by UVP and another component evaluated by equation of continuity [2]. The measured velocity data were used in Carreau-Yasuda model to visualize spatial distribution of viscosity. An alternating pattern of low-pressure region is observed in the wake region. The algorithm successfully predicted expected viscosity and pressure distribution in the cylinder wake. Also, we observed inverse relation of viscosity with pressure and enstrophy in vortex flow of shear thinning fluid.

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[2] Tiwari N, Tasaka Y, Murai Y, 2019. Pressure field estimation from ultrasound Doppler velocity profiler for vortex-shedding flows. Journal of Flow Measurements and Instrumentation (Submitted)
[3] Murai Y, Nakada T, Suzuki T, Yamamoto F, 2007. Particle tracking velocimetry applied to estimate the pressure field around a Savonius turbine, Measurement Science and Technology 18, 2491-2503.

TUESDAY AFTERNOON

Symposium KA

Keynote Lecture A

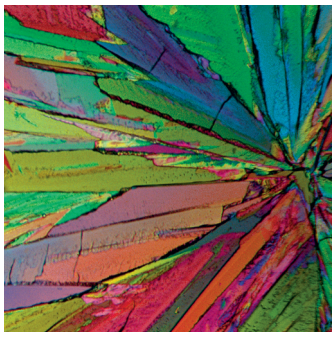
Understanding rice structure as the key to new processing solutions

Nadina Mueller[1] and Béatrice Conde-Petit[2]

[1]ZHAW, Wädenswil, Switzerland; [2]Bühler AG, Uzwil, Switzerland

Rice, the staple food of Asia, provides on average 30 % of the daily calorie intake across the continent. The Asian subcontinent accounts for 90 % of the world's rice production and consumption, China and India alone for 5 0%. Rice quality expectations differ strongly in different countries: while in China premium quality rice is expected to be very fresh with a maximal storage time of three months after harvest, aged rice is preferred in India and is stored up to two years before consumption. Pre-processing and adaptation of standard processing of rice are promising avenues to optimize both milling yield and tailor rice properties to meet consumer desire. However, as rice is prone to develop fissures upon even smallest changes in temperature and moisture, resulting in breakage under physical stress, any adaptation of rice processing is a challenge and deep understanding of rice structure and properties is necessary. The authors will touch on processing approaches with diverse goals including (i) pre-processing of rice to achieve product characteristics of aged rice without storage, (ii) processing of brown rice to minimize cooking time, (iii) new milling concept for "yield and quality", and (iv) the formation of recomposed rice kernels through extrusion. The results show that the processability and functionality of rice is strongly dependent on its structure. Rice grains are composites in which the natural polymers starch, protein and hemicelluloses are assembled with a distinct cellular architecture. A deep-dive on the role of starch and protein will be given based on the analysis of raw, enzymatically treated, dry heated, steamed and cooked rice samples. Selected results including imaging techniques, differential scanning calorimetry and small and wide-angle X-ray scattering SWAXS will be shown that suggest a shared role of crystalline starch and protein in structural cohesion of rice. Last but not least, conclusions for future processing of rice will be drawn.

Symposium IP



Influence of Processing on Structure and Rheology

The effect of purification processes on the viscoelastic properties of heat-induced gels, produced from mild to highly purified yellow pea fractions

Cornelis Kornet[1], Paul Venema[1], Atze Jan van der Goot[2], Marcel B. Meinders[1], and Erik van der Linden[1]

[1]Physics and Physical Chemistry of Foods, Wageningen University, Wageningen 6708 WG, The Netherlands; [2]Food Process Engineering, Wageningen University, Wageningen 6708 WG, The Netherlands

This study establishes a link between yellow pea protein purification processes and the chemical and physical characteristics of the resulting fractions. Protein purification was achieved by solubilisation at natural pH and at pH 8 and subsequently, precipitation at pH 4.5. Supernatants after solubilisation and the pellet and supernatant after precipitation were collected and in addition to pea flour they were labelled as functional fractions 1 - 5. These mildly to highly processed yellow pea fractions were analysed on their composition. The protein content of these fractions varied from 40 to 85 % and it was found that a major part of the milder processed fractions were saccharides. These fractions contained relatively high amounts of starch-like components and a smaller amount of other polysaccharides (e.g. pectin and galactans). The type of proteins were characterized using SDS-PAGE and SEC. To deeper understand the effect of processing on the proteins the fractions were also analysed on hydrophobicity and thiol groups. This chemical characterization was executed to understand differences in rheological behaviour before and after thermal treatment. Physical characterization mainly involved rheological characterization before and after thermal treatment. Before heat treatment, it was found that the highest purified fractions showed a stronger viscosity increase with increasing concentration. During and after thermal treatment, viscoelastic behaviour was studied at small and large deformation oscillatory shear. Milder processed fractions were compared to higher processed fractions and they showed equal elastic moduli as higher processed fractions. However, after standardizing on protein content, significant differences in elastic moduli were observed. Milder purified fractions showed higher G' than highly purified fractions (10^3 versus 10^2 Pa). These observations support recent insights about the potential advantages of mild processing on functional properties.

How multiscale structures in milk fat shape the crystal network formation

Naomi Arita Merino[1], Hein van Valenberg[1], and Elke Scholten[2]

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Behind the rheological properties of milk fat (MF), there is an arrangement of multiscale structures with a great impact on the functionality and sensory properties of several dairy products. At high temperatures, MF crystallizes in one step, while at lower ones, two steps are commonly observed. One- and two-step processes have been linked to the formation of β' polymorph from the melt and mediated by α, respectively. Crystallized MF is highly stable in its β' form, while its β polymorph has been reported in literature only intermittently. In this study, the rheological changes of MF crystallizing isothermally at different temperatures were linked to the multiscale structures of the network. The developing microstructures were characterized from their polymorphism (by WAXD) and lamellar stacking (by SAXS) to the morphology of their polycrystalline particles (by microscopy). Our rheological data revealed the occurrence of three-step network formations in addition to the typical one- and two-step crystallizations. This peculiar G' evolution was observed at intermediate temperatures and consistently concurrent with the formation of β polymorph. The inflection point between the second and third G' increases matched the transition time from α to β and β' and lamellar thickness from 4.5 to 4 nm. The initial micrographs showed the formation and growth of small (15 μm) randomly oriented spherulites, while the polymorphic transition period was marked by the appearance of larger (up to 75 μm) highly branched spherulites. Thus, the first and third G' increments can be linked to increases in the volume fraction of different polycrystalline particles, and the second step to an intraparticle densification. The rheology of crystallizing MF resulted sensitive enough to reflect microstructural changes in a broad length scale. This will allow formulating mechanistic hypotheses for complex crystallization processes using a very convenient technique.

Improvement of the stability of wheat flour doughs containing a high water content: interest of a two-steps structuring-process

Laurena Masbernat, Sophie Berland, Giana Almeida, and Camille Michon

Ingénierie Procédés Aliments, INRA, AgroParisTech, Massy 91300, France

In the context of the characterization of wheat doughs rheological behaviors, most of the studies have focused on the example of bread dough (i.e. water content of about 45 - 50 percent by weight on wet basis (wt%)). Few works have studied dough with higher water contents from 60 up to 80 wt%. In these dough, after mixing, a phase separation occurs, resulting in a global instability of the dough. This instability is a matter of importance for the bakery industry, for instance to improve the stability of ready to use liquid pancake doughs. In the present work, the viscoelastic properties, the consistency and the stability of wheat flour dough made with water content above 50 wt% were studied. To improve the stability of water-rich dough (above 60wt%of water content), process parameters known to affect the micro-structure of dough were adjusted. A process based on a two-steps water addition related to two specific mixing times was developed. The first step of the process was carried out by adding water at a level of 50 wt%. It led to the formation of a dough involving a well-developed gluten network, observed by confocal laser microscopy. The second step consisted in a further addition of water under mixing to reach a water content above 50 wt%, followed by a defined mixing time. A structured high water content dough was obtained. Different water contents and mixing times were tested. The rheological properties of the two-steps processed doughs and their stability were characterized and compared to one-step mixed doughs. Doughs with water contents higher than 60 wt% were more stable upon time when produced with the two-steps mixing process in comparison to dough made using the one-step process. Confocal laser microscopy demonstrated that the structure of the gluten network developed in the first step, remains during the second addition of water but is more and more modified with the increased quantity of water.

Effect of N2 injection before spray-drying on the microstructure and physico-mechanical properties of regular and agglomerated high protein milk powders

Valentyn Maidannyk, David J. McSweeney, Vinay Mishra, Sharon Montgomery, and Noel A. McCarthy

Food Chemistry & Technology Department, Teagasc Food Research Centre, Fermoy, Cork 0000, Ireland

Poor solubility of high protein milk powders causes problems during the production of nutritional formulations, as well as for end-user customers. One of the possible ways to improve powder solubility is through high pressure gas injection before spray drying. The aim of this study was to examine the effects of injecting nitrogen (N2) gas in to the concentrate high-pressure line prior to spray drying. Four types of powders (MPC, 80%, w/w, protein) were produced, i.e., regular or agglomerated and with or without N2 injection. Resultant powders were examined using scanning electron (SEM), optical light (OLM), and confocal laser scanning (CLSM) microscopy techniques powders. Differential scanning calorimeter (DSC) and dynamic mechanical analyser (DMA) were used to measure glass transition (Tg) and a-relaxation temperatures (Ta). Structural strength parameter was obtained for powders humidified at various water activities as well as for reconstituted liquid systems (10, 20, 30 and 40% of solids). Atomic force microscopy (AFM) was used to perform topographical analysis, attractive and repulsive phase imaging, nano-indentation, force modulations and analysis of different surface properties. Visualisation confirmed that N2 injection increased the sphericity of powder particles and created fractured structures with pores for both regular and agglomerated systems. Strength approach indicated that N2 injection did not influence Tg, but changed Ta. AFM showed that systems after N2 injection exhibit significant differences in nano-mechanical properties compared to control MPC powders. The results of this study provide an in-depth understanding of the changes in the morphology and physical-mechanical properties of gas injected MPC powders.

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Symposium KB

Keynote Lecture B

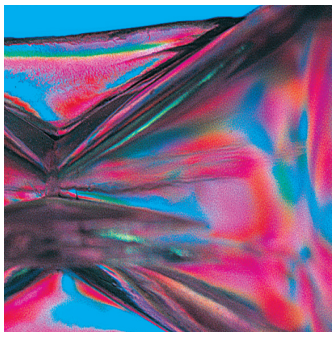
When grains flow: The rheology of particulate systems

Olivier Pouliquen

IUSTI, CNRS, Aix Marseille University, Marseille 13013, France

Flows of granular media in air or in a liquid have been a research field for several decades. Sometimes solid, sometimes liquid, these particulate materials are of importance in many industrial applications and have motivated many studies at the frontiers between physics, fluid mechanics and rheology. This lecture will present a summary of recent advances in the field, with a focus on the rheology, which make it possible to treat granular media as a complex fluid. We will show how the concept of "a pressure imposed rheology" and the development of specific experimental tools have been very helpful for better understanding the behavior of different particulate systems, from dry granular media to dense complex suspensions. The limits of the approach and the remaining open challenges will also be discussed.

Symposium RM



Rheological Methods

A chemically-selective rheo-MRI method to study dense food emulsions

Maria R. Serial[1], Joshua A. Dijksman[2], Luben N. Arnaudov[3], Camilla Terenzi[1], Henk Van As[1], and John P. van Duynhoven[1]

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Emulsions are an important part of our diet, since they allow formulation of otherwise immiscible oil and water phases. An important class are food products, such as mayonnaise, which exist as dense oil/water emulsions. These systems are known to show a non-linear rheological response when subjected to shear [1]. Since these emulsions may display heterogeneous flow behavior under shear, it is necessary to couple standard rheological experiments with localized shear rate measurements in order to fully understand their rheological properties. In the past years, rheo-MRI velocimetry has successfully proven to be a suitable technique to study multiphase flows, allowing to assess the structure and rate of deformation of complex food materials, locally and in a non-invasive way [2]. However, the current implementation of rheo-MRI experiments does not fully exploit the potential to map oil/water phases with chemical specificity. In this work, we implemented a chemically-selective rheo-MRI velocimetry method able to measure oil concentration and velocity profiles of a dense oil/water emulsion under shear. The method was validated with respect to quantification of oil concentration and velocity under industrial relevant conditions. Later on, we applied the technique to study non-linear flow behavior of oil/water emulsions stabilized by Yucca saponin extracts. The rheo-MRI method allowed for real-time assessment of both local shear rate and stress, as well as local oil concentration. Under shear, the emulsions displayed transient shear-banding behavior indicating shear-induced ageing at the hour time-scale.

[1] Ovarlez G et al.: Wide-gap Couette flows of dense emulsions: Local concentration measurements, and comparison between macroscopic and local constitutive law measurements through magnetic resonance imaging, Physical Review E 78 (2008) 036307.
[2] Nikolaeva T et al.: Manipulation of recrystallisation and network formation of oil-dispersed micronized fat crystals, Langmuir 35 (2019) 2221-2229.

An idea to contactless in-line rheometry using ultrasonic velocity profiling

Yuji Tasaka, Taiki Yoshida, and Yuichi Murai

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Evaluating rheological properties of fluids flowing in a pipe without inserting any instruments into the fluids has been desired to control quality of products in many industries including food manufactures. Ouriev & Windhab (2002) established in-line rheometry utilizing ultrasonic velocity profiling (UVP), which can measure time variation of instantaneous velocity profiles through opaque pipe walls, and pressure difference along a pipe. The measurement technique, termed UVP-PD, however, cannot avoid inserting pressure sensors to be contacted with test fluids. The authors established rheometry also utilizing UVP for evaluating rheological properties of complex fluids, which cannot be evaluated by conventional spinning rheometers. In this method, the properties are determined as the measured velocity profiles in an oscillating cylinder filled with test fluids satisfy the equation of motion for simple one-directional flows in the azimuthal direction in the cylinder and rheological models as constitutive equations (Tasaka *et al.* 2015; Yoshida *et al.* 2017; Yoshida *et al.* 2018; Tasaka *et al.* 2018). Applying this idea to achieve contactless in-line rheometry requires a breakthrough to avoid the pressure difference measurement. We introduced an idea to realize the contactless rheometry utilizing information of unsteady flows. Numerical experiments using exact solution of pulsatile flows in a pipe were performed to evaluate applicability of the idea and applicable range of the rheological properties.

Ice cream rheology

Fredrik Innings[1] and Arlov Dragana[2]

[1]Research & Technology, Tetra Pak, Lund, Sweden; [2]Research & Technology, Tetra Pak, Lund, Sweden

In this work we describe the development of a methodology to do rheology measurements and CFD simulations of ice cream in an industrial environment. In an ice cream plant, the ice cream of about minus 6 degrees are pumped from the freezer to the molding or extrusion equipment. At this stage in the production line the consistency of the ice cream is like soft ice cream, a very viscous liquid. In this development both bob and cup as well as pipe rheometers have been used and the experiments have been made with different recipes, temperatures and as the ice cream is a foam, at different pressures. The measured rheology models combined with the CFD methods used have been validated both using pressure drop measurements over different flow geometries and with simple velocity profile measurements [1 - 4].

[1] Wildmoser J: Impact of low temperature extrusion processing on disperse microstructure in ice cream systems, Ph.D. thesis, ETH Zurich (2004).
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[3] Martin PJ, Odic KN, Russell AB, Burns IW, Wilson DI: Rheology of commercial and model ice creams, Applied Rheology 18 (2008) 12913.
[4] Elhweg B, Burns IW, Chew YMJ, Martin PJ, Russell AB, Wilson DI: Viscous dissipation and apparent wall slip in capillary rheometer of ice cream, Food and Bioprodicts Processing 87 (2009) 266-272.

Rotation, oscillation and more: The rheometer as a universal tool for the investigation of complex food formulations

Fritz Soergel, Valerie Pietsch, Klaus Oldörp, and Fabian Meyer

Material Characterization, Thermo Fisher Scientific, Karlsruhe 76227, Germany

In food characterization, rheometers are increasingly evolving into modular universal tools with manifold accessories and capabilities. The classical rheological approach covers rotational testing for the determination of flow properties and parameters for optimized processability as well as oscillatory testing for investigation of e.g. gelation, cooking, baking or freezing. Beyond rheological tests, a universal rheometer can also give information about the texture of foods using its precise lift in combination with its normal force capability. In addition, simultaneous microscopy and spectroscopy facilitate investigation of structure/property relationships. A calibrated normal force (Fn) sensor provides absolute readings of forces occurring in direction of the measuring axis. Such forces can be induced for example by shear deformation (Weissenberg Effect), shrinkage, swelling or drying. With an Fn control loop, a set normal force value can be maintained by moving the lift gently up or down. This can be employed for example to measure changes in sample height (and thus changes in volume) or to adjust and hold Fn in tribology experiments or to record e.g. the softening and melting of chocolate over a heating ramp (squeeze flow). With an axial measuring element, either a set Fn value can be maintained or a lift movement profile can be controlled. In both modes, a rotational movement can be superimposed, either in CR or in CS mode (controlled rate or controlled stress). This allows for more complex experiments for example simulating chewing, or to conduct tack, penetration, indentation or breaking tests. A modular rheometer with normal force control capability facilitates rheological characterization and texture analysis measurements with one and the same sample and same sample history. The benefit of the combination of rheological food characterization with force controlled experiments will be illustrated with extruded plant-based meat substitutes where sensory properties play a key role for consumer acceptance.

Symposium PL

Plenary Lectures

Molecular and macromolecular engineering of foams: Drainage kinetics and rheology

Vivek Sharma

Chemical Engineering, University of Illinois at Chicago, Chicago, IL 60607, United States

We pursue an understanding of molecular and macromolecular principles that determine the three desirable attributes - stability, lifetime and rheology- of foams. Drainage in foam films formed by surfactant concentrations above the critical micelle concentration proceeds in a non-monotonic, step-wise fashion called stratification in contrast to the monotonic thinning exhibited by films containing no micelles. In reflected light microscopy, stratifying films display regions with distinct shades of grey implying that domains and nanostructures with varied thickness coexist in the thinning film. Stratification proceeds by formation of thinner domains that grow at the expense of surrounding films, and stratification dynamics determine foam properties and lifetime. In this contribution, we solve the long-standing experimental challenges of characterizing thickness variations by using interferometry, digital imaging and optical microscopy (IDIOM) protocols that we developed recently. We show that nanoridges and mesas that form and grow at the moving front around the expanding domains can be visualized and analyzed with an unprecedented spatial (thickness ~ 1 nm, lateral ~500 nm) and temporal resolution (< 1 ms). We show the complex spatio-temporal evolution of nanoridges, mesas and domains can be modeled quantitatively by using the nonlinear thin film equation amended with supramolecular oscillatory surface forces. Finally, we investigate the influence of adding polymers and proteins to micellar foam films, paying close attention to the additional influence of enhanced extensional viscosity and viscoelasticity. We elucidate the influence of choice of polymers (flexibility, extensibility, and charge), surfactants on the stability and drainage of stratifying foam films, and highlight the challenges and opportunities for molecular and macromolecular engineering desirable and controllable rheological properties, processability and stability in foams (especially food foams).

Symposium KA

Keynote Lecture A

Foam flows: A mesoscale system par excellence

Antonio Delgado

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A wide spectrum of natural and technical processes generate foams. They consist of condensed matter capable of flow and a gaseous phase dispersed in it, as well as at least another substance that is absorbed by the gas-liquid surface. Together with the rheological properties of condensed matter, the latter leads either to high values of the Deborah number De, as exhibited by "solidified foams" or to a small value of De, as typical for "liquid foams". Anyway, foams always carry the traits of non-equilibrium systems with a wide range of effective characteristic length and time scales. The relevant length scales begin with the molecules in Ångström range up to the characteristic dimensions of the interesting foam volumes, namely those of the relevant production volume mostly in the meter range. Finding the effective characteristic time scales requires a more nuanced consideration. In addition to the eigentimes, with which a foam responds to deformations with predetermined kinematics, further time scales appears due to the non-equilibrium state. The latter are due to the stochastically occurring disproportionation and coalescence processes as well as to diffusive and convective transport processes that takes play with completely different time scale. Based on selected experimental observations, theoretical and simulative considerations, this contribution aims to illustrate that foam flows represent mesoscale systems per excellence. In addition, this contribution intends to show that including mesoscale based tools plays a fundamental role in modelling and simulation of foams. Using the example of protein food foams, it is proven that such global parameters such as foamability and the behavior of foams under static and dynamic conditions can be predicted reliably. This has been done with an information technology hybrid that uses information from different information sources. The obtained results elucidate that a very small error in the order of magnitude of about 10 % can be achieved.

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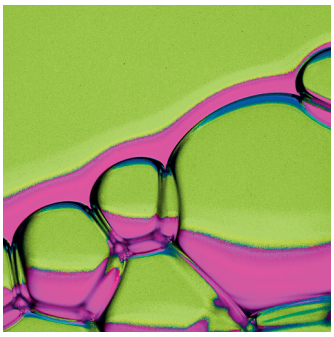

Emulsions, Foams and Interfaces

In-situ rheological and structural characterization of milk foams in a commercial foaming device

Annika R. Völp[1], Jan Engmann[2], Deniz Z. Gunes[2], Cécile Gehin-Deval[3], and Norbert Willenbacher[1]

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Processing, lifetime and sensorial perception of food foams are tightly related to their flow properties. Characterization of the foam structure and yielding behavior are central for product development and quality control. Here, we present an experimental setup, which allows for characterization of foam rheology and structure at different heights of a foam column within a commercial whipper. The cylindrical container with excentric whipping coil and heating plate at the bottom is built into a rotational rheometer. Yield stresses are measured with a vane rotor of 1 cm height at intervals of 1 cm along the rotational axis of the container. Bubble size distributions are analyzed by endoscopic imaging and automated bubble detection at same heights with temporal resolution on the order of seconds. Gas volume fractions are obtained from electrical conductivity measurements. Brass rings at the inner container wall and a height adjustable ring submerged into the foam serve as electrodes to detect the conductivity at intervals of 1 cm along the foam axis. Foams made from UHT milk and reconstituted milk have been investigated for up to 20 min foam age. The gas volume fraction increased linearly with height within the foam column, whereas the mean bubble size and bubble size distribution remained constant. The yield stress increased with height as expected from the change in gas volume fraction. During foam aging, the mean and width of the bubble size distribution increased linearly with time. In contrast, the gas volume fraction saturated over time, and accordingly, the yield stress passed through a maximum. The gradients in gas volume fraction and yield stress along the column axis increased over time depending on milk composition. At higher fat content of the milk the gas volume fraction gradient was reduced and foam lifetime was longer. The developed method offers in-situ characterization of key morphological and rheological parameters of foams.

Interfacial behaviour of plant-dairy protein blends: Comparison between oil-water and air-water interfaces

Emma Hinderink[1], Leonard M. Sagis[2], Karin Schroën[3], and Claire C. Berton-Carabin[4]

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A considerable number of food products exist as multiphase systems such as oil-in-water emulsions. The interface between the two phases is often covered by proteins, which form viscoelastic layers and provide physical stabilisation of the droplets. Recent work suggests that using blends of animal and plant proteins for this purpose could be an optimal way to mitigate sustainability and functionality concerns. Yet, the precise interfacial behaviour of such blends is still unexplored. In the present work we study whey protein/sodium caseinate-pea protein isolate blends and perform dilatational rheology at the air-water and oil-water interface using an automated drop tensiometer, and analyse the data via Lissajous-Bowditch plots. For the individual proteins, comparable trends were observed at the air-water and oil-water interface, with whey protein forming the most elastic and sodium caseinate the most viscous interface. The response of the blend-stabilised interfaces deviated from the average of the individual protein-based ones. At the air-water interface, the response of the sodium caseinate-pea protein blend had similar responses to protein stabilised interface. At the oil-water interface the whey-pea protein blend- and the pea protein-stabilised interfaces had similar responses. Overall, higher elastic moduli and more rigid interfacial layers were formed at the air-water interface, compared to the oil-water interface, for all proteins. These insights are linked to the microstructure of the protein films, using Langmuir-Blodgett deposition and analysing the films with atomic force microscopy. We also determined surface loads using ellipsometry. Our results show that the synergy between plant and dairy proteins is highly protein-specific and dependent on the nature of the interface.

Measuring the interfacial rheology of soluble surfactants using controlled foam Plateau border and node geometries

Christopher R. Clarke, Aris Lazidis, Fotis Spyropoulos, and Ian T. Norton

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The production of foams in the food industry is often reliant on the use of soluble surfactants, whose ability to quickly reduce surface tension can dramatically improve formation and longevity. Despite their widespread use however, the relationship between interfacial rheology and macroscopic foam properties is still poorly characterized. Existing theoretical work predicts the interfacial rheology of the liquid channels between bubbles to influence net drainage rates and hence foam longevity. However, without the means to directly measure this relationship *in-situ*, firm conclusions cannot be drawn. This study addresses the clear need for a reproducible, accurate and standardized approach to studying interfacial rheology at the scale of well-characterized liquid foam channels. A novel Plateau Border and Node (PB-Node) setup allowing precise control of liquid flow through isolated foam channels has been developed; analysing their spatial profiles to infer interfacial rheological characteristics. Solutions of Sodium Dodecyl Sulphate (SDS) exhibited unusual channel distortions at higher liquid flow rates, which were shown to be well described by microscale drainage theory. Interfacial rheology was measured in the form of an apparent surface viscosity, including contributions from surface shear and dilational viscosities, and Marangoni Stresses. The apparent surface viscosity measured here was in good agreement with literature data of SDS surface shear viscosity, showing virtually inviscid behavior, while flow rate dependence was primarily attributed to the contribution of Marangoni stresses. These findings indicate that the interfacial contribution to liquid flow dissipation in SDS foams is negligible, suggesting other soluble surfactant systems to follow a similar trend. It is believed that further assessment of such systems with controlled variations in physicochemical properties could yield significant insights into the relationship between interfacial rheology and macroscopic foam characteristics.

Interfacial behavior of plant proteins

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Challenges of public health and sustainable development require replacing in food products animal proteins by plant proteins. It is thus crucial to understand the structure and kinetic of formation of a film of plant proteins to improve the control of emulsions and foams stabilized by these proteins. We will present experimental results on the behavior interfacial properties of wheat gluten, sunflower and rapeseed proteins at liquid interfaces. Thanks to a combination of tensiometry, dilatational rheology, and ellipsometry, rational physical pictures of the dynamics of the interfacial properties are achieved, for the various proteins and at both air/water and oil/water interfaces. For gluten proteins, a time-concentration superposition is evidenced whatever the subphase concentration, which reveals that the kinetics of protein adsorption at the interface is dominated by bulk diffusion. We propose a consistent physical picture of the multistep diffusion-controlled irreversible adsorption of the gliadin proteins at an air/water interface, and evidence surface-induced conformational changes of the proteins and film gelation. Sunflower and rapeseed proteins by contrast do not reorganize once adsorbed at an interface and display a simpler dynamics of film formation. In addition the failure at high concentration of the time-concentration superposition of the tensiometry and viscoelastic data strongly suggest a surface-induced aggregation process, which we confirm with turbidity measurements. By quantitatively comparing the surface pressure dependence viscoelasticity of the various interfaces, we hightlight the crucial role on the behavior of plant proteins at liquid interfaces of the solvent quality and of the protein softness, that is discussed in regard to the protein structure.

Interfacial properties of whey protein in recombined dairy cream

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Recombined dairy cream is cream prepared from anhydrous milk fat, and needs additional stabilizers to protect oil droplets from (partial) coalescence. In this study, the interfacial properties of milk fat-water interfaces stabilized by whey protein isolate (WPI) and whey protein aggregates (WPA) at different bulk concentrations (0.1 and 4.0 %wt), were studied by Large Amplitude Oscillatory Dilatation (LAOD). Lissajous Plots were used to analyze the nonlinear response of the interfaces as a function of strain amplitude and frequency. The elastic modulus was quantified based on the tangent modulus at zero instantaneous strain E'_M and the secant modulus at maximum strain E'_L [1]. At 0.1 %wt, WPI and WPA formed elastic interfaces with similar E'_M and E'_L. At 4.0 %wt, we hardly found a difference in E'_L between WPI and WPA at any amplitude. However, the E'_M of WPI decreased drastically with the increase of oscillation amplitude and clearly displayed intra-cycle yielding during expansion, while the E'_M of WPA was nearly constant. The frequency of oscillation showed little effect on the value of E'_M and E'_L for both WPI and WPA. Based on a study on bulk stability of emulsions, neither WPI nor WPA could form stable emulsions (20 %wt milk fat) at 0.1 %wt. Both WPI and WPA could stabilize the emulsion in quiescent conditions at 4 %wt. Due to the yielding behaviour above a critical strain, we suspect that WPI could be a worse stabilizer in dynamic conditions when the emulsion is processed and subjected to high deformation rates. This will be examined in follow-up studies.

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Controlled ice crystal formation in ice cream by plant based ice structuring proteins

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Ice Structuring Proteins (ISP) are naturally produced by a variety of living organisms, including certain vertebrates, plants, fungi, and bacteria that permit their survival in subzero environments. ISP adsorb at the surface of ice crystals and form a barrier layer which inhibits growth and recrystallization processes. The application of plant-based ISP is of high interest for formulations of frozen foods as natural ingredients preserving the initial ice crystal structure. We report on developed procedures for the cultivation, harvest, and extraction of plant-based ISP as well as on their application potential in ice cream. Cultivars from winter rye were selected, sown and cultivated under semi-controlled conditions in glasshouses as well as in growth chambers applying a defined light and temperature protocol to stimulate the ISP formation in plants. Afterwards, the leaves were harvested and subjected to an extraction process. The ice structuring potential of the extracts was investigated by monitoring changes of the ice crystal structure after heat shocks. Finally, the efficacy of plant-based ISP was validated in ice cream products on a pilot scale. Ice cream was analyzed concerning quality attributes like e.g. fat agglomerates, ice crystal and air bubble size distribution, melting behavior, firmness and stability as well as sensory properties. Results were compared to ice cream produced with a reference ISP from artic fish confirming a significantly preserving effect of plant-based ISP on ice crystal structure in ice cream. Summarizing, this study demonstrates that plant-based ISP from winter rye leaves have the potential to prevent product damage by stabilizing the initial ice crystal structure of frozen foods and, therefore, maintain the ice cream quality also after temperature fluctuations occurring during transport and storage.

Controlled clustering of oil droplets in o/w emulsions: Rheological and tribological properties and the link to sensory perception

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Clustering of oil droplets allows modifying flow and lubrication properties of o/w emulsions. Oil droplets were clustered using electrostatic or chemical interactions. The effects of controlled droplet clustering on rheological, lubrication and sensory properties of emulsions were assessed. By combining emulsions stabilised with positively or negatively charged emulsifiers, oil droplet cluster size could be varied from 1 to 50 μm. By clustering protein-stabilised droplets with polyphenols, cluster size varied between 1 and 150 μm. The type of interactions affected the morphology of the clusters and the interaction strength, which was determined by measuring the critical strain. With increasing interaction strength, the clusters were denser with higher critical strain. When large, open clusters were obtained, the effective volume fraction of the oil increased 5x compared to single droplet emulsions of the same size, and thereby viscosity increased by 100x. Clustered emulsions were better lubricants than non-clustered emulsions; an improved formation of a lubricating layer might explain this observation. Creaminess and thickness intensities of clustered emulsions were significantly higher than those of single droplet emulsions and increased with increasing cluster size. Emulsions with strongly bound clusters were perceived as gritty, while weakly bound clusters did not give rise to a gritty sensation. Several sensory attributes were highly correlated to rheological properties, such as consistency, flow index, and critical strain. Tribological properties correlated well to sensory attributes when interactions of the emulsions with saliva were taken into account. We conclude that clustering of oil droplets provides an effective method to structure foods. Controlled structuring in liquids allows to tune flow and lubrication properties, and consequently perception of emulsions. This approach may enable the industry to design low-fat emulsions without thickeners while maintaining the perception of the full-fat version.

Symposium KB

Keynote Lecture B

SAXS imaging for the characterization of soft-matter

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Small-angle X-ray scattering (SAXS) probes structures in the size-range of one to several hundred nanometers. Raster scanning a sample through a focused X-ray beam allows to record SAXS pattern spatially resolved and can be combined with computed tomography to study the inside of three-dimensional samples. The information extracted from each scattering pattern can be used to construct images with different contrasts. Besides density also orientation of ultrastructure can be retrieved, for example the orientation of collagen fibrils in bone. The technique is mainly advantageous for hierarchical samples, since information about nanostructures can be obtained in macroscopic samples of several millimeter or even centimeters in size. A range of applications will be shown from biological tissues to various soft-matter systems. Linking for example the structural layers induced by the injection-molding process in semi-crystalline polymers used in food packaging industry. to their anisotropic mechanical properties. Combining SAXS imaging with microfluidics extends the method from imaging solid sample to mapping of nanostructures in flow.

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Symposium RS

Rheo-SANS and SAXS, Tomography

Multiscale in-situ characterisation of network formation and disruption in micronized fat crystal dispersions

John P. van Duynhoven[1], Tatiana Nikoleava[1], Adrian Voda[2], Ruud den Adel[2], Evgenii Velichko[3], Wim G. Bouwman[3], and Henk Van As[1]

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Supercritical melt micronization of fat crystals presents an appealing alternative for the conventional melt-cool routes for manufacturing fat based food products. By dispersing micronized fat crystals (MFC) in oil, fat crystallisation and network formation can be decoupled. This can bring significant simplifications of current industrial melt-cool processing routes of fat based food products. In order to assess the industrial application scope of these micronized fat crystal dispersions we carried out a multiscale investigation on MFC network formation and disruption by means of USAXS, rheo-SAXS, confocal Raman imaging and rheo-MRI. In situ measurements by Rheo-SAXS and rheo-MRI showed that upon dispersion in oil, MFC network formation was concomitant with recrystallisation. Oil type, temperature and shear rate collectively determined MFC recrystallisation rate, which inversely correlated with the strength of the resulting weak-link network where crystal aggregates are embedded in a continuous net of crystalline nanoplatelets. USAXS revealed that the rough surface of MFC nanoplatelets hampers stacking into one-dimensional aggregates ('TAGwoods'), which explains the high mass fractal dimension of the networks formed in MFC dispersions as compared to those formed by melt-cooling. RheoMRI demonstrated that application of shear to matured MFC networks led to a gradual and irreversible loss of yield stress. Shear did not have an impact on network fractal dimensions (USAXS) and also did not disrupt micronscale MFC aggregates (confocal Raman imaging). Rheo-SAXS revealed that loss of network strength can be attributed to release of nanoplatelets from the weak-link network. These subsequently align in the shear field and then undergo rapid recrystallisation. Our insights in the factors that govern MFC network formation and disruption bear relevance for simplified manufacturing of fat-based food products by effectively turning their design into a colloidal aggregation game.

Nanostructure of colloidal calcium phosphate in milk, cheese and related products studied by laboratory SAXS

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SAXS profiles of milk, cheese and related products were measured by laboratory SAXS with Mo target. Because of high penetration of Mo-Ka, we can use plate cell. Therefore, we can measure SAXS profiles of milk and cheese with same optics and cell. Another advantage of laboratory-SAXS is the flexibility of the machine time and much closer distance from production team. This make us possible to measure same series of milk and cheese with different aging time. It is also possible to measure raw milk without heat treating. Using these features, we focus on the change of CCP structure in milk with different heat treatment and also observe CCP structure continuously, from curd state to cheese picking up from one block of cheese. Inflexion point around 0.6 to 0.7 nm^{-1} which has been reported in many other studies has been observed in both milk and curd. In most of previous studies, it is attributed to CCP. Interestingly, the inflection point in the cheese after 4 days from flesh curd, shifts to 0.3 nm^{-1} and keep the position till four month aging. Some differences in SAXS profiles of milk with different pasteurize temperatures has also been observed. In this talk, we will also show SANS results of some dairy products measured in our in-house compact neutron source.

Brush-like polysaccharides with motif-specific interactions: Probing the architecture of gel assemblies using USANS/SANS and rheology

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Here we report the discovery and rheological and structural characterisation of a special class of polysaccharides found in seed mucilage of plants from the Plantago genus. These polysaccharides have a xylan backbone densely decorated with an array of side-chains. Almost 95% of xylose resides of the backbone are decorated with side-chains, which classifies these polymers as bottle-brushes. In particular, we have isolated two gel-forming arabinoxylan (AX) fractions by solvent specific extraction of Plantago ovata seed mucilage: AX-A(alkali) and AX-W(water). Both fractions are found to be neutral bottle-brush polysaccharides exclusively comprised of xylose and arabinose with similar molecular weight and linkage composition. Despite compositional similarity, their rheological and structural properties are markedly different. Using a combination of USANS/SANS with rheological characterisation techniques, we have established the hierarchical nature of hydrogel assembly, with pore sizes in the range between 30 and 1000 nm. Using enzymatic cleavage of terminal arabinoses of side chains we induced significant changes in rheological and structural properties of these complex arabinoxylans, which enabled us to corroborate that (i) hydrogen bonding between side chains of neighbouring molecules is a key driver of gel formation; (ii) the strength of hydrogen bonding is motif specific, which explains differences in physical properties between two fractions. It is envisaged that discovery of motif-dependent interactions will opens new opportunities for rational design of polysaccharides with targeted and highly tuned physical properties, providing a far-reaching potential for delivering novel tailor-made hydrocolloids for use in foods, pharmaceutical industry, and smart materials.

Full spatio-temporal elucidation of sheared multiphase materials

Stefan J. Gstöhl[1], Christian M. Schlepütz[2], Judith Wemmer[1], Jörg Läuger[3], Marco Stampanoni[4], Peter Fischer[1], and Erich J. Windhab[1]

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Multiphase materials exhibit complex flow behavior due to microstructural rearrangement and orientation of the disperse phases. In nature, landslides and other catastrophic events emphasize the importance to understand multiphasic microstructural evolution under controlled macroscopic mechanical stress or deformation. A causal understanding of complex flow comes when imaging techniques capable of adequately detecting the events couple in synchronism with rheometry. A full-volume approach to structure elucidation in real-time under full control of shear flow parameters is missing, especially at high forces to understand natural and industrial processes. Current methods are constrained by the mode of deformation, force control and the acquisition speed or they are restricted to fewer dimensions when operated at subseconds [1, 2]. We present a method to recover the microstructural evolution by time-resolved µCT X-Ray imaging while controlling or measuring all macroscopic rheological quantities. The setup consists of a modified two-motor rheometer in co-rotating mode with a custom 3D printed shear cell. A differential speed induces flow and allows full volume µCT scans, which are acquired synchronously with rheological data. To demonstrate the method, we present a study of glass beads immersed in different Newtonian fluids. Such dense granular model suspensions appear both in nature and industry. We apply a pressure-imposed instead of a volume-imposed flow [3] in combination with our method. Measurements extend into unknown territories in synchronism with full volume structural information. This method may generally prove microstructural theories in complex flow.

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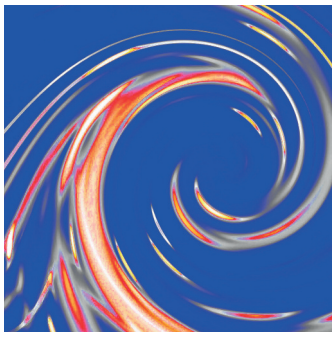

Tribology

From bulk to system behavior: Combining rheological and tribological testing in food oral processing

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Sensory perception of food is a highly complex process. It is influenced by various factors such as age, sex or consumer expectations, but also chemical and physical factors like saliva quantity and composition as well as food structure, which are covered within the field of food oral processing. During food intake, the tongue is pressed against the palate while sliding against it. Activation of mechanoreceptors on the oral surfaces, as influenced by the mixture of food and saliva, known as the bolus, partially influences the mouthfeel. The interaction of food and food-saliva mixtures with the tongue and the palate can be simulated by means of model scale tribological testing. Within this study, the interface between the tongue and the palate is represented by a glass/polydimethylsiloxane (PDMS) tribopair. Experiments were carried out on an MCR Tribometer with different types of food samples of different structures. Results are presented in the form of extended Stribeck curves. Complementary rheological measurements with food samples enable for discussing differences between bulk properties and tribosystem behavior. The authors also discuss the role of saliva and show how statistical analysis enables for insights into the correlation between the friction coefficient and particular mouthfeel attributes.

Designing mouth-mimicking rheo-tribometers to quantify oral processing

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What do brakes of a car, ice skates and a dessert pudding have in common? In all cases, consumer satisfaction strongly depends on the frictional behavior experienced. Friction is a common phenomenon, yet remains challenging to grasp. Frictional measurements, also referred to as tribology, have gained increasing appreciation in food science. It is believed that a combination of rheology and tribology will lead to better comprehension of oral processing and food perception in terms of creaminess, smoothness or even astringency. Such complex sensory attributes cannot be explained by rheological parameters only. Most commercial tribometers are however not designed to measure tribological properties between soft solids. In this presentation, we show three tribometers that we use to simulate in-mouth lubrication: a 3D-printed gel-on-gel rheo-tribometer, a commercial ball-on-pins tribometer and a modified pin-on-disk tribometer. We mimic the soft surfaces of the mouth using different hydrogels with various degrees of roughness. Overall, all hydrogels are slippery, and gave low friction. We use cryo-SEM and X-ray tomography to uncover the natural surface roughness of different hydrogels and find that a less homogenous surface leads to higher friction. To further highlight the mechanisms behind hydrogel friction across length scales we systematically designed patterned hydrogels. As lubricant, we use model food systems made of microgel particles of the same material, which were varied in size and stiffness. We find that hydrogels also provide good lubrication when added to lubricant fluids as microparticles. Using these soft tribological set-ups and model systems, we intend to pave the way for better determination of friction events for in-mouth conditions, and to gain correlations between sensory experiences and rheo-tribological behavior of soft solids.

A tribology test to measure friction of molten chocolate in a model tongue-palate contact

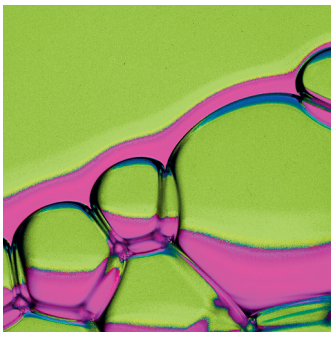
Georgios Samaras[1], Dimitrios Bikos[1], Josélio Vieira[2], Christoph Hartmann[3], Maria Charalambides[1], Yannis Hardalupas[1], Marc Masen[1], and Philippa Cann[1]

[1]Mechanical Engineering, Imperial College London, London SW7 2AZ, United Kingdom; [2]Nestlé Product Technology Centre, York, United Kingdom; [3]Nestlé Research Centre, Lausanne 1000, Switzerland

The perception of some food attributes (creaminess, smoothness) is related to mechanical stimulation and friction experienced in the tongue-palate contact during oral processing. The aim of this work is to study the effect of aeration on measured friction of chocolate samples and to relate this to panel sensory attributes. The study first develops a model test to mimic the tongue-palate interaction by measuring friction profiles of molten chocolate over a period of 120 s rubbing. Two chocolate samples with and without aeration were studied. In addition to chocolate, the effect of artificial saliva was tested. The experimental set up consists of a flat PDMS (polydimethylsiloxane, tongue model) disc loaded and rubbing against a stationary lower glass surface (palate model). Most of the samples showed the same general form of the friction-time curve: an initial increase to a maximum friction coefficient after 10 s of rubbing under load, followed by a decrease to a final stable friction value. Differences were observed in the maximum friction attained and the final equilibrium value. In all cases, this value was reached in less than 120 s. Addition of artificial saliva (phosphate buffered saline and mucin) to molten chocolate dissolves sugar and decreases the viscosity of molten chocolate so that lower friction coefficients are achieved. At the end of the test, the rubbed films on the lower slide were examined by optical microscopy and a structure degradation was observed in the rubbed surface. The new test provides a method to distinguish between the friction behaviour of aerated and non-aerated chocolate, including the effect of artificial saliva, in a rubbing low pressure contact. Therefore, the research highlights how aeration is related to the change of tribological properties during structural breakdown, providing insights into the mechanisms related to the oral perception of aerated foods.

WEDNESDAY AFTERNOON

Symposium EF



Emulsions, Foams and Interfaces

The effect of aeration on the mechanical and thermal response of chocolates during the oral process

Dimitrios Bikos[1], Georgios Samaras[1], Antonis Sergis[1], Maria Charalambides[1], Philippa Cann[1], Marc Masen[1], Yannis Hardalupas[1], Christoph Hartmann[2], and Josélio Vieira[3]

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Aeration in food products has been widely utilised in the food industry to develop healthier foods by reducing the energy content and improving the consumer's experience. Aeration in chocolates has a significant impact on the mechanical and thermal oral processes, which may lead to improved sensory attributes. This study aims to describe the early stages of oral processing and the thermo-mechanical phenomena taking place during mastication in two chocolate products, an aerated and a non-aerated one. Mechanical experiments were performed to highlight the effect of aeration on the mechanical performance. The results showed that the aerated chocolate exhibits a more brittle behaviour with a lower Young's modulus and fracture strain compared to the non-aerated product, suggesting higher fragmentation. The difference in the fragmentation behaviour is also observed through exploratory in-vivo mastication tests for both chocolates. The size distributions of the fragments extracted from the mastication tests were further analysed and fed into Finite Element models to evaluate the heat transfer rate for different chocolate fragment sizes in both aerated and non-aerated chocolate. The work provides a new insight of how aeration affects the initial stages of the oral process, leading to potential tools for designing products with improved consumer perception and/or higher nutritional profile.

Rheological properties of the low calorie mayonnaise that a part of the oil content was replaced with agar micro-gels

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Mayonnaise is a high oil content O/W emulsion. It is serious issue that the patients with metabolic syndrome are increasing. Therefore the development of low calorie foods is needed. If oil content of mayonnaise is reduced, its consistency decreases. It a big issue for low calorie mayonnaise development. We attempt to prepare a new type of low calorie mayonnaise that a part of the oil content was replaced with agar micro-gels. The agar micro-gels have completely spherical shape, because they were prepared using W/O emulsion system. The flow properties of model low calorie mayonnaise were analyzed with a weak-gel model. We reveal that the flow properties of the model low calorie mayonnaise which reduced quantity of oil to half, showed almost similar to traditional mayonnaise. The result show that the spherical shaped agar micro-gel particles was playing a role as an oil particle in the low calorie model mayonnaise. It is considered that the frictional behavior at the interface would be an important factor for the flow behavior of mayonnaise. Therefore, we also attempt to observe the nano-structure of the interfaces of the model mayonnaise using SAXS technique.

Symposium KA

Keynote Lecture A

Functional bacterial biofilms at interfaces

Patrick A. Rühs

ETH Zurich, Zurich 8093, Switzerland

Bacteria create and degrade materials with intricate precision and efficiency due to their highly effective and diverse metabolic activity. Despite recent advances to control the spatial composition and dynamic functionalities of bacteria in biotechnology, bacterial localization into complex 3D geometries remains a major challenge. Here I present innovative 3D structuring techniques to create bacteria-derived functional materials by combining the natural diverse metabolism of bacteria with the shape design freedom of 3D printing and emulsion/foam templating. We exploit the bacteria's natural ability to form biofilms at liquid-liquid and liquid-air interfaces [1-3] to create self-grown foams and microcapsules. Foams are formed by foaming a bacterial suspension of bacterial cellulose producing bacteria with Xanthan to prevent water drainage and Cremodan as a surfactant [4]. With microfluidics we further confined bacteria into emulsion droplets to create self-grown capsules. To obtain complete structural freedom, we developed a 3D printing technique that allows us to incorporate bacteria within the same 3D printed material [5]. We embedded bacteria in the biocompatible and functionalized 3D printing ink and printed two types of 'living materials' capable of degrading pollutants and of producing medically relevant bacterial cellulose. The approach presented will transform biotechnology by structuring microbes in 3D, allowing precise design and engineering of the material properties and form of microbe-laden functional materials.

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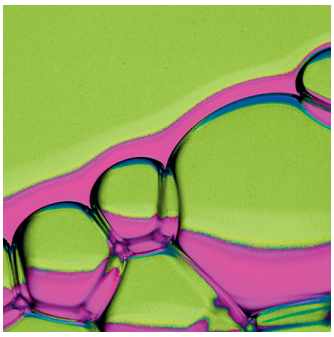

Emulsions, Foams and Interfaces

Rheology and microstructure of foams generated from viscous shear-thinning liquids using a continuous rotor-stator device

Saifullah Jabarkhyl[1], Pip Rayment[2], David M. Lloyd[2], Shiping Zhu[2], Damiano Rossetti[2], and Mostafa Barigou[1]

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Aeration in the food industry is becoming ubiquitous due to increasing demand from consumers for innovative aerated products (i.e. whipped cream, ice cream, mousse, etc.), with higher quality but lower calories and cost. For effective shelf life, it is essential to supress or eliminate some of the destabilisation mechanisms. One way of achieving this is by adding a food-grade surfactant (i.e. polyglycerol ester of fatty acid, PGE 55) which stabilises the air-water interface against bubble coarsening. Alternatively, polysaccharides such as xanthan gum (XG) or guar gum (GG) can be utilised to impart viscous properties to the continuous phase. The main aim of this study was to generate well-controlled foams from shear-thinning model fluids using a pilot-scale continuous rotor-stator device. The effects of the processing parameters (rotor speed, fluid residence time, air and liquid flowrate) and physical properties of the foaming solution (dynamic surface tension, continuous phase viscosity) on foam overrun, bubble size distribution and static stability were investigated. X-ray micro-Computed Tomography was used to non-invasively characterise the 3D microstructure of the foams. Increasing the gas-liquid ratio led to a finer foam texture with improved viscosity and stability. Together with rotational speed, dynamic surface tension and liquid rheology, these were the main parameters influencing foam microstructure and stability.

Nonlinear surface rheology and interfacial microstructure imaging of WPI particles and their constituents

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Food-grade Pickering stabilizers are often produced by cross-linking proteins, which typically results in a mixture of cross-linked particles and non-cross-linked proteins. This smaller material could potentially contribute to the interfacial behaviour of the total mixture. The aim of this work was to understand the interfacial properties of air/water interfaces stabilized by whey protein isolate (WPI) particle suspensions. The particles were produced by cold-induced gelation of WPI aggregates, using calcium crystals. To understand the interfacial properties of the total mixture, we have studied the whole hierarchy of structures, including native WPI, aggregates, and particles. Air/water interfaces were subjected to large amplitude oscillatory dilatation (LAOD) and shear (LAOS) using a drop tensiometer and a double wall ring geometry coupled to a rheometer. The non-linear responses of the LAOD and LAOS experiments were analysed using Lissajous plots. The plots of native WPI- and aggregates-stabilized interfaces showed a rheological behaviour of a viscoelastic solid, while interfaces stabilized by the particles tended to have a weaker and more fluid-like behaviour. The interfacial microstructure was studied by imaging Langmuir-Blodgett films of the protein systems using AFM. For the WPI particles we observed that they are present in the interfacial film, but are scattered throughout the film, separated by large areas, where smaller material is present. This suggests the presence of smaller material between the particles and also explains the weak layer found in the surface rheology experiments. The smaller material present in this WPI particle suspensions is surface active and plays an important role in interface stabilization, and could also influence the macroscopic properties of foams and emulsions. Based on these observations the WPI particle system does not behave as a classical Pickering system, but instead forms mixed interfaces consisting of particles and non-cross linked proteins.

Rheological study of selectively hydrolysed soy proteins in emulsions and gels

Wenjie Xia and Leonard M. Sagis

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Soy proteins have been widely used in many food products as functional and nutritional ingredients. Glycinin (11S) and ß-conglycinin (7S), the major components of soy protein, account for approximately 70% of the proteins in soybeans and are believed to determine the functional properties of soy protein. It is widely known that they have different structures and show considerable differences in functional properties, such as gelling and emulsifying properties. Many studies have demonstrated that proteolysis can improve the emulsifying and gelling properties of soy protein. Compared to the traditional limited proteolysis, only few studies attempt to enzymatically modify soy protein by selectively decomposing a specific component such as glycinin or ß-conglycinin. This selective proteolysis results in new soy protein products which consist of one kind of intact soy protein component, and peptides from the other main component. Recent studies claim that selective proteolysis can substantially improve the functionality of soy protein isolates. To test the functionality of selectively hydrolysed soy protein products, as a function of the degree of hydrolysis, we studied their interfacial rheology at the oil/water interface, with large amplitude dilatational rheology, and their gelling behaviour with small and large amplitude oscillatory shear rheology and creep tests. Preliminary results show that the degree of selective hydrolysis is a crucial parameter in the improvement of functionality and that low levels of proteolysis can even decrease functionality, particularly with respect to emulsification.

Obtain three-phase interfacial tension in coacervate/water/oil systems from coacervate filament thinning

Xiufeng Li[1], Philipp Erni[2], Jasper van der Gucht[1], and Renko de Vries[1]

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Biopolymer coacervates have been used extensively for encapsulation purposes, an application that sensitively depends on various interfacial tensions. The low interfacial tension of highly viscous coacervates are typically very difficult to measure. We present new surface tension data for a very viscous zein simple coacervate, obtained from capillary thinning of coacervate filaments [1]. Generally, coacervate filaments spontaneously break up in a second fluid if their configuration is out of the static Rayleigh-Plateau stability limit. This process is driven by the interfacial tension against viscous and elastic stress of the coacervate filament. If the coacervate viscosity and elasticity are known, interfacial tensions of the filament can be calculated from the thinning dynamics. This method is particularly suitable for coacervates in view of the typically low interfacial tension and high viscosity of coacervates, leading to slow thinning dynamics that can be precisely recorded. We have determined the interfacial tensions of highly viscous zein simple coacervates with their excess aqueous phase (0.83 mN/m), as well as with an oil phase (2.39 mN/m). Such data is crucial in order to determine whether encapsulation with simple zein coacervates could be effective or not.

[1] Dardelle G, Erni P: Three-phase interactions and interfacial transport phenomena in coacervate/oil/water systems, Advances in Colloid and Interface Science 206 (2014) 79-91.

international symposium on food rheology and structure

ISFRS 2019

8TH INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

Symposium KA

Keynote Lecture A

Nanoscale engineering of fat crystal networks: Structure to rheology

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Department of Food Science, University of Guelph, Guelph, ON N1G 2W1, Canada

A new understanding of the nature and organization of fat crystal networks (FCN), in particular at the nanoscale, has arisen in the last 8 years. These findings have shown that the smallest structural unit within such FCN is the crystalline nanoplatelet (CNP). CNPs aggregate into polycrystalline clusters in the micrometer range, ultimately forming a three-dimensional network, and trapping liquid oil within. This talk reviews the current methods available for the characterization of such nanoscale structure, including cryogenic transmission electron microscopy, ultra-small angle X-ray scattering (USAXS) and NMR. External fields can strongly influence this nanostructure. Here we show the effects of cooling rate and shear on CNP size, and how the nanostructure can be used as structural marker in the engineering of material properties. We will also show how USAXS can be effectively used to characterize all structural levels in a FCN in about 20 min in a non-destructive fashion, from solid-state structure to nanoplatelet size, to the fractal aggregation of CNPs into progressively larger clusters. In addition, USAXS has allowed us to propose the structural basis for the difference between a laminating fat, used to make croissants, and an all-purpose fat. Laminating fats are plastic, while all-purpose fats are more brittle. The difference in macroscopic rheological behavior was attributed to the existence of 3 characteristic structural length scales in laminating fats, while only two were identified in all-purpose fats. Mechanical failure would take place in a more gradual fashion (length-scale by length-scale) in a plastic materials as opposed to in a more catastrophic fashion in a brittle material. This provides a structural basis for material plasticity. Engineering the nanoscale structure of fats and oil is a reality today.

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Plenary Lectures

Global challenges and the critical needs of food science and technology

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Increasing global population and its increasing affluence is driving demand for food. Climate change and water shortages will disturb even current practices, a major problem for primary production. In the developed world, where relatively less money and human employment are spent on primary production, we see waste and overconsumption of calorie rich but micronutrient poor food types. As the rest of the world develops and becomes increasingly urbanised, we already see overweight in populations with access to convenience foods, simultaneously with nutrient restriction and imbalance remaining with the very poor. Global opportunities and challenges for the science base of food, nutrition and agriculture sustainability have been recently been analysed by scientific academy networks in an Inter Academy Partnership project. These reports have examined all science requirements across the food chain from primary production to diet and health. The aims of this paper are to focus on the conversion of raw materials to finished food products (food manufacturing), to reveal the vital position of food science and technology and the essential need to adapt and find solutions to global challenges in relation to food and nutrition security. We have examined the current capabilities of food science and technology, and the future challenges it will face to cope with changes in supply and demand, throughout future supply chains. After the identification of challenges, scenarios for research and development in the form of eight "Mission Oriented" programmes across the food chain are proposed. This mission approach allows the identification of the critical needs for future food science and technology, in the context of input from other scientific advances throughout the food chains of the future, and highlights the need for collaborative, multidisciplinary research.

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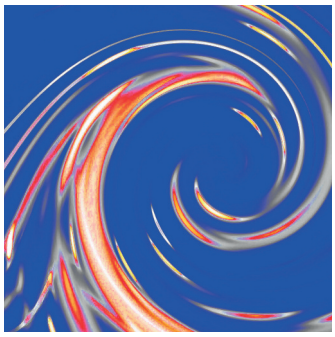

Tribology

From rheology to soft tribology of biocompatible microgels in complex continuum

Efren A. Andablo-Reyes and Anwesha Sarkar

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The mechanical performance of soft colloids, including their rheological and tribological response, is of great interest for biological applications. Biocompatible microgels have shown relevant lubrication properties to act as substitutes in biological contacts or as exogenously administered materials, such as food or drug delivery systems [1]. However, most studies to date have focused on dispersions in simple aqueous continuum, providing no information on their behaviour in complex media to predict their performance in relevant environments. Additionally, a satisfactory connection between rheological and lubrication properties of colloidal microgels characteristics is still lacking in literature. In this work, the shear rheology and lubrication performance of whey protein microgels dispersed in Newtonian or complex non-Newtonian xanthan gum solutions continuum was studied. Microgels diameter was estimated to be about 90 nm by means of dynamic light scattering. Shear viscosity of microgel dispersions and continuum was investigated using rotational rheology (cone-plate geometry) in a range of shear rates from 0.1 to 1000 1/s at fixed temperature of 37°C. Viscosity data was fitted using the Cross model to extrapolate the high shear rate plateau viscosity. In the high shear limit, relevant for lubrication performance, the viscosity of the continuum determines the role of soft microgel particles as thickening or thinning agents. Soft lubrication performance of the microgel dispersions was studied using a silicon ball on disc set-up under rolling-sliding conditions. Friction coefficient was measured on a range of entrainment speeds from 1 to 300 mm/s covering the mixed and hydrodynamic lubrication regimes. Connection between rheological and tribological properties of the dispersions was established using the second Newtonian plateau viscosity. The European Research Council is acknowledged for its financial support (ERC Starting Grant 2017, No. 757993).

[1] Sarkar et al.: Langmuir 33 (2017) 14699-14614.

Mechanistic insights into unexpected powder collapse in amorphous-crystalline mixtures

Xin Yi See[1], Laurent Forny[2], Marina Dupas-Langlet[2], Vincent Meunier[2], and Weibiao Zhou[3]

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Powdered mixtures of amorphous and crystalline solids are commonplace in the food industry. Unfortunately, ensuring powder stability may be challenging, given recent reports which showed that ingredients in dry amorphous-crystalline mixtures. In dry mixtures, each discrete particle retains its original amorphous or crystalline form inside a continuous phase of surrounding vapour: (i) Liquefied at lower relative humidity (RH) than the original crystals, (ii) had lower glass transition temperatures T_g than the original amorphous material and (iii) absorbed more moisture than the individual ingredients. Understanding and controlling this phenomenon (named 'sintescence') is critical to prevent instability issues such as caking from arising unexpectedly. Firstly, scanning electron microscopy-energy-dispersive X-ray spectroscopy was used to observe the progression of sintescence in dry amorphous maltodextrin (MD)-crystalline NaCl mixtures. Results showed that small amounts of amorphous MD-NaCl hybrid mixture were formed at the amorphous-crystalline interface during capillary condensation. Hybrid mixtures are amorphous molecular mixtures in which at least one ingredient is transformed from its crystalline state. To determine the impact of hybrid mixture formation, powder stability of dry and hybrid mixtures of MD and NaCl (9:1 by weight) were compared based on absorption behaviour and T_g. At 25°C, between 20 to 70%RH, hybrid mixtures absorbed the most moisture. Additionally, dry mixtures began to absorb more moisture than the weighted sum of their constituents after 50%RH. On the other hand, T_g data indicated that hybrid mixtures were least temperature stable, followed by dry mixtures then pure MD. Based on these findings, additional precautions should be taken to limit the exposure of dry and hybrid mixtures to moisture and heat as these mixtures tend to be less stable.

Symposium KB

Keynote Lecture B

Hydrocolloid-based food design considering interaction with saliva

Xinxin Li[1], Liling Zhang[1], Miroslaw M. Kasprzak[1], Mahamoud Hussein[2], Rebecca Ford[1], Stephen E. Harding[1], Peter Wilde[2], and Bettina Wolf[3]

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Following ingestion, food is mixed with saliva while is it being broken down by the mechanical action of teeth and tongue. The purpose of these oral processes is to generate a food bolus that is safe to swallow. Concurrently, flavours are released and mouthfeel perceptions are generated. The interaction between food components goes beyond simple dilution. It may therefore be exploited in the design of foods by changing functional properties through physical interaction or amylase mediated hydrolysis of starches. Comparing the rheological properties of guar gum to xanthan gum mixed with saliva or water, it was shown that for rigid rod polymers extensional viscosity increases. As this observation is of potential interest in the context of designing foods for dysphagia patients, for which increased extensional viscosity assists a safe swallow an extension of this study to anther hydrocolloid with predicted rigid rod conformation, and its molecular characterisation, will be presented. Amylase mediated hydrolysis of starches may also be exploited to affect viscosity, but, more interestingly, it can be taken advantage of in the design of emulsion interfaces for oral destabilisation. Recent advances in the stabilisation and breakdown of starch stabilised emulsions, including the use of non-chemically modified starches, to contribute to salt and sugar reduction targets will be presented.

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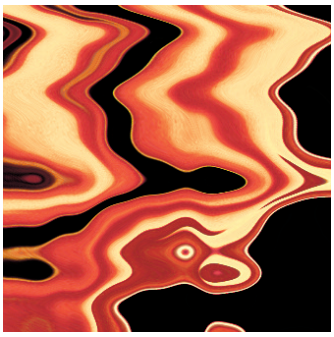

Physiological-guided Rheology

Rheology during oral processing and swallowing

Mats Stading[1], Waqas Mohammad Qazi[1], Ekberg Olle[2], Patricia Lopez Sanchez[1], Vincent Schaller[3], and Johansson Christer[3]

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The complete sequence of oral processing and swallowing is an intricate combination of voluntary and involuntary actions. The process takes place in a complex flow geometry that involves a mixture of shear and extensional flow during which texture, aroma and taste are perceived. We seldom register the process, but during the few seconds it lasts we form our complete opinion of the food we eat. The main oral processing activities are chewing, production of saliva and transportation of the food and drink, including the prepared bolus. We know relatively little of how rheological and mechanical properties of the food change during oral processing, much due to lack of suitable monitoring techniques. By remote, non-invasive monitoring the rotation of magnetic ironoxide particles (E172) it is possible to follow the nano-rheology of the ingested food. Fluids with increasing rheological complexity have been characterized by nano-rheology and small amplitude oscillatory shear techniques. The fluids range from water, through PEG to xanthan and gelling alginate, and the results show good correspondence for the simpler fluids whereas fluid and gel microstructure have to be taken into account for the gelling systems. Swallowing disorders, or dysphagia, is a growing problem especially as the population gets older. In the age group above 70, 40 % suffer due to factors such as degenerative diseases and side effects of medication. These persons must eat texture adjusted foods. The Gothenburg Throat is an in vitro model of the pharynx, designed to elucidate the effect of food bolus rheology on swallowing and to simulate disorders. A bolus is injected at controlled volume and speed, and the pressure at four different places is monitored together with the velocity profile during the passage through the pharynx. The model has been thoroughly validated and bolus flow determined. Shear rates for commonly used thickeners have been observed in the range 10-200 1/s and have also been validated by in-vivo studies.

Determining the rheology of fluids for dysphagia treatment in the field

Adam S. Burbidge

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Dysphagia (swallow dysfunction) is a condition affecting large numbers of elderly people, which, if untreated, can lead to aspiration pneumonia and frequently death. Fortunately, in many cases, a controlled modification of fluid rheology can mitigate the dysfunction. Nevetheless, measuring the rheology of modified fluids in a simple manner in the absence of expensive equipment presents a significant challenge. In this contribution we will discuss this measurement issue from the perspective of the recently introduced IDDSI standard 'syringe' test. How does this relate to previous standards and the actual conditions which occur in a real swallowing flow? Are there cases in which certain kinds of fluids could give unexpected results?

Impact of interfacial and bulk interactions between cellulose ethers and bile salts on the control of lipid digestion

Jennifer Zornjak and Cristina Fernández-Fraguas

Virginia Tech, Blacksburg, VA, United States

Lipid digestion is a multistage process that relies on several colloidal and interfacial mechanisms to optimize the enzymatic hydrolysis of lipids (lipolysis) and transport processes. Delaying lipid digestion kinetics may have important implications for preventing hypercholesterolemia and hyperlipidemia, and so for combating chronic diseases. The adsorption of bile salts (BS) onto the surface of lipid droplets is a key step for the progression of duodenal lipolysis, and hence, lipid digestion could be controlled ultimately by limiting access of BS to the lipid surface. This study aimed to investigate the potential of Cellulose ethers (CE), surface-active, non-ionic and indigestible biopolymers, to control lipid digestion either by creating interfaces that resist complete displacement by BS, or by sequestering BS in the bulk phase. We used surface-analysis techniques to explore the formation of CE-BS layers onto hydrophobic surfaces by means of sequential adsorption, as representative of interfacial interactions, and by simultaneous adsorption, to study how CE-BS bulk interactions affect disruption of the lipid surface. In order to investigate the impact of BS structure on BS interfacial behavior and their interactions with CE, sodium-taurocholate (NaTC) and taurodeoxycholate (NaTDC) were chosen as representative BS. We discuss results obtained with Hydroxy-propylcellulose (HPC). Subtle differences in BS structure and formation of micelles affected both, their adsorption onto bare surfaces and the properties of the HPC interfacial layer. The route of formation of the HPC-BS layer played a decisive role on their displacement by BS. HPC seemed to be more effective at binding BS in the bulk than forming a strong pre-adsorbed layer resistant to BS displacement. In vitro digestion of HPC-stabilized emulsions showed delayed lipid digestion when NaTDC was used to simulate duodenal conditions. These findings can be exploited in developing novel matrices with enhanced functionality to modulate lipid digestion.

Tailoring emulsions for controlled lipid release: Establishing in vitro - in vivo correlation for digestion of lipids

Nathalie Scheuble, Andreas Steingoetter, and Peter Fischer

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The use of oil-in-water emulsions for controlled lipid release is of increasing relevance for poorly water-soluble drugs and gained major interest for treating obesity. In this conceptual study, we highlight the relevance of interfacial rheology combined with neutron reflectivity measurements in designing emulsion systems with stimuli-responsive biopolymers to generate specific lipid digestion kinetics. Stimuli-responsive biopolymers change their mechanical and emulsion stabilization properties at specific physicochemical conditions present in the human environment. We used whey protein isolate because of its sensitivity to pH, methylcellulose because of its sensitivity to temperature, and nanocrystalline cellulose because of its sensitivity to ionic strength [1]. The biopolymer-dependent effects on gastric lipolysis and gastric emulsion structuring, more precisely droplet size and viscosity, during digestion and resulting pancreatic lipolysis was investigated in-vitro. In order to establish a valid in-vitro/in-vivo correlation, emulsion structures formed during gastric digestion in-vitro were validated by in-vivo magnetic resonance imaging experiments visualizing emulsion structuring and stomach emptying [2]. In parallel, in-vivo lipid sensing and release patterns were investigated by repetitive measurements of measuring plasma triglycerides and cholecystocinin concentrations in healthy human volunteers. By taking all relevant physicochemical and mechanical processes of human digestion into account, we are thus able to propose design concepts for food and drug delivery systems [3, 4].

[1] Scheuble N et al.: Biomacromolecules 17 (2016) 3328.
[2] Steingoetter A et al.: J. Nutrit. 147 (2017) 706.
[3] Scheuble N et al.: Anal. Chem. 89 (2017) 9116.
[4] Scheuble N et al.: ACS Appl. Mater. Interfaces 10 (2018) 17571.

THURSDAY MORNING

Symposium PL

Plenary Lectures

Physiology guided food structure and process design for tailored rheology and functionality

Erich J. Windhab

IFNH, ETH Zurich, Zurich 8092, Switzerland

Physiology guidance for food quality tailoring means focusing on optimally achievable pleasure (sensory related) and health (nutrition related) benefits. This can satisfy consumers' preferences and needs, but may not be sufficient for appropriate acceptance. For the latter environmental sustainability of production methods and naturalness of resulting products are of increasing importance for millennials and generation Z. Sustainability and naturalness are connected to the right choice of food raw materials and minimal/smart processing methodologies. Accordingly, the overarching task is to sustainably produce food of natural characteristics for optimal, physiology guided pleasure and health supporting impact on consumers. Rheological characteristics of food systems play a prominent role for physiological interactions during meal preparation, oral processing, swallowing and digestion. The flow behavior of food in its forms as masticated paste, bolus, chyme or gastric juice component determines oro-gastro-intestinal mass transfer of functional components. This holds for aroma/taste related aspects in the oral cavity or retro-nasal space as well as for micro-/macronutrients in the GI-tract. Moreover, locally acting flow stresses determined by the food rheology and the related digestion-processing flow fields, impact on disintegration of food structure together with corresponding bio-chemical reactions down to the molecular level on which metabolic reactions take place. In order to impact on physiological interactions by the rheology of a food along the OGI-tract, pre-structuring based on formulation, in-factory processing, choice of storage format and meal preparation processing can be modified. elated examples will be given for functional multiphase food systems. In addition, selected processing methodologies to generate such with optimal naturalness under environmentally friendly processing conditions along the entire food value chain will be demonstrated.

Microstructure design: A key for processing of food systems

Anne-Marie Hermansson

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Food structure processing has been a central research theme for many decades. Almost all challenges require engineering solutions, where inherent structure properties can be mastered during structure formation to deliver the desired food quality. The research at ETH by Professor Windhab´s group has demonstrated the power of microstructure processing not only from a strict engineering point of view but also sophisticated process-structure design for microcapsules and interphases with application for a wide range of food and pharmaceutical products and lately also physiology guided approaches. Collaboration over 20 years, where the engineering approach has been combined with an in depths of microstructure understanding has been very fruitful. Results will be presented from model studies of biopolymers, where process parameters such as shear and kinetics of heating/cooling can have a dramatic effect on structure formation and related properties as well as more sophisticated studies of process design, where a combination of process parameters and kinetic arrest can be used to create new product characteristics. In addition, precision engineering such as microfluidics will be shown to produce exact multiphase structures. Such structures can be used not only to give the desired texture but also to design release properties of importance both for food and pharmaceutical products. The area of food processing, structure and functionality will always be crucial for product and process development and new techniques and interdisciplinary collaboration provides powerful tools to meet technological and societal challenges.

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Symposium KA

Keynote Lecture A

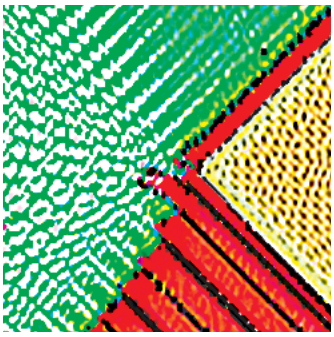
Food 4D: Adjusting functional properties by three-dimensional structuring

Christoph Denkel and Tobias Kistler

School of Agricultural, Forest and Food Sciences, Bern University of Applied Sciences, Bern, Switzerland

Additive manufacturing techniques in the food industry, also termed 3D Food Printing, are bringing new approaches to the future of food production. At present, there is a lack of fundamental knowledge regarding suitable materials, their functional properties and relevant production speeds. Furthermore, current concepts show a lacking in added value. On the other hand, additive manufacturing offers the possibility to introduce new sensory or functional properties by modulating the three-dimensional structure of products via an additional structuring level gained by the separation of materials in terms of composition and localization. This requires a combination of adapting existing approaches with new ways of thinking by food engineers. Major product related organoleptic aspects include texture and aroma sensory perception, both the overall perception of texture and aroma are strongly dependent on the absolute and relative perceptibility of different product compartments and their interfaces. Considering that such perceptions are very specific in their nature, there exists definite potential to tailor product textural and sensory profiles in a highly individualized manner whilst altering the way in which the taste of certain ingredients is perceived. In this presentation, we show how single-phase and two-phase product systems provide an insight into the strategies with which sensory perception can be influenced using the three-dimensional structure of foods whilst retaining overall product composition.

Symposium PF



3D Printing of Food

Characterization of casein-whey protein mixtures differing in pH, protein content and denaturation parameters for extrusion based Food Layered Manufacturing

Kilian J. Daffner, Tom Mills, and Ian T. Norton

School of Chemical Engineering, University of Birmingham, Birmingham B15 2TT, United Kingdom

3D-Printing of food (also Food Layered Manufacturing, FLM) allows for creation of complex and novel shapes and designs, personalized nutrition and fast production of small amounts of customized dishes. Moreover, no storage - and less distribution costs make the whole process much more sustainable than the current mass production. Since several years, different types of food were 3D-printed by researchers, including chocolate, cake frosting, meat purees, dough, hydrocolloids and more. To date, FLM of dairy materials was conducted using different types of feed materials including processed heated cheese, sodium caseinate combined with pectin, sucrose and starch as well as cold acidified milk concentrates differing in pH and casein content. Goal of the current study is to find a library of edible and printable formulations of dairy materials for extrusion based 3D-Printing, including a full phase transition from sol to gel. By changing pre-process parameters (denaturation temperature, - time and - pH) and formulations parameters (protein type, - content and pH) cold-acidified samples are gelled via a heating step (pH-T route). Formulations undergo a phase transition in a nozzle or a printing bed, from sol to gel, and are deposited layer by layer. Parameters like the particle size, the phase transition temperature as well as the aggregation rate help to characterize formulations regarding their printability. Depending on the heating pH, formulations can be manipulated regarding their phase transition temperature and their aggregation rate. Rheological measurements showed that sol-gel phase transition temperature (G'=1 Pa) decreased and aggregation rate of the casein-whey protein mixtures increased with increasing heating pH. A schematic model of the influence of the heating pH on the behavior of casein-whey protein mixtures was developed. Printed gels will be post-characterized via rheology, CLSM or syneresis to better understand their microstructure and the influence of the printing process on the proteins.

Extrusion-based 3D printing of food pastes: Correlating rheological properties with printing behaviour

Sicong Zhu[1], Maarten A. I. Schutyser[1], Markus Stieger[2], and Atze Jan van der Goot[1]

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Development of 3D food printing applications requires in-depth knowledge on printing behaviour of food materials. In extrusion-based 3D printing the rheology of the food paste is vital to achieve successful printing, e.g. in terms of dispensing behaviour and stability. The objective of this research is to predict printability of formulations from simple rheological properties. We used tomato paste as a model system to investigate the correlation between printing stability, dispensability and their rheological properties. In this study, printing stability was defined as the normal stress acting on the bottom layer of printed objects at the collapsing moment. The results showed a linear correlation between ingredient's flow stress, viscosity and corresponding printing stability. Dispensability was defined as the dispensing pressure needed to extrude printing material out of the syringe barrel. The higher the dispensing pressure, the worse the printability. It was found in our study that the dispensing pressure needed to extrude tomato pastes increased linearly with increasing flow stress. A larger nozzle diameter resulted in better printing stability and lower dispensing pressure. From additional experiments with additional water-containing foods its was found that these aligned well with the results of tomato paste; however, for fat-based products showed different printing behaviour was observed. The overall results suggest that printing behaviour of water-based food paste systems can be well predicted with corresponding rheological properties, e.g. flow stress obtained by oscillatory stress sweep.

Extrusion 3D printing of nutraceutical oral dosage forms formulated with oleogels and phytosterols mixtures

Ivana M. Cotabarren[1], Sofia Cruces[2], and Camila A. Palla[1]

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Within the concept of personalized nutrition arises the demand for "tailor-made" technological solutions that combine nutrients and functional compounds. In this regard, 3D printing emerges as a group of technologies capable of producing customized formulas with the desired shape, dimension, and microstructure. The extrusion-based 3D printing (E3DP) method is the most widely adopted for obtention of foods and pharmaceutical forms. The aim of this work was to evaluate the production of nutraceutical solid forms by E3DP using mixtures of monoglycerides (MG) oleogels and phytosterols (PS) as printing materials. To this purpose, molten oleogels were prepared using MG (10 or 20 %wt) and high oleic sunflower oil. Printing materials were obtained adding variable amounts of PS to oleogels, between 0.2 and 0.5 wt PS/wt oleogel. An ad-hoc extrusion 3D printer composed of a heated syringe and a cooling build platform was used. The hot mixtures were introduced into the syringe and the solid forms were printed under previously defined parameter setting. Oscillatory temperature sweep tests were carried out to determine the mixtures gel point in order to select appropriate printing temperatures. Mechanical properties of printed solid forms were obtained by compression test. The mixtures gelation temperature increased with the increase of PS content. Values ranged between 70.3 and 91.1°C and 55.3 and 95.2°C for oleogels formulated with 10 and 20 %wt of MG, respectively. Furthermore, it was found that solid forms were successfully printed when using mixtures containing a maximum of 0.3 wt PS/wt oleogel and 0.4 wt PS/wt oleogel for oleogels formulated with 10 and 20 %wt of MG, respectively. All these solid forms were structurally stable, with hardness values that increased with the rise in PS and MG content. The highest value of hardness was 12.55 N, obtained for the mixture formulated with 0.4 wt PS/wt oleogel and 20 %wt of MG.

The effect of rheological properties of oleogels on 3D printing cheese cake

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3D food printing faces three main challenges i) food materials printability, ii) processing parameter, and iii) post-processing parameters. The former is determined by the ability of a food material to extrude from the nozzle of a printer and hold its structure after deposition. In this regard, rheological properties play a crucial role determining the printability of a material, and predicting the accuracy of the 3D printing process. Here we present the effect of rheological properties of different oleogels (i.e. structure oil-based food using oleogelators; structuring agents for oils) on 3D printing process. Different oleogelators were used and the concentration of the oleogelators were optimized to increase the gel strength of the material and improve the printing accuracy. In addition, the effect of oleogel in 3D printing accuracy, rheological and textural properties of cheese cake was evaluated. Finally, results showed that the printability of the cheese cake highly depends on the mechanical shear stress, storage modulus, and dynamic mechanical loss tangents of the oleogels used.

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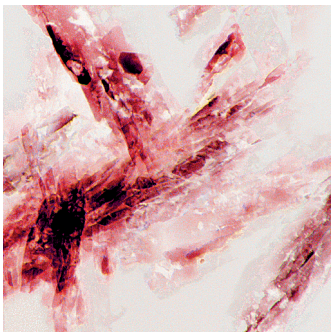

Meat Analogues

Relationship of compositional, mechanical and textural properties of extruded pasta containing specific varieties of quinoa (Chenopodium quinoa)

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Quinoa epitomizes the drive for healthier foods with ethnic concepts in developed countries, particularly among millennials. As a result, the popularity of quinoa as a gluten-free grocery item has steadily grown over the last 20 years. Despite this, little is known about the impact of specific varieties on processed foods. The purpose of this study was to examine the impact of quinoa varieties (variety type and content) on the mechanical and textural properties of buckwheat-based extruded pasta (spaghetti). Peruvian native (var. Rosada Taraco, Kuchivila, Negra Kollana and Mistura) and Latvian-grown (var. Titicaca) varieties were independently incorporated to pasta between 5 and 20 %(w/w). Mechanical properties such as firmness (M-firm), hardness (M-hard) and stickiness (M-stick) of cooked pasta were investigated. Similarly, optimal cooking time and cooking loss were determined. Six judges were trained (up to 12 hours) on the development of sensory descriptors, definitions and references associated to texture (firmness, cohesiveness, stickiness, smoothness, grainy), taste (overall taste, bitterness) of spaghetti-like pasta. Correlations between compositional, mechanical and textural characteristics were investigated using Principal component analysis (PCA). Pasta containing 20 % quinoa var. Negra Kollana, which presented the largest content of fibre and lowest content of saponins, were strongly associated to firmness, cohesiveness, smoothness, and low cooking loss. Conversely, pasta containing 20 % quinoa var. Titicaca was associated to low firmness, long cooking time and high cooking loss. Despite having the highest content of saponins, pastas containing 20 % quinoa var. Rosada Taraco and Mistura were considerably firm and cohesive. Unlike the rest, pasta containing 5 % quinoa var. Kuchivila showed greater firmness, cohesiveness, M-firm and M-hard than the one containing 20 %. In conclusion, the relationship of compositional, mechanical and textural properties of pasta was strongly variety-dependent.

Plant attitude: Great taste from within

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Meat substitutes are becoming popular all around the world, especially with new texturing technologies like high moisture extrusion cooking. Today, most producers flavor meat substitutes after extrusion, via marination processes, which tend to flavor an outside layer of the product. As consumers start demanding larger meat substitute pieces, marination techniques cannot deliver the authentic experience of chewing a piece of meat while releasing flavor with each chew. The solution is to flavor meat substitutes pre-extrusion, providing flavoring throughout the pieces, and delivering a more authentic meat profile. However, flavor components may affect texture formation and texture can affect flavor perception. Why is this happening and how can we address it?

Processing of novel plant protein and fibre by high moisture extrusion cooking

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Sixty percent of all vegetable protein produced is used as animal feed to still our tremendous appetite for meat. Meat production is associated with negative impact on the environment and is causing large greenhouse gas emissions. A more sustainable solution is therefore to forgo the animal and produce fibrous meat-like textures directly from plant proteins and fibres using high moisture extrusion cooking (HMEC). During HMEC protein and fibre rich powders get hydrated, mixed, sheared, heated, texturized, to create a melt and get finally cooled under a shear and elongational flow in a long die to create fibrous structures. The resulting products are so called high moisture meat analogues and provide a similar mouthfeel experience to meat. So far, soy bean protein and wheat gluten are used as raw materials. These show favourable processing conditions but are not well suited for people suffering from soybean allergy or gluten intolerance. We have investigated the usage of proteins from other pulses and oilseeds and identified key aspects in raw material requirements, and recipe and process design rules to tailor the product structure and properties. These novel texturized products will provide a more sustainable alternative to meat with only a fraction of the environmental impact.

international symposium on food rheology and structure

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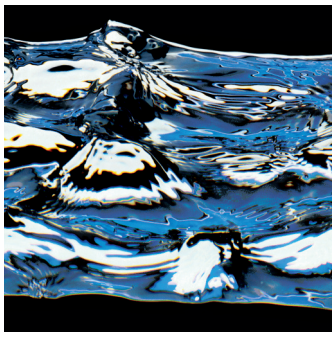
8TH INTERNATIONAL SYMPOSIUM ON FOOD RHEOLOGY AND STRUCTURE

Poster Session

Symposium PO



Poster Session

Rheological and inner structural assessments for complex materials using ultrasonic spinning rheometry

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Non-contact and non-invasive techniques to assess rheological properties and inner structural visualization are strongly desired from the aspects regarding quality control and efficiency improvement of productive process in the industry. We proposed a novel methodology to assess the rheological properties (fluid) and inner structure (solid) as a non-contact and non-invasive technique, namely ultrasonic spinning rheometry (USR). The final goal of the rheometry is to develop in-line technique by applying the theoretical concept of USR; it will make a significant contribution to various kinds of fields. The theoretical basis of USR is an essential piece of information for the development of the in-line rheometry (see Tasaka et al.: An idea to contactless in-line rheometry using ultrasonic velocity profiling). In this research, we conducted experiments to evaluate the rheological properties of complex fluids and visualize the inner structure of fruits utilizing ultrasonic echography and velocity profiling. Polymer solution, clay dispersion, Newtonian fluid with dispersed bubbles, liquid food, and fresh fruits were chosen as test materials. Measuring velocity fluctuations in unsteady oscillation, by applying the equivalent methodology (USR), the rheological characteristics of shear-thinning, viscoelastic, thixotropic, multi-phase fluids could be evaluated. In addition, measuring Doppler velocity and echo amplitude in a rigid rotation, inner structures in solid materials (e.g. rubber ball, apple, tomato, kiwi, and cherry preserved in syrup) were visualized. In conclusion, considering the advantages and limitations of conventional rheometers and USR, USR can be expected to offer complementary rheometry together with conventional rheometers on rheological and inner structural assessments.

High throughput size distribution analysis using an image processing tool based on template matching

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The bubble size distribution is a major characteristics of foams determining the interfacial area, diffusive transport, rheology and strength. Understanding and predicting these properties inevitably requires reliable methods for obtaining size distribution of the dispersed gas phase. Precision and statistical significance of the size distribution evaluation is frequently limited by the quantity of bubbles captured in manual analysis. To overcome this limitation, we have developed an endoscopic foam imaging setup and a software tool enabling to detect the size of high quantities of bubbles within minutes based on a template matching technique [1]. The foam images are screened for matches with a scalable template, i.e. a circle, which has a similar pixel pattern as the bubbles in the foam. Possible matches are validated and position and scale of the final matches are displayed. This method overcomes the difficulties with edge detection algorithms. The images are screened for entire objects rather than continuous object edges, which makes it more robust against losses in detection caused by broken edge lines or false detection because of overlapping objects. The template matching method reaches its applications limits, in cases were self-similarity of the objects vanishes, i.e. during ongoing drying of foams when bubbles undergo a transition from spherical to distinct polyhedral objects. The software tool is not limited to bubble size distribution determination but inheres the possibility to analyze size distribution of any kind of uniform objects even with varying aspect ratios.

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Rheo-microscope tool in the food research

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The MMA is a compact, self-contained microscope for high quality sample imaging in conjunction with rheological measurements using the DHR. The MMA attaches to three lower mounting points below the DHR dress cover. An optical stage attached to the Smart Swap(tm) base provides a transparent lower plate through which the sample can be observed during rheological experiments. The MMA can be operated in parallel plate or cone-and-plate configurations and collects images or videos in the shear-vorticity plane, facing up through the lower plate. A precision x-y-z micrometer-based positioning system allows the microscope's field of view to be adjusted within the sample, from the center to the edge of the plate and anywhere between. Sample illumination is provided by a blue LED light source (nominal wavelength of 470 nm). A cross-polarizer is included with the MMA and a dichroic beam splitter is available as an optional add-on for fluorescence microscopy. In this poster, we present the advantages of using MMA and theology in the field of emulsions.

Screening of textural properties of starters and proteins during yogurt preparation

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Milk fermentation in yogurt preparation has become an important field of research in the recent years. The demand of new recipes is continuously growing. Especially the development of new and innovative starters and proteins is of outmost importance. This work is focused on the use of passive microrheology as tool for protein and ferment evaluation. Rheolaser Master© uses Multi Speckle Diffusing Wave Spectroscopy (MS-DWS) for the evaluation of structural and textural properties of yogurts during its fabrication. A coherent laser beam is applied to the sample and multiple scattered by the scatterers (particles, droplets, fibers...) present in the sample. The interfering backscattering waves form an interference image (speckle image), which is detected by a multi-pixel detector. The scatterers' motion is directly related to the spot movement of the speckle image and can be analysed in the dynamic mode. The determination of the Mean Square Displacement (MSD) curve enables to characterize completely the viscoelastic properties of the sample. This work will present the results of a regional funded research program, called "Rheolact'". Its innovative aim was the correlation of the microrheological data obtained during fermentation (day 0 of yogurt fabrication) with sensorial tests done at day 7 (earliest possible day of consumption). The project had two main steps: Firstly, several recipes with different parameters (whole or skimmed milk, different starters, different proteins), were tested, both with Rheolaser Master and with a trained panel jury. A first series of tests allowed to create a data base for the development of descriptive models via multiparametric linear regression. In a second series, the models were tested for validation. The models can predict risk of syneresis, heterogeneity after stirring, firmness, and mouth feeling.

Development and application of micro-computed tomography and proton NMR to determine the structural changes of cooked noodles

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Several conventional methods have been applied to investigate the physicochemical change of noodles during cooking. However, the conventional methods that are generally dependent on visual and sensory properties are not suitable for quantitatively estimating the structural properties of cooked noodles. Therefore, there is an urgent need to develop more reliable and objective methods for the noodle industry. In this study, the structural changes of cooked noodles were quantitatively characterized by using microcomputed tomography (micro-CT) and proton NMR depending on cooking times and then correlated with several conventional methods. The noodles gradually absorbed water with increasing cooking times, and their white core became thinner and finally disappeared when they were completely cooked. When the noodles with different cooking times were subjected to micro-CT analysis, the 3-dimensional images of the cooked noodles clearly showed density differences over cooking time in accordance with visual appearance, and their degree of cooking could be successfully calculated based on their volumes collected from the stacked cross-sectional images. Regarding water mobility in cooked noodles, the proton NMR analysis demonstrated that the cooked noodles exhibited three obvious water distributions. The proportions of weakly bound water increased along with cooking time, while tightly bound water decreased. Furthermore, the increasing tendency of T2 relaxation time was observed with increasing cooking times. The noodles cooked for longer times typically exhibited a softer texture and also higher cold initial/lower final viscosities. Pearson correlation and principal component analysis demonstrated that the parameters obtained by micro-CT and proton NMR were well-correlated with those by the conventional methods in a highly linear way.

Modelling heat-induced viscosity of milk protein concentrate using kinetic data

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Liquid milk protein concentrate (MPC) obtained after membrane filtration is frequently subjected to heat treatment (high temperature - short time) to inactivate microbiological organisms. However, such heat treatments result in denaturation and aggregation of proteins leading to an increase in viscosity and subsequently causing adverse effects in the manufacturing process such as a reduction in pump efficiencies, fouling on evaporation distribution plates/tubes of calandria, and effectively limiting the total solids level achievable prior to spray drying. This work aimed to model the effect of heat treatment on viscosity of milk protein concentrate (MPC) using kinetic data. MPC obtained after ultrafiltration (19.8% TS and 17.3%, w/w, protein) was subjected to heat treatment temperatures ranging from 65 to 120°C. Heat treatment at high temperature and short time (i.e., 100 and 120°C×30 s) led to a significant increase in viscosity in MPC systems. Second-order reaction kinetic models proved a better fit than zero- or first-order models when fitted for viscosity response to heat treatment. A distinct deviation in the slope of the Arrhenius plot at 77.9°C correlated to a significant increase in the rate of viscosity development at temperatures above this. The sharp deviation in the Arrhenius plot can be attributed to the transition of whey protein from the unfolding to the aggregation stage. This study demonstrated that heat-induced viscosity of MPC as a result of protein denaturation/aggregation can be successfully modelled in response to thermal treatment, providing useful new information in predicting the effect of thermal treatment on viscosity of MPC.

Rheological characterization of sugar inhibited CO_2 hydrate slurries

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Gas hydrates are non-stoichiometric crystalline structures consisting of 'guest' gas molecules entrapped in a 3D lattice of polyhedra formed from 'host' water molecules. These structures, stable under moderate pressures and low temperatures, are often found in natural gas or oil pipelines where they can cause severe plugs. Flow assurance covering gas hydrate prediction and prevention e.g. via thermodynamic or kinetic inhibition has therefore been widely studied. In this study, flow properties of sugar inhibited CO2 hydrate slurries were studied at varying shear rates in an in-house built high-pressure loop (4.5 L, pipe diameter ½") and rotational rheometer (MRC 302) with a vane geometry (25 ml) under pressures of the CO2 hydrate stability zone (30 - 35 bar) bar and temperatures between 0 and 4°C. The results from the two methodologies were interpreted with a rigorous thermodynamic model resulting in the volumetric fraction of the suspended solid hydrate phase in a concentrated liquid sugar solution. Classical suspension rheology models were fitted to the acquired data. With the growing gas hydrate solid phase, the sugar solution becomes more concentrated and the system converges to an eutectic point followed by blocking. Combining thermodynamic and rheological modeling it was possible to derive critical volumetric hydrate fractions for blockage prevention. The rheometer data were next compared to high-pressure flow loop data for 50 wt% sugar solutions. The two methodologies were in agreement for similar solid volumetric fractions. The conformity of a precise laboratory scale rheometer with the flow loop data justified the use of a robust pilot-plant loop reactor for flow measurements on-line.

Rheology of Swiss cheese fondue

Pascal Bertsch, Laura Savorani, and Peter Fischer

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Cheese fondue is a traditional Swiss dish prepared by melting cheese under the addition of wine, starch, and seasoning. Thus, fondue is a multi-phase system with complex colloidal interactions and rheology. The viscosity of fondue is of particular importance for mouthfeel, flavor perception, and making the cheese cling to the bread for consumption. We tackled the complex multi-phase system fondue from a material science perspective, providing a scientific framework for the influence of fondue ingredients and their interactions on the rheology of cheese fondue [1]. Fondue can be considered a water continuous system whose viscosity is influenced by the interactions of its main colloidal ingredients: dispersed casein micelles, emulsified fat droplets, and swollen starch granules. A model moitié-moitié fondue was prepared with water and the influence of starch concentration, ethanol content, pH, wine polyphenols, and emulsifying salts on fondue rheology were assessed. Further, alternative thickening agents (xanthan gum and iota-carrageenan) were employed to investigate their behavior in a complex food system like fondue.

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Application of high resolution ultrasonic spectroscopy for monitoring of osmolality and average molar mass in infant milks during lactose hydrolysis

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Digestion of dairy products is accompanied by hydrolysis of disaccharide lactose by enzyme lactase (ß-galactosidase) into monosaccharides glucose and galactose. Deficiency of lactase results in a medical disorder, termed lactose intolerance, which affects approximately 70 % of the global population. Overcoming lactose intolerance is particularly important for infants as their feeding is entirely based on milk. This can be done with application of ß-galactosidase enzyme supplements. However, hydrolysis of lactose increases the osmolality of milks, which causes a range of health problems arising from dehydration effects at high levels of osmolality. Therefore, development of ß-galactosidase supplements requires the capability for real-time monitoring of osmolality in milks, which is a difficult task for analytical techniques especially as lactose hydrolysis is accompanied by synthesis of various galacto-oligosaccharides (GOS) which also contribute to osmolality. In this work, we have utilised the capability of High-Resolution Ultrasonic Spectroscopy [1] to measure non-destructively and in real-time the concentration of ß-galactosidic bonds hydrolysed [2] for monitoring of osmolality in infant milks. The ultrasonically measured time profiles of the concentrations of ß-galactosidic bonds were converted into the time profiles of osmolality of milks and into the average molar mass of milk oligosaccharides. The ultrasonic data were in excellent agreement with the discontinuous data of vapour pressure and freezing point osmometry, and with HPLC profiles. Our results illustrate the capability of High-Resolution Ultrasonic Spectroscopy in non-destructive real-time measurements of osmolarity and of evolution of molar mass of oligosaccharides in complex systems (e.g. milk) during enzymatic hydrolysis of lactose.

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Application of high-resolution ultrasonic spectroscopy for real-time monitoring of enzymatic hydrolysis of globular and non-globular proteins

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Enzymatic hydrolysis of proteins is the most commonly used method for preparation of peptide hydrolysates exhibiting bio-functional properties. Namely, milk proteins serve as accessible and affordable sources of the such peptides [1]. The functional properties of protein hydrolysates depend on the extent to which the proteins are hydrolysed [2]. Control of the enzymatic hydrolysis of proteins requires efficient and non-destructive analytical methods for real-time monitoring the hydrolytic process in native media under processing conditions, which is a difficult task for majority of existing bioanalytical techniques due to the structural complexity of the media, opaqueness, and other factors. Recently we have demonstrated that High-Resolution Ultrasonic Spectroscopy (HR-US) [3] is can be applied for precision real-time measurements of concentration of peptide bonds hydrolysed by enzymes [4] in solutions and complex systems. This poster describes application of HR-US technique for real-time monitoring of hydrolysis of milk proteins, 'rheomorphic' bovine ß-casein, and globular ß-lactoglobulin and BSA, by a mixture of serine proteases under various reaction conditions. The ultrasonic profiles of hydrolysis were compared with the discontinuous data of TNBS (2, 4, 6-trinitrobenzenesulfonic acid) assay. The obtained ultrasonically time dependence of concentration of peptide bonds hydrolysed were used for real-time evaluation of the average degree of polymerization and molar mass of milk protein hydrolysates during the hydrolysis.

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Effect of high-pressure processing on the microstructure and rheological properties of bean flours

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Consumption of dry beans has many health benefits including lowering blood LDL-cholesterol levels. However, processing and cooking techniques may induce structural changes on the bean matrix which can impact the rheological, sensory and physiological properties of beans, including their ability to bind bile salts (BS), and ultimately their potential to reduce cholesterol levels. High-hydrostatic pressure (HHP) has recently demonstrated technological improvement in legume processing. This study aimed to investigate the effect of thermal and non-thermal processing on the microstructure and rheological properties of bean flours and how these changes affect the capacity of beans to retain BS. HHP treatments were evaluated as a function of pressure and time, and compared with convectional cooking at atmospheric pressure. The microstructure of bean matrices was studied by Field-emission scanning electron microscopy. The viscoelastic and flow behavior of bean matrices were studied using small amplitude oscillatory shear measurements and steady shear measurements, respectively. The BS-binding ability of bean flours was determined under physiological conditions using a mixture of primary BS, and individual unbound BS were quantified separately by reversed-phase HPLC. Heated bean matrices, which showed the greatest viscoelastic moduli, almost complete starch gelatinization and major microstructural modifications, retained the lowest amount of BS. On the contrary, HHP processing maintained or increased the BS-binding ability of bean flours, and the effect of pressure level was dependent on the length of treatment. The highest BS-binding ability was observed in flours pressurized at 600MPa, which showed partial starch gelatinization and increased apparent viscosity, and preferentially retained hydrophobic di-hydroxy-BS. This study shows that HHP is a promising minimal-processing technology to transform beans into ingredients with desired rheological properties while maintaining their biological functionality.

Reconstituted Aloe vera hydrogel formation and its applications in high methoxy pectin mix gel formation

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Structural analysis of Aloe vera powder using 1H NMR showed peak signals at 2.0-2.3 ppm, mass of signals at 3.0 - 3.5 ppm and 5.1 ppm confirmed the presence of bioactive acetylated polysaccharides, carbohydrates and glucose, respectively. The effects of different methods of reconstitution such as shaking (S), combined heating-shaking (HS) and heating (H), at various concentrations (0.2 - 1.6 % w/v) of NFAIR powders, on gel strength and stability were studied by rheological analysis. H method was found to be suitable as compared to S and HS methods in terms of higher G' value (24 - 195 Pa), and disappearance of terminal zones. Further, at a fixed concentration of 1.6 % w/v the effect of heating process temperature (30 - 90 °C) and time (15 - 60 min) on viscoelastic behavior was analyzed. At 50 °C for 30 min, the G' and complex modulus G* was well described by Power law ($R2 > 0.95$) and weak gel ($R2 > 0.94$) models. The conjecture for obtaining reconstituted hydrogel by formation of networks like pattern was evidenced by SEM, TEM and HRTEM analysis and further utilized for high methoxy pectin mix gel formation. The interaction effects of Aloe vera/HM pectin mix ratio (0.25 - 1.0), sucrose (0 - 60 % w/w) and pH (3 - 7) on Power law model fitted responses were studied by ANOVA and response surface plots. Aloe vera concentration was found to be more influencing parameter for mix gel formation followed by sucrose content and pH. The numerical optimization technique suggested that reduction of sucrose from 60 to 40 % w/w could be possible by addition of Aloe vera/HM pectin with a mix ratio r varying from 0.40 to 0.59 to obtain similar gel strength and having higher desirability. The obtained results helps to design new process or product developments (viz., mix food gels) by reducing the addition of external gelling agents for wide ranges of targeted applications in food and medicinal sectors with improved mechanical strength.

Wine viscosity: Which compound influences the most the viscosity

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Viscosity is a crucial physical property when studying complex formulations. However rheological studies remain a challenging task as traditional techniques are time and sample consuming and real process operating conditions of shear rate and temperature are not always reachable. Fluidicam provides precise and accurate viscosity measurements as a function of shear rate in a single experiment set-up. Using small sample volumes. Thank to the microfluidic system viscosity measurements of products with at low viscosity values is possible with high precision and accuracy Fluidicam is a microfluidic rheometer that measures the viscosity by flow visualization by a digital camera. A laminar co-flow of a sample and a reference fluid presents a defined interface, its position is related to the viscosity ratio between both fluids and their flow rates. in this work red wines and wine analogs have been analyzed over shear rate ranging from 300 to 100000 1/s, representative of the swallowing conditions in order to identify the impact of each wine element on viscosity. The wines analyzed show a newtonian behavior over the applied shear rates.we studied also the main compounds of wines which is a complex formulation: Water, ethanol (11 to 15 %), glycerol (≈ 12 g⁄L), sugar (≈ 20 g⁄L), polysaccharide (≈ 2 g⁄L), and tannins up to 0.6 mg⁄L. The later are mixed together following the order as mentioned to constitute wine analogs and the viscosity of each blend is measured. Alcohol rises the viscosity of the mix (water+EtOH) by 9 % from 11 to 15 %, then comes glycerol with an impact of 3 %. However, fructose rises the viscosity by 10 % and polysaccharides by 7 %, whereas tannins show no viscosity variations. With these additions we could catch up to 94 % of wine viscosity the small offset left is related to other compounds. Fluidicam is a suitable to compare blends with a very small viscosity variations.

Complex coacervation of food grade cationic surfactant lauric arginate with anionic algal polysaccharide lambda carrageenan

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Lauric Arginate Ester (LAE) is an approved food grade preservative that is cationic surfactant. It is a powerful anti bacterial and anti fungal agent. It is synthesised from three bio friendly components viz. Lauric acid, ethanol and arginine. In this study, we explore the underlying interactions, structure and foaming properties of cationic Lauric Arginate and anionic Lambda carrageenan complex coacervates. Lauric Arginate interacts with carrageenan due to electrostatic interactions forming complexes[1]. It was observed that at low surfactant to carrageenan ratio they form soluble complexes, which are smaller than 50 nm in diameter. As the surfactant to carrageenan ratio was, increased formation of large insoluble complexes occurs leading to eventual phase separation of the system. Interestingly, with further increase in the surfactant concentration the complexes start to redispering in the water phase. This is due to the overcharging of complexes that makes them unstable and leads to dispersion. This is confirmed by particle size and zeta potential measurements. The coacervation was observed to be significantly affected by addition of anionic surfactant Sodium dodecyl sulphate (SDS). LAE-SDS mixed micelles when mixed with carrageenan at different LAE/SDS ratio showed different extent of coacervation. While pure LAE-carrageenan mixtures show strong tendency to coacervation, addition of SDS indicated a sustained reduction in LAEs ability to form coacervates with carrageenan. This effect was due to formation of mixed micelles/vesicles of LAE-SDS which are more negatively charged than pure LAE micelles. At higher concentrations of SDS, the coacervate formation was totally suppressed, which is interesting. This was also confirmed by measuring heat of reactions using isothermal titration calorimetry. Structure of LAE-carrageenan complexes was also determined by Cryo-TEM and SAXS. It was observed that at LAE/Carrageenan ratio above 3 formation of lamellar complexes[2] was observed.

Synergistic gelation mechanism of xanthan gum with galacto- and glucomannan and their interaction with salt

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The heat induced synergistic gelation of different hydrocolloid solutions, xanthan gum types (XG) and galactomannan like guar gum (GG), locust bean gum (LBG) and konjac glucomannan (KGM) is investigated. The physical mechanism of the synergy in viscosity and gelation of blends depends on the monomer structure, the molecular weight, the charge, the polarity, and the chain stiffness of the hydrocolloids. Particularly the properties of the electrically neutral galacto- and glucomannans mixed in combination with XG strongly affect the synergistic impact. These effects are influenced by the number and distribution of mannan side chains and thus their flexibility. As the pure components do not show gelation by their own, they form viscoelastic solutions or even gels when mixed together and heated. In this study, rheological properties of the resulting composite gels of 0.5 % (w/w) were examined under different physicochemical and thermal conditions. The focus was on thermally induced gels, as these gels showed higher synergistic effects compared to the non-heated ones. The gelation mechanism was investigated by strain and temperature dependent oscillatory rheological measurements. Blends with XG-GG (20:80) showed the weakest synergism, followed by XG-LBG blends (20:80), whereas XG-KGM (60:40) blends showed the highest increase of the storage modulus. This can be explained by different local interactions in combinations of the flexibility of the various components. Furthermore, the impact of monovalent salt on the interactions was also investigated. Addition of sodium chloride at 0.05 % and 0.5 % (w/w) concentrations influenced the gelling due to Coulomb screening of the negative charges of the XG. In consequence, the synergism, i.e. storage modulus in particular is strongly affected by variation in salt concentration. We propose specific models based on the gel formation in case of XG-LBG and XG-KGM blends, whereas XG-GG shows an entropic phase separation due to flexibility of GG.

Rapid temperature screening of protein solutions

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Diffusing Wave Spectroscopy (DWS) [1, 2] has already proven to be a useful tool for hydrocolloids analysis. This work will present several examples, how the combination of this technique with a precise temperature control, can give very accurate data on phase transition of hydrocolloids using microstructure analysis for thermal profiling. Diffusing Wave Spectroscopy consists of analysing the interferential images of backscattered light, so called speckle images, which are detected by a CCD camera. These images change in time due to the Brownian motion of the particles that scatter the light. Under heating or cooling, hydrocolloids undergo phase transitions, such as gel formation, gel dissolution or conformational changes, which impacts the particle mobility in the sample. This work presents the technique of DWS, the instrument and several examples on how this new technique can be used for thermal profiling of proteins or other hydrocolloids. During heating of protein solutions, conformational, as well as denaturation of enzymes and proteins can be observed in a rapid and straightforward measurement. Moreover, heterogeneous samples can be studied, as sample volume ranges from some mg to some g. This is especially useful in finished products in the food or cosmetic industry.

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Bambara groundnut protein gels: A rheological and microstructural characterisation

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Nowadays there is a growing interest in exploiting new sources of plant proteins as functional ingredients in food products. Legume seeds in particular are considered as promising protein sources seeing as they accumulate large amounts of proteins during their development. In recent years, Bambara groundnut (*Vigna subterranea* (L.) Verdc.) has been explored as such a potential plant protein source, as a means of value-addition to this indigenous African crop. Included in several of these explorative studies was the determination of the protein concentration at which a solid-like material was formed (the so-called least gelation concentration). Considering the importance of gelation in the formation of distinct textures and consequent sensorial properties characteristic of many foods, it remains an important phenomenon to investigate when establishing novel and/or alternative proteins. To that end we have investigated the rheological and microstructural properties of Bambara groundnut protein gels. The results indicated that vicilin as the major protein fraction present in Bambara groundnut seeds determines the elasticity of the protein isolates. Furthermore, the elasticity dependence on protein concentration has been described using scaling theories from which fractal dimensions of the gel structures could be extracted. The fractal dimensions obtained were in agreement to those previously reported for various protein (whey, bovine-serum albumin, soy) gels prepared in the presence of salt and also provides an indication for the type of gels formed; a deduction further probed through confocal microscopy measurements. This study provides new insights which further cements the potential of Bambara groundnut as a new plant protein source.

How does the composition in fat and interfacial proteins of the droplets influence the structure and texture of high-fat stirred yogurts?

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A set yogurt is an emulsion-filled gel composed of fat droplets dispersed in a protein network and interacting with this latter via the interface. When applied, stirring breaks the gel into a concentrated dispersion of microgels. While emulsion-filled gels are relatively well analyzed in the literature, the dispersions of emulsion-filled microgels obtained after stirring have rarely been studied. By making different creams, this study aimed to understand the impact of the compositions of fat and interfacial proteins on the structural and textural properties of stirred yogurts. First, the effect of the formulation on the thermal properties (fusion, solid fat content) and on the structural properties (fat droplet sizes) of the creams was evaluated. The impact of the different creams obtained on the textural properties (rheology, tribology), then structural properties (microgel sizes, protein network coarseness and pore size) of the resulting stirred yogurt were evaluated next. By using this multi-scale approach, the study clearly demonstrated the existence of a relationship between formulation, structure and macroscopic properties of stirred yogurts. It provided new interpretations that highlight some structuring mechanisms of this complex system, depending on both the fat fraction and the interface. In particular, the study showed that crystallized fat droplets can reinforce the texture whereas liquid droplets tended to weaken it. The level of interactions between the interface and the protein network can be driven by the type of the adsorbed proteins and can modify both the thinness of the protein network and the rheological properties of the stirred yogurt. In addition, small microgels may have been formed when the protein network was thinner, likely because they were less deformable when stirred. As the stirred yogurts were realistically formulated and characterized in conditions taking oral processing into account, this work provide interesting levers for innovations.

Influence of selected non-starch hydrocolloids on methylcellulose gelling process

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This work is focused on the rheological properties of water systems containing ethylcellulose and selected non-starch hydrocolloids like: tara gum, guar gum, konjac and xanthan gum. The aim of work was to study the influence of non-starch hydrocolloids on the gel point Tg of ethylcelullose in water systems with the use of complex rheological techniques. The measurements procedures included SAOS and LAOS (Small- and Large Amplitude Oscillatory Shear). To find gel point values the oscillatory tests were made as a function of changing temperature. For detection of the Tg values two parallel ways were applied. The first one was the analysis of the first derivation of G*(T) dependence, calculated with the help of a central schema. As the second way the analysis of normal forces Fn generated during temperature changes was done. The comparison of these results revealed compatibility between the temperature of the maximum of first derivation of G*(T) and the temperature of the maximum of Fn dependence. Basing on results it can be concluded that the presents of non-starch hydrocolloids influenced the gelling temperature. In comparison with gelling point of ethylcellulose in water, three hydrocolloids used: guar gum, tara gum and konjac gum decreased the value of gelling temperature. In contrast, the presence of xanthan gum in ethylcellulose water system did not influence on the value of Tg. The analysis of linear viscoelasticity region showed, that the increasing of temperature from 20 to 80 ºC caused the widening of frequency range characteristic for parallel course of G' and G''. These conclusions were supported by the results of energy dissipation coefficient analysis as a function of temperature and deformation amplitude applied.

Associative properties of rapeseed napin and pectin: A competition between liquid-liquid and liquid-solid transition

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Using a droplets-based millifluidic device [1], we successfully map, through turbidity measurements, the phase behavior of a plant protein, rapeseed napin (NAP), mixed with a plant polysaccharide, pectin (PEC). The optimum pH for NAP-PEC interactions was found at pH 4, corresponding to the highest electrostatic contribution between the two biopolymers. Additional optical microscopy performed at pH 4 highlighted a solid-to-liquid phase transition overtime [2]. We showed that charge neutralization is a requisite for the transition as no rearrangement was observed when residual charges remain. In addition, this transition was found to be temperature-dependent suggesting that secondary interactions, such as hydrogen bonds, may play a role in this phenomenon. To the best of our knowledge, such solid-to-liquid transition has never been reported for protein-polysaccharide mixtures. We question the role of protein flexibility in this phenomenon as NAP is predicted to be partially disordered. To test this hypothesis, we used lysozyme which is similar to NAP in terms of size, molecular weight and charge density but more rigid. We showed that kinetics of rearrangement were slowed down in the case of LYS. The polysaccharide rigidity (i.e. persistence length) could also influence time and temperature-dependence of the solid-to-liquid transition even though it was not investigated here.

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Changes to the molecular structure of konjac glucomannan during deacetylation-induced gelation

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Konjac Glucomannan (KGM) is a soluble dietary fiber from the konjac plant, widely used in Asia, and having many reported health benefits. One of its characteristic properties is that at high pH, it will be deacetylatyed. This deacetylation leads to gelation already at low concentrations of KGM. Using rheology, light scattering and Atomic Force Microscopy, we here investigate in detail the changes to the molecular structures of KGM that are responsible for this gelation behavior. Rheology shows that for a low weight percent (0.5 %) of KGM, gelation at high pH only occurs quickly above a critical temperature of about 70°C. From Dynamic light scattering (DLS) of dilute KGM as a function of concentration and temperature, we infer that it is the deacetylation reaction rather than KGM aggregation is the rate-determining step in the gelation proces. Finally, using AFM on dilute KGM solutions we show how deacetylation opens up the rather compact structures of the KGM molecules and leads to the formation of very open networks at low concentrations.

Chain conformation, rheological and physicochemical properties of polysaccharide extracted from Tremella fuciformis: A potential flexible coil polyelectrolyte in food material

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Known as "plant hyaluronic acid", the polysaccharide extracted from a widely consumed fungus (Tremella fuciformis, TFP) receives increasing attention in food and cosmetics industry. Chain conformation and physicochemical properties of TFP was investigated by utilizing HPSEC-MALLS-Visc-RI, AFM, micro-electrophoresis and rheology measurements. The conformation parameter as (0.58 ± 0.02), the Mark-Houwink-Kuhn-Sakurada exponent a (0.47 ± 0.01), ah (0.50 ± 0.01) and AFM observation consistently manifested that TFP adopted a flexible coil conformation. This polysaccharide demonstrated a shear-thinning rheological behavior and easily formed gel structure at high concentration. TFP also exhibited as a negative polyelectrolyte in wide pH and ionic strength ranges. Based on these characteristics, TFP and sodium caseinate (NaCS) stable complex were formed by electrostatic bonding in acidic solution (pH 3.0), which can enhance the stability of NaCS for enriched beverage development and establish the bioactive component delivery system.

Controlling viscoelastic and thermal properties of salmon gelatin by variation in the pH during extraction

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High protein byproducts from the salmon industry, such as the skins, can be recovered to produce gelatin. Extraction conditions may have a significant effect on the functional properties of gelatin. In fact, more acidic extractions may generate greater denaturation, resulting in lower molecular weight polymer chains. The objective of this work was to determine the effect extraction pH on the properties of the gelatin produced. The extraction process was carried out at pH 3, 4, or 5 at 60 ºC for 3.5 h. Gelatins were analyzed in terms of molecular weight distribution by electrophoresis (SDS-PAGE), chemical configuration (Raman spectroscopy), thermal properties (DSC), gel strength (compression test), viscoelastic properties (Rheology). Gelatins extracted at pH 5 presented high average molecular weight Mw (~ 173 kDa) whereas samples extracted at pH 3 showed shorter chains (Mw < 65 kDa), suggesting greater hydrolysis toward single chains. The peaks associated with structures of Amide I and III type indicate the helical configuration of all gelatins. The most hydrolyzed gelatins (pH 3) showed greater intensity at 2900 cm-1 which corresponds to the C-H stretching vibration compared to the less hydrolyzed gelatins (pH 5). The gelling temperature was significantly lower for the gelatins produced at pH 3 (~ 1.1 ºC) than at pH 5 (~ 10.3 ºC). These results correlated directly with the mechanical and rheological properties: higher gel strength, higher G' modulus and lower tan d from frequency sweeps for the gelatins produced at pH 5. In conclusion, the polypeptide Mw had greater influence on the mechanical, thermal and rheological behavior of salmon gelatin than the content of hydroxyproline. These results suggest the possibility of modulating the physical properties of gelatin by the use of different pH during the extraction process, which is relevant to obtain an ingredient with defined funcionalities for specific applications.

Visible light induced salmon gelatin based hydrogel with controlled viscoelasticity as a potential edible food coating

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Salmon gelatin (SG) is a versatile biomaterial that can be used as edible coating. Its low viscosity at a wide range of temperatures allows its use on foods surfaces while keeping refrigeration conditions. In order to improve gelatin stabilization, riboflavin, a photosensitive GRAS vitamin, can be used as crosslinking agent through a free radical reaction energetically induced by UV-light. The goal of this work was to produce and characterize SG-riboflavin hydrogels using visible-light as a safer crosslinking strategy. SG was obtained from salmon skin by acid extraction. Suspensions were prepared at 10%w/v and riboflavin was added 0.25 %w/w with respect to dry SG. The pH was adjusted to 8.3 for efficient crosslinking. The suspensions were exposed to blue light (450 nm) for 2min at a distance of 4 cm. Rheological measurements were performed (Discovery HR-2, TA Instruments Rheometer) using a 5 cm diameter flat geometry. For gel strength determinations, hydrogels were incubated at 3 °C for 18 h and measured using a Texture Analyzer (TA-XT Plus, Stable Micro Systems) with a 4 mm diameter (P/4) cylindrical probe. Optical properties were measured with a UV-Visible Spectrophotometer, where transmittance %T was quantified at visible range (400 - 700 nm). Results showed that SG gelation temperature Tgel and gel strength were not modified by crosslinking. Moreover, the final viscosity and elastic modulus after gelation upon cooling was not significantly changed. However, hydrogels showed an irreversible increase in viscosity and elastic modulus at temperatures higher than Tgel (up to 30 ºC). Optical measurements indicated that hydrogels were transparent with a yellowish color which was reduced during crosslinking. %T values of the hydrogel was only 10 % lower that for the SG suspension at 500 - 700 nm. These findings suggest that this hydrogel could be suitable to be used as an edible coating in foods since its mechanical stability and good optical properties. Moreover, all components used are natural and safe.

Effect of native and chuño starches from andean potato addition on rheological properties of pot-set yoghurt

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Yoghurt is a widely-consumed fermented dairy food internationally and also renowned for its health benefits, nutritional value and digestibility. Hydrocolloid stabilizers, are often added to the milk base to maintain or enhance yoghurt properties including texture, viscosity and consistency and to prevent whey separation. In this work, the influence of starch from andean potato cultivars (Solanum species called Rosadita) before and after freezing and drying process of potatoes known as "chuño" on the rheological and pshysical properties of pot-set yoghurt were evaluated. Cow's milk yoghurt was prepared following a standard formulation made with skim milk powder (10 %w/v) without (CY) and with the addition of 2.5 %(w/v) of native potato starch (NY) and "chuño" potato starch (CHY). Fermentation of yoghurt was monitored via pH value. Rheological properties and syneresis rate were determined at 1 and 7 storage days. Our results showed that yoghurt with starch (native and chuño) addition exhibited higher apparent viscosity-shear rate profiles and dynamic viscoelastic moduli and less syneresis than control yogurt without hydrocolloids (CN). On the other hand, viscoelastic moduli and syneresis of CHY were higher than of NY. All the samples studied, showed little variation in their flow, viscoelastic behaviour and syneresis rate with storage time (7 days). This work showed that andean potato starch and chuño area promising alternative to improve physical and rheological properties of yoghurt.

Influence of pH and salt on the rheological properties of dispersions prepared from the galactomannans extracted from Gleditsia triacanthos seeds

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The exploration of new renewable and non-traditional sources of hydrocolloids as alternative to traditional additives has increased in the last years. Gleditsia Triacanthos (Gledi) is a woody species that is widely spread in Argentina and the wide variety of resources offered by this species has not been exploited yet. It is suggested that galactomannans extracted from Gledi with hot water treatment could be a potential additive in food industry. The objective of this work was to study the effect of pH and salt on the rheological properties of dispersions from galactomannans extracted from Gledi seeds. The yellowish-powder obtained from the extraction of Gledi milled seeds with hot-water and ethanolic precipitation was dispersed in water (Gledi-H2O) and in 2 %w/w aqueous solution of NaOH (Gledi-NaOH), HCl (Gledi-HCl), and NaCl (Gledi-NaCl). pH and salt concentration were chosen considering the values typically found in food matrices. The systems structure was evaluated by frequency sweeps, Capillary breakup extensional rheometry and thixotropy oscillation test. The samples showed a macromolecular-solution behavior with viscoelastic-fluid behavior at low frequencies and viscoelastic-solid behavior at high frequencies. The characteristic relaxation time increased as a consequence of lower pH and salt presence. Gledi-H2O dispersion showed the lowest G' and G'' moduli. These results suggested the formation of an entangled polymer network as a consequence of the chemical-environment changes of the galactomannan dispersions. The apparent extensional viscosity and relaxation time of Gledi-HCl and Gledi-NaCl samples were higher than the others dispersions due to an increase of the chains-entanglement density which suggests a larger network structure. On the other hand, Gledi-NaCl sample exhibited the most deformation tendency and the longer time to recover its structure at the measurement conditions. Gledi-NaCl dispersion structure was more sensitive to high shear rate deformation.

Automatising cookie dough production based on industrial scale measurement of dough rheology

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Dough rheology is the critical factor in predicting cookie quality. Everything from the ingredients to the process affect the dough rheology. As the rheology varies recipes and/or process parameters are adjusted manually, which require a lot of knowhow. When that knowhow is lacking it creates large waste volumes in an industrial factory. Automatic regulation of the recipe and process are thus desired. Different techniques have been used to measure the physical behaviour of the dough including rheology, Mixolab analyses, Texture Analyser analyses and Differential Scanning calorimetry analyses. Pre-studies were carried out on dough produced in laboratory scale imitating a batch and a continues process. These have been compared to studies of industrial scale produced doughs in batch and continuous process. The aim is to find correlations to at-line techniques such as image analysis and spectroscopic techniques that can be implemented into the production system.

The structural and mechanical properties of chickpea starch gels

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This work focused on structural and mechanical properties of the chickpea starch gels. The experiment plan involved the extraction of starch from chickpea flour and the use of it to gels preparation. The influence of natural chickpea fiber, fiber obtained enzymatically from starch and saccharose addition on rheological properties was studied. The Large Amplitude Oscillatory Shear (LAOS) was used to study the nonlinear viscoelastic properties of gels. It was demonstrated that all tested systems exhibited viscoelastic properties. Also geometrical decomposition of Lissaojus curves was used to calculate Chebyshev coefficients. The changes of normal force generated during shearing were determined and correlated with viscoelastic properties. Next step involved correlation of viscoelastic properties with textural parameters of gels stored in different moisture conditions. This research was financed by the Ministery of Science and Higher Education of the Republic of Poland in year 2019.

The gelation of chickpea starch: Interactions of polysaccharides' chains in water solutions

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The work was focused on physicochemical properties of chickpea starch extracted form chickpea flour. Molecular mass distribution and amylose to amylopectin ratio were determined. The first step of investigations involved osmometry, dynamic light scattering (DLS) and intrinsic viscosity measurements. The results allowed to determine first (overlap) and second critical concentration of chickpea starch, and swelling and hydrodynamic properties of starch chains in water solutions. The gelation phenomena was studied using DLS and NMR techniques as a function of temperature and concentration. This research was financed by the Ministery of Science and Higher Education of the Republic of Poland in year 2019.

Chickpea flour as a partial and total replacement of wheat flour: Mechanical properties of sweet batters and sponge cakes

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A new approach for producing batter from chickpea flour as gluten-free components and mixed with wheat flour, was explored. This work focused on tests of rheological properties of the batters, planning the experiments in such way as to be able to compare the obtained results with a reference batter using only wheat flour. The experiment plan involved the partial replacement of the wheat flour part with new starch raw materials: chickpea flour. Large Amplitude Oscillatory Shear (LAOS) was used to study the nonlinear viscoelastic properties of raw batters. It was demonstrated that all tested systems exhibited viscoelastic properties, but the area of linear dependency was limited. The systems containing chickpea flour exhibited the intersection of G' and G" (first Fourier harmonic) in the range of deformation from 0.01 to 1.0. Such behaviour is characteristic for systems of strain overshoot type, comprising long polymer chains. The replacement of wheat flour with chikpea flour caused the changes in yield stress values and energy dissipation coefficient. Also geometrical decomposition of Lissaojus curves was used to calculate Chebyshev coefficients. Moreover, the changes of normal force generated during shearing were determined and correlated with viscoelastic properties. The textural properties of sponge cakes were determined and correlated with rheological properties. This mechanical study allowed to point the main factors determining rheological properties of gluten-free batters and showed the future direction of industrial application of chikpea flour.
This research was financed by the Ministery of Science and Higher Education of the Republic of Poland in year 2019.

Cassava (Manihot esculenta C.) starch as a texturing agent in the formulation of yoghurt

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The present study showed that yoghurt formulations with the lowest concentrations of added cassava starch (0.75 and 1 %) did not visibly create aqueous exudates, and the yoghurts had a smooth surface, similar to formulations containing 0.5 % gelatin. Indeed, gelatin, being protein in nature, has a greater tendency to retain water and to confer a smooth nature to yoghurt. In contrast, higher concentrations of added cassava starch (1.5 and 2 %) caused exudations of water on the surface of the yoghurt. Indeed, starch and milk proteins compete to absorb water molecules; thus, an increase in the concentration of starch will tend to increase the absorption of water, and with cooling, the rejection of water is even higher, as a result of a recrystallization of starch. This would explain the aqueous surface of yoghurts with relatively high added starch concentrations. The result is a composite gel (milk protein and starch) with a more rigid structure and a significant exudation of water, which consequently constitutes unfavorable properties for the conservation of cold yoghurt (i.e. water layer on the surface). This rigidity of the composite gel explains the elastic strengths before flow or higher shearing thresholds with yoghurts at higher rates of added starch (14 Pa at 1.5 and 2 %) compared to those of yogurts with low starch added (10 Pa at 0.75% and 1%). However, this structure which seems more rigid at a low stress, weakens more strongly at higher stress due to a composite mixture of casein gel and starch gel rendering it less cohesive and more brittle at a certain threshold of constraint. This explains the lower consistencies of yoghurts with higher starch concentrations (50 and 100 g at 1.5 and 2 %) and higher values for yogurts with low added starch (150 and 200 g at 0.75 and 1 %). In addition, the same trends were observed in viscosity profiles by rheometer measurement. This study shows that owing to the thickening effect of cassava starch in yoghurt a formulation with 1 % added starch seems promising.

Rheological and physical evaluation of rare sugars as alternatives to sucrose in baked goods

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Various alternative sweeteners have been receiving much attention with increasing concerns about health. More recently, scientific and industrial attention has been paid to so-called rare sugars that occurs in very small quantities in nature, due to their health-functional and low caloric properties. However, there are few studies on the processing performance of rare sugars in processed food products. Thus, this study aimed to investigate the effects of sucrose replacement with rare sugars (allulose, tagatose, arabinose, and turanose) on the rheological and physical properties of baked goods, specifically, cookies. The use of rare sugars for sucrose affected the pasting profiles of wheat flour. In addition, the retrogradation patterns of the paste samples with sucrose and rare sugars were studied by oscillatory viscoelastic measurements, demonstrating that their viscoelastic properties were dependent on the types of rare sugars by showing different changes in storage and loss moduli over time. When the rare sugars were incorporated into the formulation of cookies instead of sucrose, the cookies prepared with turanose exhibited the textural and geometrical properties comparable to the sucrose cookies. However, the cookie samples with allulose, tagatose, and arabinose had distinctly low hardness value and spread factor. Also, more bright and dark colors were observed at the arabinose and allulose cookies, respectively. Thus, this study might provide fundamental information on the processing performance of various rare sugars in foods, probably contributing to the extension of rare sugars to a wider variety of food products.

Moisture barrier properties of edible coating and its application on bread

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Moisture content, water activity as well as moisture migration are the crucial factors for food quality and safety. During storage, moisture migration plays an important role in physical properties, sensory properties, microbial stability and shelf life of food products [1]. Application of edible coating is one of the common approaches to control moisture transfer in foods. An edible coating is a thin layer of edible material on the food product surface (between food and environment) or in the product itself, providing a barrier to mass transfer (moisture, oxygen etc.) [2]. Lipids are widely used in edible coatings as moisture barriers, due to their low affinity with water. In this study, a lipid-based edible coating was developed. Its moisture sorption and desorption properties and water vapor permeability were analyzed. The coating was then tested on bread to further investigate its moisture barrier properties. Our results showed that such lipid-based edible coating demonstrated good moisture properties. And the application of the coating on bread could help reduce moisture loss of bread during storage and reduce bread staling.

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Effect of RuBisCo introduction on wheat dough mechanical properties related to protein-protein interactions and protein polymerization

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In the current context of dietary transition, development of plant protein ingredients with high functionality is a major challenge. RuBisCo (ribulose-1,5-bisphosphate carboxylase/oxygenase), a leaf protein, has an interesting nutritional profile and presents promising functional properties. The introduction of RuBisCo in staple food, as wheat-based products, would lead to a protein enrichment and an improvement of their essential amino acid profile, in particular in lysine. This work aims at studying the effect of RuBisCo on wheat dough mechanical properties, protein polymerization dynamics and protein-protein interactions. Doughs with increasing content of RuBisCo (from 0 to 37% of total proteins) were prepared using a 2 g-mixograph at constant hydration with or without thermal treatment. Mechanical properties of enriched doughs were evaluated using Dynamic Mechanical Thermal Analysis in relation with protein polymerization as determined by size exclusion chromatography. Interactions between RuBisCo and gluten proteins were also analysed by sequential extraction. RuBisCo enriched dough properties were compared to those of benchmark plant proteins (pea proteins and gluten). The poster will present protein-protein interactions and their impact on dough mechanical properties.

Investigating gas bubble nucleation during high pressure foam extrusion of gluten-free dough

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A novel high-pressure (HP) micro-foaming extrusion process enables the production of high-end gluten-free (GF) products. This process makes use of a co-rotating twin-screw extruder to mix and subsequently pressurize the GF dough in order to dissolve gaseous carbon dioxide into the dough matrix. The foaming is achieved upon pressure release at the extruder die at the end of the extrusion process. There, the CO2 undersatured system shifts to a supersatured state due to the pressure drop down to atmospheric conditions. This leads to gas bubble nucleation followed by gas bubble expansion and finally, to the foam. Despite the fact that foam extrusion is a well-established process in the polymer industry, it is still poorly understood in food systems such as GF doughs. The aim of this study was to investigate the influence of shear viscosity, temperature, shear rate, pressure drop gradients and die geometry on the gas bubble nucleation in GF dough. The rheological measurements were conducted using a HP shear cell. The effect of the pressure gradient and viscosity on gas bubble nucleation and foaming were determined using a HP foaming vessel. Furthermore, a pilot plant scale extruder equipped with different die geometries was used to track foam formation in elongational flow. Therefore, two model systems were used, namely a simplified GF dough formulation for the extrusion trials, and solutions at different concentrations of hydroxypropyl-methylcellulose (HPMC) and cornstarch for rheological and lab-scale measurements. It was shown that gas bubble nucleation can be influenced by changing dough viscosity, composition, process conditions as well as the extrusion die geometry. Larger die cross-sections lead to earlier gas bubble nucleation along the extrusion process due to reduced resistance to flow. It could be demonstrated that starches act as a nucleating agents during HP foam extrusion of GF dough.

Toasting as a route to alter functional properties of fababean concentrates

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A route towards a more sustainable diet is a (partial) transition of animal protein products by plant-based alternatives. Currently, soy is mostly used due to its excellent rheological and other functional properties towards structuring, but a need exists to diversify plant protein sources used for this transition. An important crop could be fababeans. This crop can be split into a protein-enriched fraction by using a range of technologies. Functional properties of these protein-enriched fractions deviate from those of soy protein fractions. A heat treatment to fababean protein can alter the functional properties. Toasting of fababean protein concentrate powder at 150 °C resulted in higher complex viscosity, higher water holding capacity and less soluble protein, partially matching values of soy protein concentrate. To understand the mechanisms leading to this change in functionality, the material was extensively analyzed, including: changes in the secondary structure of the protein, bonds responsible for increased insolubility, protein-protein interactions, changes at the protein particle surface and overall interaction of protein particles. Possible influences from non-protein particles such as reducing sugars were also considered. The presented results illustrate that toasting of mildly fractionated pulse protein powders can alter the functional properties of the protein fraction, which could enlarge their potential use in novel product concepts. The underlying mechanisms are discussed.

Differences in local surface amorphization of sucrose particles after grinding

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The two different grinding principles are mainly applied in chocolate manufacturing, roller grinding and ball mill grinding. Both result in different processing behaviors of the molten chocolate despite similar particle size distributions. After grinding using a ball mill, yield value and viscosity of the chocolate mass are higher compared to those after roller grinding. Reasons for such differences in flow behavior might be attributed to differences in the interaction between the solid particles, mainly sucrose, and with the surrounding liquid cocoa butter phase in the chocolate mass. Because of differences in the physical state of the sucrose particle surface, e.g. crystalline or amorphous, different interactions occur. Although a partial modification of the initially crystalline surface due to mechanical impact during grinding is well-known, the distribution of different kinds of surface states on the particle surface was unclear. We used the atomic force microscopy (AFM) to measure topography and the local surface state in microscopic scale. The latter one was determined using an AFM technique called local thermal analysis (LTA). This technique enables a direct measurement of local softening temperatures which clearly indicates the surface state, e.g. crystalline or amorphous. The presented results of our study show that different distributions of the local surface states are generated after application of different grinding methods and that these distributions can be correlated to differences in interaction of particles and cocoa butter as well as the resulting flow behavior of the chocolate mass.

Modification of the rheological properties of apple pomace by extrusion processing

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In recent years, the demand for healthy, sustainable, and additive-free food products has significantly grown. Therefore, by-products from fruit and vegetable processing have gained a lot of attention as a novel and promising source of dietary fibers. Especially, the by-products such as apple pomaces are of special interest, as they supply not only dietary fibers, but also various functional properties such as water binding, stabilizing and thickening properties at the same time. However, to achieve this, the structure of the cell-wall polymers must be modified. One promising option is extrusion processing. Our research shows that extrusion of apple pomace at certain processing conditions leads to a dramatic change in their gelling and water binding properties. Addition of water into extruded and milled samples leads to immediate gel formation, which is not observed for the raw materials. This contribution will focus on the influence of processing conditions on the modified cell-wall structure responsible for the improved functionality. In addition to process-structure analysis, the rheological properties of extruded apple pomace will be presented, and the underlying mechanism responsible for the improved functionality will be discussed.

Structuring of very low fat mayonnaise

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With the dawn of greater awareness of nutrition in foods, manufacturers are coming under increasing pressure to reduce the calorific content. Fluid gels; a suspension of gelled particles present a novel solution to this. In previous studies it has been shown that these suspensions give desirable rheological and textural properties for fat-reduced foods. However, with the increased consumer awareness of ingredients, there is a desire for food products to be clean label containing natural, familiar ingredients that are easy to recognise. Egg White proteins present an alternative ingredient for fluid gels. However, proteins are heavily influenced by pH thus an understanding of the influence of pH on properties of fluid gels produced from proteins is required if they are to be used in food formulations. Rheology and tribology were used to investigate the textural properties of these protein fluid gels to determine their suitability as a fat reducer in foods.

Effects of drying and grinding processes in the physical and rheological properties of cactus cladode mucilage (Opuntia ficus-Indica)

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Recently, there is a growing interest in incorporating cactus cladode flour in foods, in order to improve their nutritional value, while cactus cladode mucilage exhibits potential as a thickener. In the present study, cladodes from Opuntia ficus-indica were dried at 45 ^{o}C in a hot air dryer for 24 hours, followed by a 18 hours long drying in a vacuum oven at two different temperatures (45 or 60 ^{o}C). The dried cladodes were then ground with two different methods (hammer mill or hammer mill followed by jet mill). The physical and rheological properties of the four flours were studied. Color parameters were affected both by drying and grinding process. Cladode flour aqueous solutions (5 %w/v) exhibited a non-Newtonian shear thinning behavior, while micronization with jet mill led to lower apparent viscosity values for the tested shear rate range (0.3 - 300 s^{-1}). These findings show that micronization of cladode flour deteriorates its thickening capacity.

Smoothness as a tactile percept: Correlating 'oral' tribology with sensory measurements

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Consumer preference for and acceptance of foods and beverages is influenced by two critical quality parameters: texture and mouthfeel. Sensation and perception of textural properties of foods were classically believed to be governed by their rheological behaviors. It was realized only recently that oral tribology is equally important in influencing oral texture sensations. The objective of this work was to conduct a comprehensive study to investigate the tribological response of oil-in-water (o/w) emulsion systems. We hypothesized that the lubrication behavior of emulsions (constant oil droplet size) with varying oil mass fraction and stabilized by different emulsifiers would influence sensory smoothness perception. Emulsions were prepared with different emulsifiers (whey protein isolate at pH 6.7 or pH 3.5, modified starch and lysozyme) and oil mass fractions (30, 20, 10, 5 and 1 w%). A steel ball-on-polydimethylsiloxane disc tribology assembly (3 point-contact) was used for tribological evaluation. Samples were tested with and without artificial saliva. Oral and finger tactile sensory measurements were also recorded to determine emulsion smoothness, and tribological and sensory measurements were correlated. Linear regression analysis showed good correlation ($R2 = 0.9 - 0.98$) between smoothness scores and friction coefficient data at sliding speeds between 0.1 and 10mm/s, which comprised the mixed and hydrodynamic regimes. Oral tactile measurements showed better correlation with friction coefficients compared to correlations observed between friction coefficients and finger tactile measurements. Smoothness generally did not show significant correlation to other factors like viscosity. These sensory correlations confirm the importance of saliva in in vitro studies of smoothness perception.

Analysis of cocoa content in chocolate using tribo-rheometry and its correlation to mouthfeel

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Natural and processed foods all contain hierarchical structures and phases that range from nanoscale to macroscopic length scales. Each phase contributes to the overall perceived texture and mouthfeel during consumption, which directly correlates with consumer acceptability. As the demand rises for healthier food alternatives, the food industry is pushed to truly understand the role each structure plays in texture and mouthfeel. Currently, sensory panels are used for qualification of new foods, but there are some disadvantages: Costly, well-trained personnel required, time consuming, limited on quantitative analysis, reproducibility difficulties. In this work, we propose a method to characterize cocoa based products using triborheometry.

The effect of hydrogel preload foods with different tribological properties on food intake

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Certain oral processing strategies, such as slow eating rate, high number of chews and hard food textures, have been linked to lowering food intake [1]. However, the effects of lubrication during oral processing on food intake remain unclear. This study aimed to determine the distinctive roles of chewing and oral lubrication on subjective appetite ratings and snack intake. Hydrogels with different textural properties were used as preload after characterization of the instrumental (texture analysis, rheology, tribology) and sensory (descriptive analysis) attributes [2]. Fifty-five participants (26 ± 7 years old, BMI 23 ± 3 kg/m2) participated in a between-subjects design. Participants were asked to consume a standard lunch, followed 3 h later by one of four preloads (three hydrogels or mint tea) and an ad libitum salty snack. Appetite measures were rated on 100 mm visual analogue scales (VAS) before and after preload, and again after snack. In addition, oral processing behavior was evaluated using video recordings (n = 28). Results showed that gel bolus viscosity was correlated to early-stage oral processing attributes, such as firm, elastic and chewy. On the other hand, coefficient of friction of the gel bolus fluid at orally relevant speeds (3 - 50 mm/s) was correlated with later-stage oral processing attributes slippery and salivating and inversely correlated with pasty once the large bolus fragments were filtered out [2]. Based on the satiation study, it was found that oral lubrication rather than chewing resulted in a reduction in snack intake after consuming a hydrogel preload ($p < 0.05$) [3]. In summary, oral tribology is a promising new construct in oral processing and satiety research, also in products not containing fat.

[1] Krop EM et al.: Appetite 125 (253-269) 2018.
[2] Krop EM et al.: Food Hydrocolloids 88 (101-113) 2019.
[3] Krop EM et al.: Food Quality and Preference 74 (118-124) 2019.

Using a three-ball-on-plate configuration for oral processing applications

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Soft contact tribology relating to oral processing is a relatively new research area where soft materials mimic surfaces found in the mouth, like the tongue and palate. In doing so the friction properties of food have been quantitatively measured and linked to sensory perception, which is incredibly useful when developing new food products. Typically, food laboratories do not own specialist tribological testing equipment. It is more common for them to own or use a rheometer for which most commercially available instruments offer an attachment to measure friction. Few studies to date use these attachments; particularly with focus on soft contact tribology relating to oral processing. Therefore the objective of this study was to examine the effect of using a three-ball-on-plate rheometer attachment for soft tribology measurements by assessing the friction properties of a range of model food-like samples. Results were compared to an existing tribological system frequently used in oral processing applications (a mini-traction machine) showing good agreement between the two testing systems. Going forward, the tribology attachment will be invaluable in quantitatively assessing friction with substantial scope in food product design and manufacture.

Lubrication behaviour of beverages: Influence of oil droplets and protein with different morphology

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Tribology has gained interest in the food industry as a tool to study textural attributes. It has been acknowledged that the lubrication properties of foods, in addition to rheological properties, play a significant role in determining texture perception or "mouthfeel". For example, dispersed fat droplets act as a lubricant in the mouth, and contribute to a creamy and thick sensation in fat-containing foods and beverages. A clear relation between specific sensory attributes and lubrication behaviour has not been established, especially for foods or beverages containing multiple components. Nutrition beverages are an example of complex foods, as they contain high concentrations of proteins, fats and carbohydrates. Therefore, we investigated how each component contributed to the lubrication properties of beverages and in future studies, how this affects textural attributes. In the present study, we studied the lubrication properties of model liquid dairy systems, containing whey protein (native or aggregated), casein, and/or emulsified fat. We found that whey protein aggregates (~250 nm) led to higher friction, in the boundary and mixed lubrication regime, than casein micelles with a comparable size (~220 nm). The friction coefficients decreased as the concentration for those proteins increased. Additionally, mixing whey protein and casein micelles led to higher friction. The lubrication behaviour appeared to be influenced by the morphology and hydrophobicity of the protein particles. In the case of emulsions, lubrication was dominated by the type of emulsifiers. The emulsions stabilized with soy lecithin showed lower friction coefficients than emulsions stabilized by whey proteins. When these oil droplets (300 nm) were added to protein dispersions, the friction coefficients decreased strongly. The mechanism determining the lubrication properties of various systems will be discussed.

A combination of rheology and tribology as a novel tool to evaluate texture/mouthfeel of liquid coffee creamers

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Texture/mouthfeel are critical sensory attributes for the development and reformulation of beverage products. This sensory response is directly associated with the physics and physiology of the process of drinking and swallowing of beverages. The process by which one drinks a beverage can be broken down into a series of steps that can be further investigated with specific analytical techniques. Two of such techniques are rheology and tribology. These techniques provide powerful information on the mechanical properties of the product. In rheology the flow properties are measured in order to assess the process of drinking and swallowing. Tribology measures the friction as well as the lubrication of the moving parts activated during the drinking process. This information is useful to study the behavior of the beverage when compressed between the palate and tongue prior to swallowing. Combining both techniques can provide a fingerprint of a given formulation in order to screen and rank products prior to time consuming and subjective sensory evaluations. A series of commercial liquid coffee creamers containing varying fat contents were measured with various rheological and tribological techniques. The instrumental results were used to correlate with sensory evaluations.

Probing the complex interplay between rheology and tribology using hydrogel microparticles

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Hydrogels compose a fascinating class of materials because of their versatility, affordability and responsiveness to external stimuli. Gelatin and agar are examples of hydrogel materials used in foods while polyacrylamide- or hyaluron-based hydrogels have applications in medical sciences and cosmetics. These hydrogels are interesting for their bulk behavior, but can also be made into microparticles or microgels with special properties, creating an even wider range of applications. Hydrogel microparticles have been investigated as potential fat replacers, encapsulating agents and drug delivery systems. For all these purposes, both the flow properties and the lubricating qualities of the microgel suspension need to be carefully controlled. Here we demonstrate the synthesis of spherical hydrogel microparticles via an emulsification method. We then investigate the tunability of gelatin microparticles (GMP) by varying the stiffness and the size of the particles between 10 and 100 microns. The GMP are further evaluated in terms of their rheological and tribological characteristics. Dense suspensions made of GMP are able to generate friction coefficients as low as $\mu = 0.1$ at low velocities in the boundary regime. Upon diluting this suspension to 10% of the initial amount of particles, only a small increase in friction is found. This is surprising as the suspension viscosity is decreased significantly according to Krieger-Dougherty models. It appears that the mere presence of slippery, rolling particles is responsible for the decrease of friction. We furthermore find that the ability of the particles to retain their spherical shape under an applied load plays a larger role in lubrication ability than the viscosity of suspension. Particles with a higher stiffness therefore provide lower friction coefficients. These hydrogel microparticle suspensions are thus a well-suited model system when aiming to understand the complex connection between rheology and tribology of soft granular materials.

Structuring of soy and pea protein using screw extrusion based 3D printing

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3D food printing is an emerging technology fostering the design of foods with tailored textures and nutritional value. Compared to conventional processes, 3D printing comes along with other advantages such as process flexibility and simplification. Selected foods were already successfully used for the printing process, particularly chocolate and pasta. However, the range of natively printable materials is restricted due to specific requirements of the printing process. Especially rheological characteristics - viscosity and flowability as well as yield stress and shape stability - are crucial for the printability. To overcome these material based limitations, a 3D printer was combined with a twin- screw extruder. This system allows continuous printing and specific thermal and mechanical treatment of the substrates to influence their process behavior and material properties. The 3D printer was used with soy and pea protein mixtures. Different substrates with varying protein water ratio were characterized by measurement of viscosity, yield stress, thixotropy and differential scanning calorimetry. Correlation between the material properties and printed structures are made to obtain further information about the printability of plant based protein. The gained information can be used to optimize screw based 3D printing and to create foods with new textures and applications. Particularly continuous printing of plant based proteins represents an innovative approach creating meat-like structures.

3D-printed solid structures from milk and rye

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3D printing of food is gaining more and more attention due to its potential in enabling on-demand production of customised food products. The aim of this work was to investigate the possibility of making healthy snack-type products from milk and rye by 3D printing. Concentrated aqueous pastes for printing were prepared from whole milk powder, wholegrain rye flour and their mixture (1:1). The pastes were characterised by measuring their viscoelastic properties in a rheometer. Printing was performed with a extrusion-type 3D-printer (Foodini, Natural Machines) using a nozzle of 15 mm in diameter at ambient temperature. The printed samples had a grid-like pattern (~62 mm x 38 mm). Only one layer was printed. Right after printing, the samples were solidified by baking at 150 °C for 5 min. The weight and dimensions of the printed samples were measured to get information of the repeatability of the printing process, printing accuracy and shape stability both after printing and baking. The fracturability of the baked samples was studied with a cutting test performed with a Texture Analyser (Stable Micro Systems). The printing trials revealed that solid samples with a crispy texture and moderate hardness could be prepared from both materials and their mixture, although some variability in the dimensions and weight of the printed samples was observed. The variability was greater with the rye flour paste due to the presence of larger bran particles. Baking at 150 °C caused changes in the dimensions of the printed samples, but the grid-like pattern was pretty well preserved in the 1-layer samples after baking. The rye flour samples tended to shrink during baking, whereas milk powder samples expanded and had a glossy appearance. A mixture of both materials showed intermediate glossiness and expansion. This study demonstrated that the structural properties of 3D-printed food products can be tuned by the composition of the printing paste and post-processing.

Influence of ethanol in emulsions stability

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Many food products are emulsion-based systems, some are naturally occurring like milk or cream, whereas some require some form of processing, such as butter or mayonnaise. When other substances are added to an emulsion, for example alcohol during the production of cream liqueurs, this can influence the stability of the emulsion. In the case of dairy products, the proteins present act as emulsifiers contributing to the stability of the emulsion. However, the addition of large volumes of ethanol can modify the native structure of the proteins, affecting their functionality, therefore causing emulsion instability. This limits the amount of ethanol that is possible to add to protein-stabilised emulsions before these become unstable. Although the interaction between ethanol and proteins has been well documented, the interaction between ethanol and other types of emulsifiers has not yet been explored. Therefore, measurements of droplet size, interfacial tension, density and viscosity were taken for oil-in-water(and ethanol) emulsions stabilised with low molecular weight surfactants. Preliminary tests have shown that the presence of ethanol in the continuous phase greatly lowers the interfacial tension between oil and continuous phase, allowing for a smaller droplet size up to 40% of ethanol (in the continuous phase). At this point, it was also possible to observe a slight increase in the viscosity of the emulsions. With the addition of ethanol, the density of the continuous phase also changed becoming very similar to the density of the oil at 47% of ethanol. All this would suggest that O/W emulsions formulated with 40% ethanol in the continuous phase would be more stable than their ethanol-free equivalent.

Foaming and interfacial properties of gelatin from fish skin

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Protein foams are the essential building component of many aerated food structures, such as cakes, breads and ice cream. In food industry, the minimum work required by a foam is to remain aerated during different technological processes. The most abundant sources of gelatin are pig skin, bovine hide and pork and cattle bones, however, recent changing in consumption trends, due to ecological problems, animal welfare, allergies, sanitary and religious restrictions, have led to making a concerted effort into finding alternative protein sources that can provide similar functionalities in food systems. The by-products generated by the fish-processing industry are a potential source for the production of gelatin. The high potential of fish gelatin production is due to the large quantities of collagen by-products generated. In this work, we investigate the foaming and interfacial properties gelatin from skin fish, obtained by differents extraction methods, as an alternative to mammalian species. The capacity of the system to incorporate air (overrun), to hold structure (half-life) as well as the physical properties (contact angle and size) and their effect on the nature of the Air/Water (A/W) interface (interfacial dilatation rheology and surface tension) are explored and discussed.

Microstructural characterisation and mechanics of extruded protein-rich microalgae/starch foams

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The use of marine resources has been identified as key to fulfil the food demand worldwide. However, our understanding of the processability and interactions of sea vegetables with food ingredients is limited. Spirulina microalgae contain a high amount of protein 55 - 60 %wt and their use in many food products such as extruded snacks is currently being explored. In the study of extruded foods an important objective is to be able to relate the mechanical properties to microstructure. In this work we evaluated the impact of adding Spirulina microalgae (1 - 10 %wt) to corn starch extruded foams by means of X-ray scattering techniques, neutron tomography, mechanical compression and three point bending tests. X-ray scattering results show that the extrusion process disrupts the crystalline and lamellar structure of starch, leading to the formation of highly amorphous materials, which are able to re-crystallize to some extent into more heterogeneous structures after prolonged storage. The presence of Spirulina promotes faster re-crystallization kinetics, although only the highest loading of 10 %wt produced a slight increase in the crystallinity of the foam after storage. For the first time we visualised and analysed the 3D microstructure of extruded food foams using neutron tomography. Our results show that addition of Spirulina at levels above 1 %wt reduces foam expansion and increases foam density. Furthermore, foams containing 10 %wt Spirulina show higher compression stress, higher flexure strength and an increase in resistant starch compared to only starch extrudates.

Impact of thermal and mechanical treatments on the structure, rheology and organoleptic properties of macroalgae

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One of the greatest challenges of our society is to ensure access to food for the entire world population, without having a negative impact on the environment. Algae are considered one of the most promising sources for sustainable production of foods. However, there is a lack of research regarding the processability and consumption of algae products. Studies are needed to understand the impact of processing conditions on algae microstructure and rheology, as well as to identify the preferred characteristics of foods containing algae i.e colour, texture, flavour. In this context, the processability of two edible macroalgae species, Laminaria digitata and Saccharina latissima, was investigated. Conventional thermal and mechanical treatments were used to evaluate the impact of these processing conditions on the microstructure and rheological properties of fibre rich-seaweed. It was found that different flow and small amplitude oscillatory shear behaviour could be obtained depending on the order of processing steps, due to the differences in the way the plant material was disrupted. Furthermore, colour and sensory preferences were evaluated to determine which attributes would influence consumption of these food sources.

Applying cheese powders as emulsifiers in mayonnaise

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Cheese powers are multifunctional dairy ingredients normally used due to their flavoring properties. However, depending on differences in cheeses and other ingredients used as raw materials, the content of protein, peptides, and fat in the cheese powders will allow them to also provide functionality i.e. emulsification and water binding properties. In this study, we have focused on investigating the possibility of making mayonnaises with cheese powders as alternative to egg yolk powder. The rheological and physical properties (stability and microstructure) of the resultant products have also been studied. This study provides some fundamental knowledge for developing cheese flavor mayonnaises to the market. In total, four commercial cheese powders were tested. They differ in cheese types used (i.e. -camembert or cream cheese) and with respect to the use of melting salt during manufacture. Egg yolk powder was used as a reference. The mayonnaises were made from 3% cheese powders or 3% egg yolk powder, 65% sunflower oil, with water, salt and vinegar making up the rest. Results show that some of the tested cheese powders are well able to make mayonnaises similar to or even better in consistency than the reference mayonnaise. The products were tested for rheological properties (flow curves, oscillation measurements), the stability was evaluated by multiple light scattering (Turbiscan, Formulaction), particle size by laser diffraction (Mastersizer, Malvern), and microstructure by confocal laser scanning microscopy.

Structure and rheology of emulsions from fine and coarse fractions from air fractionation of dried peas

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The FoodProFuture project aims to develop a knowledge platform for turning raw materials from plants into tasty, healthy and attractive food products full of protein. As part of this project the ability of Norwegian plant-based materials to form and maintain emulsions, the emulsion structures formed, and the gelling behavior of the plant-based materials and their emulsions has been studied. Dried peas (cultivated in Norway) were milled and air-classified. Both the fine protein rich fraction and the coarse starch rich fraction were able to form semi-stable emulsions with corn oil. Destabilization was evident with the separation of a creamed phase over time, whilst the creamed phase itself had good stability. Both the fine protein rich fraction and the coarse starch rich fraction gave rise to complex emulsions with the formation of both oil in water (O/W) droplets and complex structured droplets (probably W/O/W) during a simple one stage emulsification. Both fine protein rich fraction and the coarse starch rich pea fraction and the emulsions formed from these fractions showed heat induced gelation. Coarse starch rich fraction gels were stronger than protein fraction gels, and in both cases the final gel strength of the emulsion gels was lower from that of the original non-emulsified fraction. The effects were much smaller for the protein rich fraction compared to the starch rich fraction, although the emulsified starch gel was still stronger than the non-emulsified protein gel. Emulsification had little effect on the initiation of heat induced gelation for the starch rich fraction but promoted the initial heat induced gelation in the protein rich fraction. The ability of both pea fractions to support complex, gelable, semi-stable emulsions opens up a number of possibilities for the creation of structured food products whilst maximizing utilization of raw materials.

Gelatin-based solid emulsions for oral delivery of bioactives

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Soft chewable gelatin gels potentially offer an excellent delivery unit for a wide range of nutra- or pharmaceuticals. With a familiar and pleasant texture, compliance issues may be minimized. The amphiphilic nature of gelatin allows the formation of gelled emulsions where gelatin forms the continuous gel network while also directly interacting with the oil droplets (active filler effect), advantageous for both texture, stability and organoleptic properties. Such chewable gelled emulsion delivery units have in the past shown the potential for improved bioavailability of e.g. omega-3 oils compared to oral delivery as bulk oil, possibly due to more efficient intestinal lipolysis of smaller pre-formed oil droplets. However, gelatin based emulsions can be susceptible to coalescense in gastric media due to pepsin degrading the stabilizing gelatin. We have examined this issue in detail in vitro and found the severity is dependent on initial droplet size of the emulsion, with larger droplets (a few μm) being most unstable, fully coalescing within 1 - 2 h in the gastric media. We also found that by introducing small amounts of carrageenan oligomers into the gelatin based gelled emulsion the gel texture and disintegration properties were mostly preserved while the emulsion stability in gastric media with pepsin was significantly improved, with even larger droplets being stable for 2+ hours. This effect was attributed to the spontaneous formation at low pH of the gastric media of protective aggregates through complex coacervation of positively charged gelatin and negatively charged carrageenan oligomers containing the emulsion droplets. In addition, the carrageenan oligomers may to some degree interact with the pepsin enzymes, reducing their activity. When the pH is neutralized, such as in the duodenum, the aggregates quickly dissolve releasing the trapped oil droplets for lipolysis.

Combined neutron reflectometry and nonlinear rheology to investigate complex bio-macromolecular films at interfaces

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Since multiphase systems such as emulsions or foams are routinely exposed to large and transient deformations or flow fields, it is of outstanding importance to also consider the high-deformation flow regime. Only the combined rheological and morphological characterization of the nonlinear response of complex interfaces will be able to fully depict the transient and spatial processes leading to the overall material properties and material performance which is essential e.g. for future food design strategies. Morphology and mechanics of 2D structures at interfaces can be investigated by the proposed in-situ combination of neutron reflectometry and nonlinear interfacial rheology. A conceptual design has been developed to allow in-situ nonlinear rheology and neutron reflectometry at the same time/place of interfacial biofilms to unify the measurement of 2D structural as well as mechanical properties of the investigated interfacial films. It is foreseen to implement the device to the neutron reflectometer AMOR at Paul Scherrer Institute after the major upgrade of the instrument in early 2021 (funding assumed)

Stabilization of fluid interfaces by cellulose nanocrystals

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Cellulose nanocrystals (CNC) have attracted attention due to their ability to stabilize fluid interfaces, allowing the formation of bio-compatible foams or emulsions. The mechanisms of CNC adsorption and stabilization are mostly unknown due to the inability to form controlled CNC adsorption layers. We pre-sent our recent advances on the adsorption of CNC at air-water (A/W) interfaces. CNC adsorb at the time-scale of hours and decrease the surface tension due to induced capillary forces, confirming a Pickering stabilization mechanism [1]. The adsorption of CNC may be accelerated by salt-induced charge screening, indicating a limiting effect of particle charges. Neutron reflectometry revealed that CNC form a discontinuous monolayer with crystallites oriented in the interfacial plane. The formation of CNC interfacial layers further allowed to determine their shear and dilatational rheology, paving the way towards improving CNC-based foams and emulsions.

[1] Bertsch P, Diener M, Adamcik J, Scheuble N, Geue T, Mezzenga R, Fischer P: Adsorption and interfacial layer structure of unmodified nanocrystalline cellulose at air/water interfaces, Langmuir 34 (2018) 15195-15202.

Adsorption of cellulose nanocrystals at varying oil interfaces and effect on emulsion properties

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Cellulose nanocrystals (CNC) have recently attracted attention for the stabilization of oil-water (O/W) interfaces, yielding biocompatible Pickering emulsions. Despite great interest and numerous reports on CNC Pickering emulsions, the adsorption and stabilization of O/W interfaces by CNC remains most-ly unknown. We investigated the adsorption and interfacial rheology of CNC at oils with varying polari-ty and alkane chain-length. Whereas CNC adsorb at alkanes such as n-octane, as determined by a reduction in interface tension, CNC adsorption was reduced at more polar chlorooctane and fully ceased at most polar octanol. Oil polarity and alkane chain length further affected the interfacial rhe-ology of adsorbed CNC layers, and ultimately the stability and droplet size in emulsions prepared from different oils. These findings underline the importance of the hydrophobic subphase in the production of O/W Pickering emulsions.

Optimized interaction chamber design for droplet disruption through single-pass emulsification

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Nanoemulsions are optically clear, highly bioavailable and stable emulsions which have extensive applications in the chemical, pharmaceutical and food industry. In high-pressure homogenisation processes, the design of the interaction chamber plays an important role on droplet break-up, and especially the subsequent recoalescence. By adapting the interaction chamber design through OpenFoam CFD simulations, the flow profile within the chamber was optimized to equalize the droplets' residence times. Therefore, the improved design is expected to ensure all droplets experience a more homogenous emulsification; resulting in narrow, mono-modal particle size distributions. This study compares the emulsification performance of this optimized interaction chamber with a standard Y-type Microfluidizer interaction chamber for a range of pressure drops, emulsifier and oil types of different concentrations. For the same pressure drop, it is found that the optimized interaction chamber requires only a single pass in reaching stable, mono-modal particle size distributions, while the Y-type interaction chamber produced a wider particle size distribution, and several passes were needed to reach mono-modality. This study presents an optimized interaction chamber design with a narrower residence time distribution that is beneficial for emulsification and more energy-efficient.

Effect of removing phenolic compounds on interfacial behavior of protein isolated from de-oiled sunflower cake

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Proteins are widely used in food industry for their techno-functional properties: foam, emulsion and hydrogel. Foam is an important aspect in food industry: ice-cream, desserts and confectionary production are based-foam product. Actually, proteins from animal sources are the most employed in food applications. However, due to some ethical, religious and health hindrances, one of the main challenge is to find alternative protein from plants with similar techno-functional properties. Sunflower seed production is one of the highest crop all around the world. High proteinaceous de-oiled sunflower cake is a valuable and cheap by-product. In addition, sunflower seeds naturally contain phenolic compounds with high antioxidant capacity. Interaction between protein and phenolic compounds are known as to have influence on protein functionalities. In this study, we present first the chemical composition of our protein isolated from waste sunflower cake. Then we investigated on their foaming and interfacial properties. We aimed to see the impact of removing the natural phenolic compounds on the interfacial rheology of the isolated protein.

Comparative study of physico-chemical and functional properties of native and pea proteins isolate

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Functional properties of native and pea proteins isolate were investigated. Native proteins were prepared from yellow pea flour using cool water extractions followed by filtration at pH 7. Whereas protein isolates were obtained from yellow pea flour using cool water extractions followed by acid and heating treatment. Following assessment of physico-chemical, solubility and rheological properties of both proteins were tested and compared. The particle size of the native proteins is smaller than the particle size of the isolate proteins and the native proteins develop a higher solubility than the isolate proteins. However, the gel properties of pea proteins isolate was superior at all tested concentrations. Adjustment of process technology could provide new opportunities to extend the range of functional properties of pea proteins in different food systems.

Influence of thermomechanical treatment on the rheological properties of soy proteins in extrusion processing

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Consumers demand for protein-rich vegan food products is steadily increasing. Extrusion processing can be used to produce these by mixing plant-based proteins with water, plasticizing the mixture, and structuring of it by pushing it through a die at the end of the extruder. Depending on the dimensions and type of the die, products with different structures can be achieved, such as meat analogues or vegan spreads. Thermomechanical stresses, caused by the barrel heating and screw rotation in the screw section, influence the proteins' molecular structure and can therefore lead to changes of the rheological properties. As the structure formation in the die is a strong function of the rheological properties, the influence of thermomechanical treatment on the rheological properties must be known. In this study, a closed cavity rheometer (CCR) is used to investigate the changes of rheological properties in the extrusion process. This device allows to treat the material thermomechanically at defined, extrusion-like conditions and subsequently analyze the rheological properties. In contrast to the extrusion process, where thermal and mechanical stresses are coupled, the stresses can be varied and investigated independently. This contribution will focus on the influence of temperature, shear and treatment time on the rheological properties of soy proteins at medium moisture contents (~20-40 % water content). To get information on the viscous as well as the elastic properties of the system, results of frequency sweeps and relaxation tests will be presented. Furthermore, the influence of the rheological properties on the structure formation will be discussed.

Rheological aspects in fabricating electrospun pullulan fibers incorporating cyclodextrins

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Electrospinning is a simple and versatile technology to produce fibers with large surface area-to-volume ratio for active-packaging, biomedical and pharmaceutical applications. Pullulan is a biodegradable water-soluble extracellular polysaccharide with capacity to form resilient fibers. Cyclodextrins (CDs) have complexation properties and could enhance the protection, apparent aqueous solubility and controlled release of poor water-soluble molecules. Blending pullulan with truncated CDs to facilitate pullulan nanofiber formation offers a powerful platform to exploit their inherent benefits. However, an essential prerequisite for successful nanofiber formation from biopolymers is to achieve a molecular entanglement during electrospinning. In this study, we evaluated the feasibility of producing electrospun fibers from pullulan and CD blends. Rheological characterization of pullulan/CD binary mixtures was performed before electrospinning, and the morphology of the produced spunfibers was studied by SEM. In order to identify the optimal polysaccharide/CD concentration for an efficient production of bead-free uniform nanofibers, the entanglement concentration (Ce), i.e. concentration at which polymer chains start to entangle with one another, was determined. Pullulan dispersions of concentrations = 8% (w/v) with or without HP-ß-CD showed Newtonian behavior, whereas at concentrations 8% shear thinning behavior was noticeable. Spunfiber morphology changed from beaded to uniform structure with increasing viscosity of pullulan solutions. The formation of well-formed spunfibers required a pullulan concentration 2.6 times the Ce equivalent to ~20%pullulan. The incorporation of 10 and 30% of CD did not significantly affect the Ce of 20% pullulan solutions, and electrospinning of these mixtures produced uniform and bead-less fibers with average diameters of 1-2 µm. This study is the first step towards the use of pullulan/CD spunfibers as encapsulation matrices of unstable compounds for demanding applications.

Rheological changes of starch-based purees under oral conditions

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Saliva plays an important role in the oral breakdown of starch-containing food, as the enzymatic effect of a-amylase, contributes to food matrix breakage. Depending on this breakage at the oral level, differences at mouthfeel and taste perception arisen. The objective of this work was twofold: i) to study the structure breakdown of equally viscous starch-containing systems with different hydrocolloids under oral conditions and ii) to study the effect of different breakage in the mouthfeel and taste perception. Carrot puree samples were prepared with three different hydrocolloids (xanthan, carboxymethyl cellulose and carrageenan) in addition to corn starch. In order to achieve initial equal sample viscosity and to adjust hydrocolloids concentration, viscosity of samples was measured in a viscometer (HAAKE Viscotester). Then in-vitro breakage was measured by using a controlled stress rheometer (AR-G2, TA Instruments) with a starch pasting cell (20 g of samples + 2 ml of artificial saliva). Sensory testing was performed using the Flash Profile technique. Regardless the same initial viscosity, viscosity decay with artificial saliva, depended on the hydrocolloids used, being the puree with xantham gum the one with less viscosity decay followed by carrageenan and carboxymethyl cellulose. The flash profile revealed that compact, creamy and thick textural attributes correlated with high apparent viscosity and low viscosity decay rate. In general, results showed that using different thickeners allows to elaborate purees with similar viscosity but different structural decay and eliciting different sensations in the mouth.

Temperature and aging time dependence of nanostructure in cheese

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Taste and food texture of dairy product relate to the manufacturing process, such as temperature, pH, aging, and variety of coagulant. One of the possible origins can be attributed to the difference of the 'nano structure'. On the nano structure of dairy products, there is some study about single structure as to only fat and protein which is separated from milk or related products. On the other hand, few study about structures in the 'real' foods without any pretreated. Therefore we study Nano structure of dairy products intact by small angle X-ray scattering (SAXS) especially with attention to colloidal phosphate calcium (CCP) and casein protein. We explain how the difference of the Nano Structure concerned with that of the manufacturing process, temperature and aging. The samples of aging trails were prepared from sterilized milk, curd, and aged cheese (1 day, 1, 2 weeks, and 1, 2 months). The samples for temperatures trails are sterilized milk and Mozzarella cheese. From the SAXS profiles of cheeses aged with different days, there is a shoulder at the Q = 0.8 nm^{-1}, sterilized milk, which comes from CCP. Intensity of the shoulder on curd increases, but that of aging cheeses disappear. Instead, at the Q = 0.3 nm^{-1}, shoulders appear and intensity increases with increasing aging time. From the SAXS profiles of milk sterilized at 60 ºC and Mozzarella with different temperature both of intensity at 0.3 nm^{-1} decrease. Over 70 ºC, another shoulder appears at 0.4 nm^{-1}, which is 2.5 times bigger than shoulder of CCP. On the other hand, milk sterilized at 120 ºC, no appear at 0.4 nm^{-1} up to the temperature. We consider that difference of this are related with solidification of milk.

Oil structuring as a way to control gel functionality and digestibility

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Fats are an important ingredient in many food products, contributing to food sensory and textural properties, providing energy and needed for metabolic pathways. High intake of saturated and trans unsaturated fats was found to be related to negative health effects leading to search for healthier fat replacements. The multi-functionality of fat in food products raise a great challenge for the development of fat replacement or reduction. Oil structuring has been proposed as a promising strategy for fat replacement due to their solid texture and high unsaturation content. The current research aim to explore the relation between gelation mechanism, oleogel texture and oleogel digestibility. Three different oil structuring agents were examined with canola oil; ethyl cellulose (EC), E471, and ß-sitosterol/g-oryzanol mixture. Mechanical analysis exhibited gel hardness in the order of E471 < EC < ß-sitosterol/g-oryzanol mixture. While simulated pH-stat lipolysis on the same samples demonstrated a significantly different lipolysis scheme for the different gels with total lipolysis percentage in the order of EC < ß-sitosterol/g-oryzanol mixture < E471 suggesting an indirect relation between mechanical properties and oleogel digestibility. The ability to control the oleogel texture and lipolysis was also examined using combination of E471 and EC. Harder gels were obtained in the E471:EC samples in comparison to simple addition of each component contribution suggesting a synergistic interaction between these two components. While combination of E471 and EC allude to new lipolysis profile with intermediate FFA release, up to 50%, in comparison to each structuring agent lipolysis. Moreover, combination of this two component improved the oleogel thixotropic behavior. Overall, this study demonstrated the ability to design new functional food materials with desired textural attributes and controllable lipid digestion based on the oleogelation mechanism used.

Molecular understanding of TAG derivatives organization in oleogels

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Oleogelation is a process by which a heated mixture of a structuring agent and liquid oil forms a solid-like gel during cooling. The gel formation is based on the self-assembly of the structuring agents within the oil phase induced by weak physical forces such as hydrogen bonds and van der Waals interactions. Crystallization and crystal network formation is one of the most common organization forms of oleogels. Tri-acyl glycerides (TAG) and there derivatives, free fatty acids (FFA) and mono-acyl glyceride (MAG), have the ability to crystallize and form crystal network in liquid oil; they all share a similar carbon chain structure, but differ by the polar head group. Thus, different arrangements are formed in the gel due to the different architecture of each molecule leading to gel network with different mechanical and textural properties. The current research aim to investigate the source for the different characteristics exhibited by oleogels based on different TAG derivatives. More specifically we aim to study the structure-function relation of these oleogels from the molecular level through the microscopic level all the way to the bulk material properties. The role of different chemical groups in the molecular organization of each TAG derivative was examined using FTIR spectroscopy, SEM imaging, and XRD analysis. The higher level crystal organization was studied using polarized microscopy equipped with a temperature control stage. These techniques provided an important understanding of the building blocks governing the formation of the crystal network. Finally, the gel rheological properties were evaluated using various experimental set-ups in order to relate the macroscopic functional behavior to the nano-and microstructural architecture. By understanding the nature of the differences between each molecular organization, we can better understand the structure-property relationship, and harness this understanding to better design and tailor these materials and their function in healthier nutrition.

Wear and viscoelastic behaviors of cheese under different conditions

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Wear or mass loss of foods is an important factor in food processing and breakdown. However, determining wear behaviors of cheese is challenging as they are viscoelastic, soft solid materials and usually undergo deformation when subjected to force. Thus, understanding viscoelastic behavior is an inseparable part of wear studies of cheese because wear behavior is related to rheological properties. The objective of this study was to determine the impact of testing parameters on rheological and wear behaviors of full-fat (50, 52, and 54% dw fat) and reduced fat (28% dw fat) hard cheeses. Wear measurements were performed at different normal forces (0.5 and 0.7 N), sliding speeds (30 and 50 mm/s), and temperatures (5, 15 and 25°C) using a steel twin-ball-on-disc sliding tribo-system. Penetration depth as a function of sliding distance was recorded; penetration volume was calculated based on the surface area of the wear scar. Large amplitude oscillatory shear (LAOS) and strain sweep measurements were also performed. Penetration volume increased at higher temperature, normal force, and sliding speed. Cheeses containing 54, 52 and 50 % (dw) fat had significantly ($p < 0.05$) higher penetration volume at higher temperature, normal force, and sliding speed compared to reduced-fat cheeses. Also, there were a significant difference ($p < 0.05$) among penetration volume of full-fat cheeses at higher temperature. Lower rigidity at higher temperatures led to greater penetration depth in all cheeses. Lissajous curves showed that all cheeses had greater deformation at higher strains. These results implied that differences in penetration volume in full-fat and reduced-fat cheeses were due to differences in rigidity. The findings of this study provided a fundamental understanding of relationships among rheological properties and wear behavior, which may be used to determine food processing ability such as shreddability and sliceability.

Characterizing wear behaviors of acid-induced casein gels by a three-region kernel-based model

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Hydrogels are soft materials used as key components in many industries, including food and pharmaceuticals. Characterizing hydrogel wear behaviors is important as wear properties are correlated to hydrogel durability, firmness, wear rate, and yield strength. However, studies on hydrogel wear behavior remain empirical and there are no available fundamental wear models that incorporate hydrogel physical properties. In this study, we took the first steps towards a fundamental wear model by developing a kernel-based statistical model to characterize casein gel wear behaviors (3 - 5 % w/w concentration, 200 mM NaCl). Casein hydrogels were evaluated under several normal forces (0.1 - 0.3 N) at 20°C. A kernel-based model was used to break down the deformation-wear process into three successive regions: 1) a deformation-dominant region, 2) a constant wear rate region, and 3) a catastrophic failure region, the length of which was based on the rate of wear. This kernel model was based on the assumptions that the deformation and wear rates changed in the deformation-dominant region, but deformation was negligible and wear rate was constant in the constant wear rate region. The length of the deformation-dominant region and constant wear rate region was defined as durability and the average wear rate in the constant wear rate region was defined as wear rate. Higher normal force and lower casein concentration resulted in higher constant wear rate (up to 0.0308 mm/contact), while durability, measured by the combined length of the deformation-dominant and constant wear regions, was shorter. In the constant wear rate region, wear was the main driver behind penetration depth increase; the contribution of deformation was negligible. In the failure region, the hydrogels underwent catastrophic surface failure, which caused a sharp increase in penetration depth. The developed kernel model can be used to evaluate the functional properties of soft materials, as well as design hydrogels with specific wear behaviors.

Study of crystallization kinetics on binary mixture of coconut oil and cocoa butter along with development of phase diagram

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Lauric fats are used as a cocoa butter substitutes in many chocolate or chocolate based confectionery applications. Therefore, the study of the Lauric fats mixed with Chocolate is widely explored, in accordance with the formulation and processing. Yet, there are challenges and limitations faced in production of lauric fat-based chocolate products. Hence, interaction between cocoa butter (CB, i.e. chocolate) and coconut oil (CO, i.e. lauric fat) would help to address such problems occurring while production process. This study emphasized on the effect of static crystallization vs. the one with applied shear on CB-CO blends. The blends were prepared in the ratio from 100 to 0 wt.% in the interval of 10 wt.%. Nuclear Magnetic Resonance (NMR) was used for investigation of solid fat content (SFC) of blends for 24 h at 22 °C. SFC profile highlighted on the static crystallization kinetics of CB-CO blends, resulting into decrease in Avrami constant (k) after addition of higher amount of CO. In addition, Polarized Light Microscopy (PLM) showed the morphological changes from spherulitic to needle like in the order from CB to CO. Furthermore, rheology analysis depicted the effect of shear delayed the crystal growth in CB-CO as compared to static crystallization. Moreover, the fractal dimension (FD) was calculated by fitting Kraus model to amplitude sweep profile in order to study the crystal network of blends. FD value was decreasing as the amount of CO was increasing. The phase diagram for binary mixture was developed by using Differential Scanning Calorimetry (DSC) technique. This phase diagram showed the occurrence of different phases of CO and CB in their respective mixtures. The phase diagram was used to determine the exact melting and solidifying temperature of different mixtures of CB and CO. Altogether we can show that crystallization kinetics and phase diagram confirmed the inhibition in crystallization of CB after addition of CO and forming eutectic point at 65 wt.% of CO and 35 wt.% of CB.

Quantifying the effect of NaCl on eye formation in experimental Swiss-type cheese: Rheology and computer tomography combined

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The influences of three brining times (0, 1, and 3 d) and two propionibacteria (PAB) strains (Prop A and Prop B) on eye formation in Swiss-type cheeses were investigated by uniaxial compression and computer tomography (CT). Force (at breaking point or at 33% deformation), eye numbers, eye volumes and eye-less border zones were compared. For the evaluation of the CT images, the newly developed software VG Studio Max 3.2 was applied. Prop A was highly active at low NaCl levels as it used both L- and D-lactate, but was strongly inhibited with increasing NaCl content and therefore formed less carbon dioxide. As a result, eye volume decreased considerably, as well as eye diameter especially towards the cheese surface. Prop B exclusively converted L-lactate, but was much less affected by increasing NaCl content, hence, eye formation changed less. The brining time also influenced the visco-elastic properties of the experimental cheeses. As expected, the increase in brining time significantly increased the firmness and thus the deformation resistance of the cheeses ($P < 0.001$). Moreover, dry ripening contributed to a firmer texture in the border zone through the loss of moisture and thereby affected eye formation. The eye-less border zone was mainly affected by three factors: CO2 diffusion to the outside, CO2 amount from propionic acid fermentation, and body firmness. The study clearly showed that CT combined with the new image analysis software VG Studio Max 3.2 is a potent method for analyzing the eye formation in cheese. The salt-dependent visco-elastic properties of the cheese matrix and the salt sensitivity of the PAB strains both influenced the number and the spatial distribution of the eyes.

Simultaneous microstructural and rheological study of olive oil-based organogels as fat phase for shortenings and emulsions

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The urgency of replacing unhealthy saturated and trans-unsaturated fats led to the development of alternative strategies in fat structuring. Edible vegetable oils, worldwide recognized as healthy fats, can be structured through a non-invasive physical process called organogelation, which has gained much interest in recent years. This is allowed by a relatively small amount of amphiphilic compounds, i.e. organogelators, able to self-assemble within the organic solvent via non-covalent interactions (mainly Van der Waals forces, H-bonds) increasing the texture of the fat. In food industry, such substances must fulfill strict law requirements. A combined simultaneous rheological and spectroscopic study of olive oil gelled by some edible substances (mostly natural) was carried out with a rotational rheometer equipped with a spectroscopy module able to generate an IR ray conveyed to the lower plate where the sample was placed; this allows a simultaneous study of rheological and microstructural properties of the materials. In this way, the spectroscopic investigation was able to highlight the kinetics of formation of weak interactions responsible of the peculiar rheological properties of the new fats. This was done with the aim of finding a link between mechanical macroscopic properties and structural microscopic properties, and, possibly, to outline a predictable behaviour of such systems in order to improve the efficiency in food engineering processes adopted for producing new healthy fats.

Differentiating between the effects of chymosin-mediated proteolysis, coagulant type, ripening temperature and calcium solubilisation on fracture behaviour of Maasdam-style cheese

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Ripening-related changes within the cheese matrix influence cheese properties, such as texture perception and development of undesirable slits and cracks. However, the individual contribution of various factors on cheese properties remains unclear; therefore, the aim of this study was to decouple and explore the individual roles of chymosin-mediated proteolysis, coagulant-type, ripening temperature and calcium solubilisation on the fracture behaviour of Maasdam-style cheese. Addition of a chymosin inhibitor, i.e., pepstatin A, to the curd/whey mixture during cheese manufacture completely inhibited the breakdown of a_{s1}-casein over 90 d of ripening, while substitution of fermentation-produced bovine chymosin with fermentation-produced camel chymosin decreased breakdown of a_{s1}-casein by ~40%. However, neither treatment influenced the hydrolysis of ß-casein or the solubilisation of colloidal calcium. Ripening of cheese at a consistent low temperature (8 °C) decreased the rate of breakdown of a_{s1}-casein and ß-casein and solubilisation of colloidal calcium as compared to cheeses ripened for a period at higher temperature (warm-room stage at 23 °C between 20 and 48 d ripening). A significant positive correlation was found between intact a_{s1}-casein content and fracture stress, suggesting that the hydrolysis of a_{s1}-casein has an important role in the softening of cheese texture. However, no significant association between levels of intact a_{s1}-casein and strain at fracture was observed, suggesting that breakdown of a_{s1}-casein had no influence on the brittleness of Maasdam-style cheese. In contrast, the strain at fracture was significantly positively correlated with the level of intact ß-casein and insoluble calcium content. Results from this study provide a new perspective on potential opportunities for cheese-makers to reduce the incidence of slits and cracks in cheese.

Viscosity measurement of homemade coconut and palm kernel oils produced in Côte d'Ivoire

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Coconut and palm kernel oils are traditionally used in sub-Saharan Africa for dietary, medical and cosmetic purposes. In Côte d'Ivoire, their production is concentrated in the southern part of the country and mainly done at small scale or household level. Sale conditions of these oils which consist of exposition to the sun can affect their physical properties such as viscosity. The aim of the present study is to determine viscosities of coconut and palm kernels oils produced in Côte d'Ivoire, by homemade processing. For this purpose, samples of both oils were bought from producers and retailers around Grand-Bassam (20 km from Abidjan, South Côte d'Ivoire). For coconut oil, samples from two different process: wet and dry treatments, were collected. Viscosity was measured with a viscometer (DV1 Viscosimeter, Brookfield). Results showed that there is no difference between viscosities of coconut oils from wet and dry methods. Values are 39.5 and 40.4 mPa.s respectively. The viscosity of palm kernel oil (56.6 mPa.s) is 4 times higher than that of commercially refined brand.

Soft colloids as rheology modifiers: Aqueous versus sugar systems

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The flow properties (e.g. viscosity, yield stress, viscoelasticity) of a given formulation can be manipulated with the addition of colloidal particles since these largely depend on the volume fraction of particles in the system. Hard-sphere colloids, though rarely composed of food-grade materials, have been intensively studied for such purposes. The development of novel colloidal particles for use as composite ingredients in foods is a relatively recent area of research. Food-grade colloidal particles are most often composed of biopolymer assemblies due to the requirements for biocompatibility and biodegradability. This implies that the resulting particles are likely to be "soft", not only in terms of their mechanical properties but also in their inter-particle interactions. Considering the technological challenges experienced when using native biopolymers for rheology modification in foods (e.g. poor solubility, multiple interactions with other food ingredients, time-dependent flow phenomena), the use of well-defined colloidal particles might permit manufacturers to overcome these issues. Such colloidal particles might have other uses for example in whitening, macronutrient replacement in reduced calorie foods, modulation of food structure during digestion etc. However, knowledge of the influence of colloidal particles on the bulk rheological properties is required before these applications can be realised industrially. Colloidal particles composed of a synthetic polymer, namely poly(N-isopropylacrylamide) (p(NIPAM)) microgels, were chosen as a model "soft colloid" since the material properties of these microgel particles in terms of their size distribution, surface charge density and crosslinking density can be tailored with variations in the synthesis conditions. The influence of p(NIPAM) microgel volume fraction and crosslinking density on the rheological properties of water and glucose solutions is reported here. Future work will compare this behaviour with natural, biopolymer-based microgel particles.

Development and characterization of foam structured hydroxypropyl methylcellulose oleogel as an animal fat replacer for saturated fat-reduced meat products

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Solid fats provide desirable structural, functional and sensory attributes to a variety of food products. However, the health-related concerns associated with the presence of saturated and trans-fat have led to the development of oil structuring technology. Oleogelation can be considerred as an emerging technique to transform liquid vegetable oil into a solid-like gel with three dimensional network without changing its unsaturated fat-rich composition. More recently, hydrocolloid-based oleogels have received much attention because most of hydrocolloids are naturally abundant, approved for use in foods, and well-characterized. Thus, the purpose of this study was to investigate the physicochemical properties of foam-structured oleogel with HPMC, and to evaluate the feasibility of HPMC oleogels as a replacement for animal fat (beef tallow) to reduce the level of saturated fat in meat patties. The foam-structured HPMC was prepared by homogenization and freeze-drying of HPMC solution and successfully used to convert canola oil into solid form. The textural properties (firmness and work of shear) of HPMC oleogels were higher than those of beef tallow and the HPMC oleogels exhibited frequency-independent visoelastic properties as a weak elastic gels. The HPMC oleogels also exhibited temperature-independent solid fat contents and they had greater resistance against oxidation than the canola oil. When beef tallow was substituted with HPMC oleogels in meat patties, the cooking loss and firmness were lowered. In addition, the replacement of beef tallow with HPMC oleogels was significantly reduced from 42% to 15% in the levels of saturated fatty acids in the meat patties. Thus, this study can encourage the food industry to extend the application of HPMC oleogels to various food products.

Yield stress dependent foaming of partially crystalline edible oils

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Oils and fats are highly complex mixtures of triglycerides and show a transition from Newtonian to non-Newtonian flow upon crystallization. Due to the highly dynamic nature of oil and fat crystallization, it is almost impossible to measure their rheological properties as function of crystal concentration and crystal morphology. Consequently, in-line measurement techniques of continuous crystallization processes are required to overcome this problem. This study shows how to accurately measure the rheological properties of oils and fats in-line. PKO yield stress t_0 as function of F_{SFC} was fitted with the model proposed by Marangoni and Rogers [1] by varying the primary particle size a while keeping the fractal dimension and the interfacial tension constant. With increasing shear rate during crystallization the fitted primary particle size a decreased from 848 to 28 nm. PLM images revealed that spherulic aggregates of single needle like crystals are deagglomerated with increasing shear rates during crystallization. This deagglomeration process influences the slope of the t_0 vs F_{SFC} curves as well as the critical crystal concentration F_{ccc} which defines the onset of t_0. The transformation from spherulic aggregates to single needle like crystals increases the aspect ratio of the suspended crystals thereby lowering the F_{ccc} from 0.035 to 0.015, which corresponds to aspect ratios of 18 and 30 according to Saar et al. [2].

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Oil structuration: A strategy to produce filling creams for sandwich cookies with reduced saturated fatty acids

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Convenience in consume and portability has made cookies a popular ¨on-the-go¨ snack among consumers, mainly between children and adolescents. In particular, sandwich cookies consisting of two cookies between which is a filling are highly consumed due to their texture and flavor attributes. However, soft filling creams (FC) used for filling sandwich cookies are often produced using a high proportion of highly saturated hard stock fat, which are unhealthy for consumers. The aim of this work was to study the formulation and characterization of FC using monoglycerides (MG) oleogels in order to obtain a product with low saturated fatty acids content and functionality similar to FC from commercial cookies (FCC, 22 %wt fat content). Oleogels were prepared using 10 %wt MG and high-oleic sunflower oil. FC were elaborated using variable amounts of oleogel (22 - 40 %wt) and sugar, free-fat powdered milk, and vanilla essence. Different techniques including rheology, textural analysis, oil binding capacity (OBC), and oil loss in sandwich cookies were used for characterization. FC formulated with 22 %wt oleogel showed viscoelastic moduli values that did not differ significantly from those measured in FFC. The OBC of FC decreased with the increase of oleogel content. Values ranged between 99.9 and 91.8 %. Texture analysis showed that hardness of FC decreased with the rise of oleogel in the formulation. Hardness values ranged between 0.66 and 3.5 N resulting lower than the one for FCC (8.7 N). When FC and FCC were used for assembling cookies into sandwiches, only FC formulated with 22 %wt oleogel was not effective to achieve the adhesion between parts. The lowest oil loss in sandwich cookies after 21 days was found for FC containing 26 %wt oleogel (about 0.9 g/100 gFC), although a little higher than the corresponding to FCC (0.3 g/100 gFC). The nutritional improvement due to the use of oleogel in FC versus FCC was 3.6 versus 10.8 %wt of saturated fatty acids and free versus 1 %wt of trans fatty acids.

Production of nanostructured lipids carriers using monoglycerides oleogels

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Vegetable oils structured by the action of organogelators are suitable systems for the encapsulation and delivery of hydrophobic bioactive molecules, as well as for controlled release purposes into the bloodstream. These systems can be utilized for oral administration formulations by dispersing them in an aqueous phase. Thus, emulsions of gelled-oil nanoparticles are proposed as new delivery carriers of lipophilic bioactive compound with sustained release. The aim of this work was to develop a synthesis process of Nanostructured Lipids Carriers (NLCs) using monoglycerides oleogels as lipid phase and curcuminoids as bioactive compound. To this purpose, molten oleogels were prepared using 10% wt of monoglycerides and olive oil, adding the curcuminoids maximum concentration that allows its solubilization. Emulsions were produced using high-speed homogenization followed by ultrasonication. The effect of surfactant type (Pluronic F-68 (P68), Tween 80 (Tw80), or 50:50 P68:Tw80) in the aqueous phase, ratio lipid phase/aqueous phase (5/95, 10/90, or 15/85), and the water bath temperature during sonication (85, 55, 25, or 1°C) was evaluated on particle size, polydispersity index (PDI), and stability during storage. The lowest particle size and PDI were obtained by applied sonication at 85°C, followed by fast cooling at 1°C. Furthermore, it was found that emulsions prepared using a mixture of 50:50 P68:Tw80 in the aqueous phase resulted more stables on time than those containing pure surfactants. Under the optimal conditions, the average value of particle size and PDI in the period of 21 days was 155 nm and 0.198, 213 nm and 0.292, and 255 nm and 0.296 for ratios lipid phase/aqueous phase of 5/95, 10/90, and 15/85 respectively. The drug loading capacity of emulsions was about 0.42%. Based on these positive results a more detail study can be carried out in order to include other relevant aspects of NLCs production.

Development of an inline online method to evaluate surface properties of chocolate

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The determination of large scale surface quality of confectionery products inline and online is up to this point a gap in modern production line. In this study we propose a novel method of assessment. This method consists of a line scan camera (LSC) with an array LED subjecting the sample to dark field illumination. In various controlled cooling trials, the surface quality of dark chocolate was studied. It was found that several defects occurring on the chocolate surface could be linked to mold, processing, and storage defects. Notable is that in a linear cooling set-up, the leading edge side of the chocolate surface could be distinguished from other parts. This is thought to be caused by different local conductive heat transfer coefficients underneath the mold. Moreover detachment rings were observed as seen during the contraction phase of the chocolate. In the case of over- and undertempered chocolate significant difference in gloss after cooling were found. These differences were analysed with two image comparison techniques. One was to use the structural similarity index method (SSIM) and the other one was to assess the mean square error (MSE) of two pictures. Both techniques showed a good overall insight on the change of total structure.

3D surface characterization of cheese by confocal microscopy

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During chewing, the food is deformed and fractured. The fracture pathway depends on the food composition, structural element interaction and spatial distribution inside the material. In cheese, the spatial distribution of fat globules may be random, clustered or self-avoiding. Depending of this spatial distribution, the fracture pathway may be different and the freshly created surfaces may contain different fat globules content that can give rise to differences in sensory perception. To test if materials with different spatial distribution of fat globules show a different fat exposure after fracture a method has been developed to quantify the proportion of fat globules present on the 3D fracture surface.

Study of the effect of cooling rates and milk fat composition on the physical and whipping properties of recombined dairy cream

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This study investigates the effect of solid fat content and cooling rate on the physical and functional properties of recombined dairy creams containing AMF and a mid-melting fraction of MAF. Different variant of creams were tested. Each cream was subjected to fast and slow cooling and was tested for their particle size distribution, rheology, microstructure, viscosity, and whipping properties. Microscopic observation showed that cooling rate could regulate crystal structure inside fat globules. Slow cooling led to bigger crystals that were tangentially positioned near the interface of fat globules. Slow cooling on cream resulted in higher viscosity, stability to shear-induced partial coalescence, prolonged whipping time, higher overrun yet lower firmness of the whipped cream compared to those with fast cooling. In the end, cooling rate as well as fat composition could be a promising tool to alter the sensitivity of cream to partial coalescence and whipping properties by changing the internal fat crystal network

Hydrothermal decontamination treatment of functional powders

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Emerging technologies such as high hydrostatic pressure, irradiation and ultrasound treatments have highlighted the current industrial challenges on energy-efficient and effective solutions for food decontamination. In particular, the safety of powders for consumption as food or therapeutics are of concern owing to the fact that food productions are not an aseptic process. With the previous understanding of the difficulties for surface decontamination of powders, a pilot-scale reactor for powder decontamination was designed and implemented. The principle of thermal surface decontamination, namely vacuum-steam-vacuum, was validated for this device as a promising remedy to achieve microbial inactivation with preservation of the thermal-liable ingredients and the essential food powder properties. In this study, native cornstarch was used as the model particles to understand the relationship between powder properties and the process parameters, including steam temperature and duration of steam injection, during the first two stages of the process. Cornstarch powders were artificially contaminated with *Bacillus subtilis* spores to allow microbiological study towards food safety aspects. It was found that 2-logs of thermal-stable *Bacillus subtilis* spores on cornstarch powders was reduced within 1s of the decontamination process. Results from microscopy and X-ray diffraction patterns have shown minimal effect on the powder surface structure after steam injection despite a significant uptake of moisture. The study suggested the potential for the preservation of powder properties under short-time thermal inactivation in the pilot-scale prototype.

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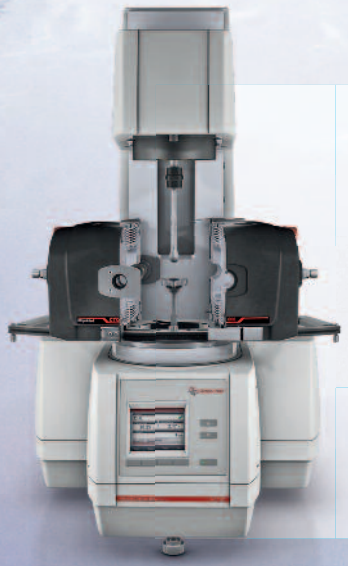

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