



international
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structure

ISFRS 2012

ABSTRACT BOOK OF THE

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INTERNATIONAL
SYMPOSIUM ON
FOOD
RHEOLOGY AND
STRUCTURE

EDITORS:
PETER FISCHER
ERICH J. WINDHAB

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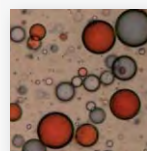
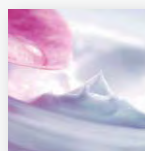
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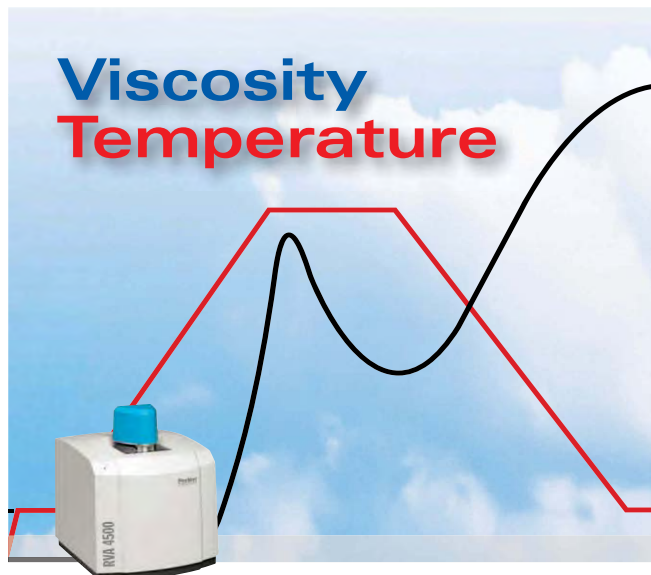
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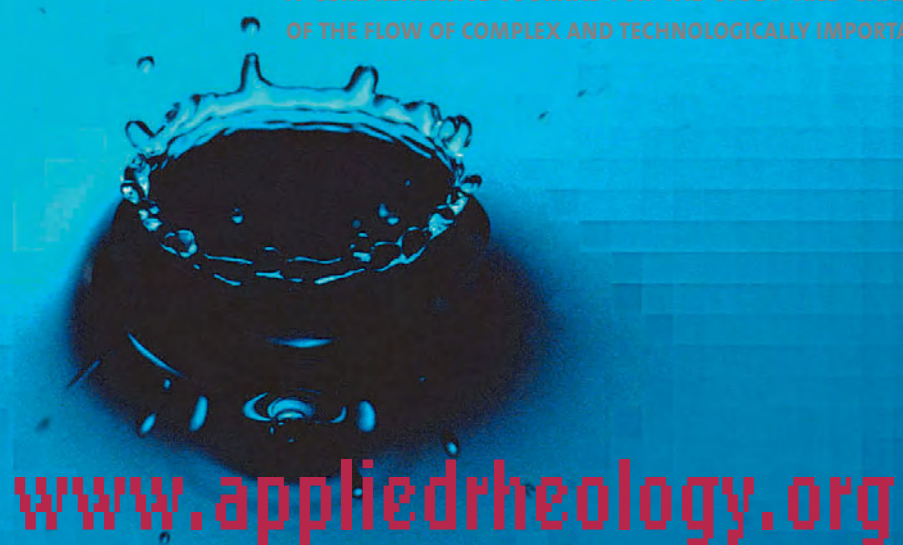


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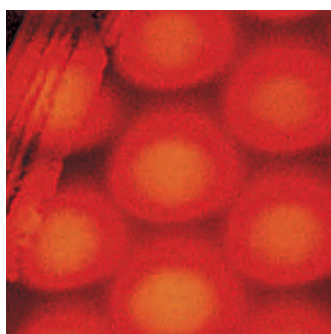
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Opening Lecture

Polysaccharide biopolymers: Lessons from nature on tuning molecular interaction

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Nature uses the pyranose ring as the basic building unit for a wide class of biopolymers. Because of their biological origin these biopolymers naturally find application as food additives, rheology modifiers, and in biomedical applications. These polymers range from being rigid skeletal material, such as cellulose that resist dissolution in water, to water-soluble polymers, such as guar, hyaluronic acid, and dextran. The flexibility of the basic pyranose ring structure to provide materials with such a wide range of properties comes from the cooperativity of the weak intermolecular interactions. We will present several examples of specific interactions for these systems: hydrogen bonding, hydrophobic interactions, and specific ion interactions. The relationship between molecular interactions and secondary structure will be our main focus. Hydrogen bonding mediated by steric interference is used to control of solubility of starch and the rheology of guar gels. A more interesting example is the hydrogen bonding induced by chemical modification in konjac glucomannan that results in a gel that melts upon cooling. Hydrogen bonding interactions in xanthan lead to gel formation at very low polymer concentrations which is a result of the fine tuning of the polymer persistence length and total contour length. Hydrophobic interactions in methylcellulose show a reverse temperature dependence arising from the entropy of the solvating water. Carrageenan gelation upon the addition of specific cations will be addressed to show the interplay of polymer secondary structure on chemical reactivity. And finally the cis-hydroxyls on galactomannans permit crosslinking by a variety of metal ions some of which lead to "living gels" and some of which lead to permanently crosslinked networks.

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Keynotes

The fluid mechanics of mastication, swallowing and digestion

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From a strongly simplified engineering viewpoint, the human digestive system can be regarded as one long tube with dramatic variations in diameter, cross-section, wall properties, pumping mechanisms, regulating valves and in-line sensors. This lecture follows the trajectory of a food bolus from the mouth to the small intestine and discusses the relevant fluid mechanical phenomena influencing sensorial perception, safe transport, and nutrient absorption. We focus in particular on three points during this 'journey':

- lubrication flow between tongue and palate
- 'pharyngeal squeeze' during swallowing
- peristaltic transport in the small intestine

Since the quantification of bolus rheological properties and its transitions during the mastication and digestion processes varies between the difficult and the impossible, we will try to construct some basic arguments that help to decide which degree of rheological modification can be expected to result in substantial differences for the success of the bolus journey.

The effect of rheology on the dispensing of complex fluids

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The determination of relaxation times in elongational flows, from the sub-millisecond scale of low viscosity solutions to the order of tens of seconds for complex formulations, presents a major challenge for current rheological instrumentation. However, in particular for free surface flows, filament formation and breakup of complex fluids, the elastic contribution and the related relaxation time has a strong influence on breaking dynamics, on droplet size and number and in particular on the formation of satellite droplets. The performance of processes as ink-jet printing, atomization, or the general dispensing of fluids relies therefore on the possibility to measure, structure and predict their viscoelasticity in extensional deformations. In this paper we will first introduce new experimental protocols in order to reliably determine elongational relaxation times from sub-millisecond to very long relaxation times from capillary breakup experiments [1, 2]. In a second step we are then going to demonstrate for several extreme classes of non-Newtonian liquids how their dispensing behavior is depending on a set of dimensionless numbers as the Ohnesorge, elastocapillary, and intrinsic Deborah number that balance the relative contribution of viscosity, inertia and elasticity to the thinning dynamics. Our aim is to give in this paper an outline of a 'map of misery' that explores for all relevant non-Newtonian fluid classes the boundaries for dispensing these fluids and that allows to predict within these boundaries the dispensing behaviour depending on the material properties of the fluid classes [3].

[1] Clasen C: Capillary breakup extensional rheometry of semi-dilute polymer solutions. *Korea-Australia Rheology Journal* 22 (2010) 331-338.

[2] Campo-Deano L, Clasen C: The Slow Retraction Method (SRM) for the determination of ultra-short relaxation times in capillary breakup experiments. *Journal of Non-Newtonian Fluid Mechanics* 165 (2010) 1688-1699.

[3] Clasen C, Phillips PM, Palangetic L, Vermant J: Dispensing of Rheologically Complex Fluids: The Map of Misery. *AIChE Journal*, 2011. DOI: 10.1002/aic.13704

Mucus microstructure and barrier properties

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Mucus layers coat and protect nearly all entry points into the body that are not covered by skin, including the gastrointestinal (GI) tract. Mucus is a complex biological material that provides lubrication, selective barrier properties, and many other critical functions in the GI tract, lungs, vagina, surface of the eyes, and other moist mucosal surfaces. Mucus serves as a physical barrier against foreign particles, including toxins, pathogens, and environmental ultrafine particles, while allowing rapid passage of selected gases, ions, nutrients, and many proteins. Its selective barrier properties are precisely regulated at the biochemical level, and are strongly dependent on length scale. At the macro-scale, mucus behaves as a non-Newtonian gel, distinguished from classical solids and liquids by its response to shear, while, at the nano-scale, it behaves as a low viscosity fluid to anything that does not adhere to it. Advances in the rheological characterization of mucus from the macroscopic to nanoscopic levels have contributed critical understanding to mucus physiology, disease pathology, and the development of drug delivery systems designed for use at mucosal surfaces. Indeed, until recently, human mucus was thought to be nearly impenetrable to drug delivery particles even as small as 59 nm in diameter. Particles that become trapped in mucus are typically rapidly cleared from various mucosal organs, usually within minutes to a few hours. Thus, while the barrier properties of mucus provide outstanding protection against infection and other potentially toxic substances, they have also thwarted efforts to achieve uniform and sustained drug and gene delivery to mucosal surfaces. Obvious parallels can be made to nutrient absorption in the GI tract. This talk will focus on our work to understand the length-scale dependent and adhesion-mediated barrier properties of mucosal fluids, and how this knowledge has guided the development of polymeric nanoparticulate carriers capable of improved drug and gene delivery to mucosal tissues.

Neutron scattering on food ingredients

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In this presentation an overview of neutron studies on food and food related systems will be given and further applications will be discussed. Neutron scattering is well suited for contributing to the understanding of food structure. The distances probed by Small Angle Neutron Scattering (SANS) match the size of typical supramolecular structures, such as micelles and polymer complexes found in food. When H₂O is replaced by D₂O, neutrons have a penetration depth which exceeds that of light and X-rays, and offer therefore an advantage for the study of turbid food systems. Wide angle neutron scattering, in combination with H/D substitution, or in combination with isotopic substitution of other elements is able to show the hydrogen bonded structure between e.g. sugar molecules and the correlation between ions and other solutes and water molecules (hydration shells). Examples of applications of SANS, Spin Echo SANS and wide angle neutron scattering will be used to show the potential of neutron techniques.

Bridging length scales and properties in food protein fibrils

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Processed food proteins, used as additives, are an appealing new way to impart food with specific structural and physical properties, as these components can be efficiently used as gelling agents, texture enhancers or interface stabilizers. The resulting physical properties at the macroscopic length scale do however depend strongly on the self-assembly process of these systems at the molecular level [1 - 3]. In this talk I will discuss how different length scales, from the single fibril to their macroscopic dispersions in water can affect the final physical properties of bulk or interfacial fluids made thereof. I will focus on our recent contributions on β -lactoglobulin and lysozyme fibrillation, which have a direct relevance to food processing. I will review our current understanding of the multi-stranded nature of these fibrils and elaborate on how colloidal and polymer physics concepts can be used to unravel the process by which these structural features convey protein fibrils specific physical properties. The talk will also address how structural properties of protein fibrils can be engineered to exploit these systems as building blocks in complex foods and biomaterials.

[1] Adamcik et al.: Understanding amyloid aggregation by statistical analysis of atomic force microscopy images, *Nature Nanotechnology* 5, 423 (2010).

[2] Lara et al.: General Self-Assembly Mechanism Converting Hydrolyzed Globular Proteins Into Giant Multistranded Amyloid Ribbons, *Biomacromolecules* 12, 1868 (2011).

[3] Jordens et al.: Disassembly and reassembly of amyloid fibrils in water-ethanol mixtures, *Biomacromolecules* 12, 187 (2011).

Rheological fingerprinting of complex fluids and soft solids

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Large amplitude oscillatory shear (LAOS) is used increasingly as a tool to measure nonlinear viscoelastic responses of soft solids and complex fluids. For many material systems the common practice of reporting only 'viscoelastic moduli' as calculated by commercial rheometers (typically the first harmonic Fourier coefficients G'_1 , G''_1) is insufficient and/or misleading in describing the nonlinear phenomena. The mathematical structure of the nonlinear response is fully captured by the higher Fourier harmonics, but these coefficients lack a clear physical interpretation. We introduce a framework for quantifying and physically interpreting deviations from linearity, which considers the 2D space of frequency and strain-amplitude first discussed by Pipkin to generate a unique 'rheological fingerprint' of a viscoelastic material. We build on the earlier geometrical interpretation of Cho et al. (2005) that decomposes a nonlinear stress response into elastic and viscous stress contributions using symmetry arguments. We then use Chebyshev polynomials as orthonormal basis functions to further decompose these stresses into harmonic components having physical interpretations. This framework naturally generates alternative measures of viscoelastic moduli, all of which reduce to G' & G'' in the linear regime, but offer different physical insight into a nonlinear response. We illustrate the technique using a gluten dough: The material functions of this gel under finite strains can be described by combining the linear viscoelastic response of a critical gel (Chambon and Winter 1987) with a Lodge rubber-like network to develop a frame invariant constitutive equation. This generalized gel equation is a simple but accurate description of the material functions in the linear regime and also at large strains, using only two parameters. In general, LAOS fingerprinting is invaluable for both characterizing and describing the nonlinear rheological response of a wide range of materials including biopolymer gels, regenerative polymer networks, foods and consumer products

Modelling eating

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Design of effective functional foods requires an understanding of how food structure responds during all stages of digestion, in order to ensure effective delivery of 'bioactive' food components and ensuing physiological and psychological responses involved, for example, in satiety control. The digestive process has traditionally been the domain of medical and nutritional science and is often focused on disease with little known about healthy functioning. An area that genuinely requires an interdisciplinary approach, much effort has been devoted in the last five years to bring diverse disciplines to bear on this subject, spanning human physiology, biochemistry, gut-to-brain signaling, physical chemistry and engineering. In particular engineering methods have been used in order to analyze the digestive systems to, understand fluid dynamics and the resulting effects on luminal events that determine the kinetics and pathways of digestion. In-vitro modeling has demonstrated that despite intestinal motility being very effective in mixing luminal contents during digestion, altering lumen viscosity by modifying food formulations can control the rate of starch hydrolysis and rates of absorption of glucose a surrogate for glycaemic response. An approach commonly used to achieve higher luminal viscosity includes addition of non-starch polysaccharides, which includes arabinoxylans, and important cereal food biopolymer which has potential to affect luminal viscosity and hence rates of absorption of cereal foods.

Structure design of soft biomaterials

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A main focus of structure design of foods, pharmaceuticals, hygiene products and other soft biomaterials is to master inherent structural properties of supramolecular biomaterials to control properties and tailor specific functions. Properties related to rheology and mass transport are crucial for a wide range of applications such as controlled release, barrier properties, swelling and dissolution, water binding as well as the sensory perception of food during consumption and release of nutrients during degradation in the body. The toolbox for microstructure characterization include a range of microscopically techniques that can cover length scales from nanometers to micrometers, but we also need to understand and follow the dynamics of structure formation and breakdown. This is necessary in order to fully understand how to tailor-make processes and conditions that give the desired characteristics of the product. Local events can have a dramatic effect on the overall properties. Therefore we must be able to master the heterogeneity and complexity of food products globally as well as locally. Global properties can be obtained by measurements such as NMR diffusometry or rheology. New techniques are available for measurement of properties such as local diffusion properties in complex structures. We also know more about the effect of structure complexity such as confinement effects in complex multiphase systems. Very interesting developments are taken place within Predictive science where experimental data on the micrometer and nanometer scale can be directly used to simulate flow and diffusion. This is the theme of the research program within the Excellence Centre: SuMo Biomaterials. The possibilities of new techniques and experimental designs together with simulations are being used to determine how mass transport can be controlled by the microstructure in a number of model materials. The intention is to identify crucial structural aspects for the design of soft materials and their surfaces for high performance biomaterials for function delivery. The knowledge will generate a new generation of structured biomaterials with tailored mass transport properties and related functionalities.

Rheology and functional structure processing in a reverse gastrointestinal engineering approach for personalized food

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Food properties related to consumers' preferences, acceptance and needs (PAN) are determined by (i) formulation and (ii) structure - a natural or process-induced hierarchically organized scheme of building blocks covering up to seven orders of magnitude in characteristic length scale from the molecule to the 'spoonable' piece. The interaction of such structure building blocks in relevant processes perform on characteristic time scales, which may also differ by several orders of magnitude. We implemented these aspects into the so-called multi-scale S-PRO² scheme addressing process-structure-property relationships as integral descriptors for a property-targeted food processing approach. An S-PRO² scheme cascade was further used to align and interconnect food processing steps from field to human cell. Optimizing manufacture of personalized foods we favor the S-PRO² cascade and applying a reverse engineering approach (REA). This starts from the site of final delivery of the PAN-related food properties in the human body and proceeds in reverse direction through the sequence of food processing steps of (i) digestive disintegration, (ii) meal preparation and (iii) industrial food production. To connect S-PRO² cascade steps accordingly one explores the optimum input-structure from the previous processing step, which allows for the best further structure tailoring towards the targeted property in the considered step. From the before said it is obvious that optimized food structure processing in the industrial process (I) should also consider structure modification under meal preparation conditions (II) and in particular structure disintegration during consumption (III) and digestion (IV). Points III and IV are major determinants for our personalized food system concept. In most food processing operations, the food or its intermediate form are treated under flow. Related rheological characteristics cover a wide range from dilute solutions to highly viscous or semi-solid composites. In the context of personalized food the delivery of target functions from a nutritional or health-supporting viewpoint are in focus. Consequently the research topic of "Flow-induced delivery of food function in the GI-tract" has been established. Delivery in this context relates to (i) food structure disintegration and (ii) mass transfer of micro-/macro-nutrients across the intestinal walls. In order to gain an improved understanding of food disintegration and function delivery under GI flow conditions three specific approaches were derived: (i) structure tailoring of functional food components for targeted physiological interaction (FFC+), (ii) characterization of gastro-intestinal deformation and flow patterns (FLOWGI) as well as (iii) rheological characterization of gastric juice and chyme (RHEOGI). Related insight into (1) functionalized food structure generation by (1a) new Surface Force Controlled Micro Media Milling and (1b) dynamic membrane multi-capsule processing technologies as well as (2) intestinal peristaltic flow characterization of non-Newtonian model chyme (FLOWGI-P) will be given in further detail and discussed with particular focus on structure-rheology relationships.

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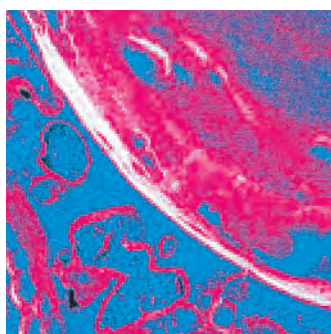
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Emulsions and Foams

Foaming of oils

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Unlike aqueous foams, very little literature exists in the area of non-aqueous foams even though this is an important field industrially, e.g. in confectionary manufacture and in the oil industry. Since the air-oil surface tension is normally at least half that of the air-water tension, the driving force for the adsorption of surfactant at the air-oil surface is much less. We have been investigating the foamability and foam stability of mixtures of surfactant or particles and oil in an effort to understand how to control the amount and stability of such foams. Incorporation of gases of different types (including air, carbon dioxide and nitrous oxide) has been achieved using different methods. These include aeration through a fine steel needle and a thermostatted foam column, incorporation of CO_2 into glass cylinders and the use of N_2O and CO_2 chargers in a soda syphon. A range of edible surfactants of the phospholipid and mono/diglyceride types as well as a number of different particle types was selected and the oils chosen varied from alcohols, alkanes, esters, aldehydes and triglycerides. Foaming was limited to oils of relatively high surface tension and the effects of surfactant concentration and temperature were probed. For high oleic sunflower oil, a high volume of foam can be generated but the subsequent stability depends on temperature and the type of gas with the latter influencing particularly the initial average bubble size. The solubility differences of the gases in oil no doubt influences the process of disproportionation of bubbles, although drainage of oil and coalescence of bubbles occurs also. The foam half-life varies from minutes to days depending on the system composition. The adsorption of surfactant at the air-oil surface has been probed by measurement of the surface tension; small reductions are observed.

Thermal behavior of phase inverted water-in-oil emulsion

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Water-in-oil emulsion stabilized by fat particles are a widely studied system [1, 2] and the microstructure of such systems has been well described [3]. Nonetheless, the formation of such structures, i.e. particle formation and migration towards the oil/water interface remains controversial when considering in which order these mechanisms take place [2, 4, 5]. Another level of complexity has to be considered as the oil phase contains more than one crystallizing material, leading to possible interactions such as co-crystallization. The aim of this work is to get a better understanding of thermal behavior of phase inverted water in oil emulsions. The emulsions studied were realized using mixtures of monoglycerides and triglycerides as crystallizing materials whereas liquid oil remained the major constituent of the oil phase. In such emulsion systems, fat crystals act as Pickering stabilizing particles, eventually leading to the formation of a solid shell around water droplets. Various concentrations of tri-palmitin and tri-stearin, exhibiting different melting points, were used as triglyceride material. Monostearate was used as emulsifiers and was therefore present at the interface, leading to locally increased concentration of crystallizing material and possibly nucleation site. Melting behavior of this crystallizing material was assessed using micro differential scanning calorimetry for emulsion systems (where these particles are present at the oil/water interface) and oil phase processed under the same conditions (cooling rate, shear conditions). Comparison of the melting profile gives information regarding the possible interactions between the two crystallizing material added and the effects of triglyceride concentration on melting behavior. Furthermore, comparing melting curves of fat crystallized in presence or absence of water allow the estimation of the level of influence of the concentrated monoglyceride at the interface on the crystallization process. The observed trend is that melting peaks occur at lower temperature for emulsion, suggesting the crystals formed in presence of oil/water interface are less stable. This reinforces the hypothesis that nucleation in emulsion systems is induced by monoglycerides present at the interface.

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Food ingredient particles for emulsion stabilisation

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Particle or Pickering stabilization of emulsions has generated a lot of interest in recent years for a variety of reasons such as widening formulation space, removal of synthetic surfactants from emulsion processing, stabilization of interfaces against Ostwald ripening and coalescence. Application in the food industry is limited due to the lack of food grade particles. Most commonly reported are the use of hydrophobised starch [1] and potentially use of modified fibers such as citrus fibers [2]. This research is concerned with food ingredient particles that are already found on the ingredient list of complex foods for which targeted structure functionality at interfaces was recently discovered [3]. The particles concerned are cocoa based. A large range of commercial cocoa powders with varying fat content, cocoa fibre and cocoa liquor were screened for their ability to stabilize emulsions prepared with oil and water as the only other ingredients. Alkaline and natural cocoa powders as well as cocoa powders exhausted of fat and water soluble molecules were included. Droplet size distributions were followed for at least 3 months and only limited changes were found. Thus, the ability of cocoa particles to stabilize emulsions appears to be independent of cocoa particle source. As was previously found with hydrophobised starch [1] the size spectra acquired on the emulsion samples using small angle laser diffraction equipment overlap the size spectra of the cocoa particle ingredient. The stabilization mechanism is still under scrutiny. Microscopic evidence points towards a Pickering mechanism with possibly only the smallest of the cocoa particles adsorbed at the oil-water interface. Increasing volume fraction of cocoa particles in the emulsions led to larger creaming indices at equivalent size spectrum. Surface tension measurements on the medium phase of aqueous suspensions of various cocoa particles as used for emulsion manufacture revealed surface activity. This could be shown to stem from particulate matter. Understanding the exact nature of the interfacially active particles taking into account that the different types of cocoa particles investigated are processed differently from primary source, the cocoa pod, and their wetting properties are the main focus of ongoing research. Further, the rheological properties of these emulsions and their stability in flow processes, and the potential to formulate complex emulsions is under investigation.

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Effects of surfactants on interfacial properties of ovalbumin layers at the air/water and oil/water interfaces

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Proteins and low molecular weight surfactants are often simultaneously present in two-phase food systems, such as emulsions, owing to the nature of the adopted raw materials (quite often containing different proteins); both of them are characterised by surface activity and, therefore, potential competitive adsorption phenomena at interface can establish. As a consequence, the macroscopic properties of the final emulsion are strongly affected by the characteristics of the obtained interfacial layers. In this work the adsorption of Ovalbumin protein in the presence of two different types of food emulsifiers, non-ionic Tween 60 and anionic Admul Datem, at the air-water (A/W) and sunflower oil-water (O/W) interfaces was investigated by means of transient interfacial tension measurements and dilational dynamic tests carried out by using a “pendant drop” tensiometer. Axisymmetric drop shape analysis (ADSA) was used to calculate drop volume, area and interfacial tension. A constant protein concentration (10 ± 2 % w/w), corresponding to the typical value used in many literature studies, was used whereas the emulsifier/protein weight ratio was changed from 0 up to 0.6. Temperature and pH of aqueous systems were kept constant at 22°C and 6.5, respectively. Differences between A/W and O/W interfaces were individuated for each studied system, probably because of dissimilar competitive phenomena among protein and tested emulsifiers. Adsorption behavior was discussed from a kinetic point of view in terms of molecular diffusion and penetration of adsorbed protein molecules in the presence of emulsifier ones. Interfacial dilatational moduli evidenced the potential formation of a 2D critical gel and they were analysed assuming the validity at the interface of rheological model used for the 3D critical gel. It was observed that the addition both of Tween 60 and Admul Datem emulsifiers on Ovalbumin layers adsorbed at both the interfaces cause a relevant weakening of the protein network. This rheological approach proved to be very useful to evaluate and to differentiate the protein network behaviour by varying the emulsifier/protein weight ratio in the solution, on the basis of rheological parameters which are a measure of the interfacial layer structure.

Interfacial and foaming properties of a popular food-grade surfactant

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The development of a suitable and stable product texture is important in foam-based food products. Air bubble incorporation into a liquid emulsion and the properties of the aerated product strongly depend on the stability and spatial arrangement of the bubbles. Surfactant molecules may adsorb at the air-water interface and stabilize air bubbles by reducing the surface tension during the aeration of food emulsions. Aqueous solutions of sodium stearyl-2-lactylate (SSL) are found to produce stable foams with very fine bubbles upon aeration. In this work, we investigated the foamability and foam stability of SSL aqueous solutions in an effort to understand how to control the amount and stability of such foams. The effects of SSL concentration, pH, temperature, solution age and method of aeration were probed. Certain of these foams are very stable. The foam half-life varies from days to weeks depending on the system composition and preparation conditions. The adsorption of surfactant at the air-water surface has been probed by measurement of the surface tension and will be discussed in connection with the foaming behavior.

Competitive adsorption of surfactant and hydrophilic silica particles at the oil-water interface: Interfacial tension and contact angle measurement.

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Understanding the stabilization mechanisms of emulsions is of crucial interest as they are used in many industries, such as food, cosmetics, pharmaceuticals, paintings, etc. If surfactants remain the source of emulsifiers, the use of fine divided solid particles has known a growing interest for the last few decades [1]. More recently, the need arises to make use of the advantages of both surfactant and particle-stabilized emulsions in order to eliminate the disadvantages associated with their individual use as sole emulsifiers. Combination of both emulsifiers has shown very interesting results in terms of emulsion stability and microstructure [2, 3]. Nonetheless, mechanisms that govern such emulsion stabilization, and particularly the adsorption of both emulsifiers at the interface, as well as their interactions, are not well understood. In this study, the effect of surfactants' type and concentration on the oil-water interfacial tension and three-phase contact angle in the presence of hydrophilic silica particles was investigated. Silica particles have been shown to have an antagonistic effect on interfacial tension and contact angle in the presence of both W/O (lecithin) and O/W surfactants (Tween 60 and sodium caseinate). Silica particles, combined with W/O surfactant, have no effect on interfacial tension, which is only dictated by the surfactant concentration, while they strongly affect interfacial tension when combined with O/W surfactants. At low O/W surfactant, both particles and surfactant are adsorbed at the interface, modifying the interface structure. At higher concentration, interfacial tension is only dictated by the surfactant. By increasing the surfactant concentration, contact angle that an aqueous drop surrounded by oil makes on a glass surface decreases or increases depending on whether the surfactant is of W/O or O/W type, respectively. This is due to the modification of the wettability of the glass by the oil or water induced by the surfactants. Regardless of the surfactant's type, the contact angle profile was dictated by both particles and surfactant at low surfactant concentration, whereas it is dictated by the surfactant only at high concentration. More specifically, in the presence of Tween 60, initially enhanced at very low surfactant concentration, the wettability is then reduced by increasing the surfactant concentration.

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Investigation of changes in formulation and processing parameters on the physical properties of cocoa butter emulsions intended for fat reduction in chocolate.

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The consumption of reduced fat food products offers a way of reducing the fat and energy ingested, with beneficial effects for body weight and health. This is particularly apparent for snack or treat products, that provide only limited nutritional benefits. Such is the case for chocolate. It has been shown that the fat content of chocolate could be reduced by introducing water droplets (i.e. a fat continuous W/O emulsion). The aim of the current work was to further understand the physical characteristics of cocoa butter emulsions produced using a process typically used for margarine production (a scraped surface heat exchanger and pin stirrer). Processing parameters were manipulated (shaft speeds, temperatures of the units and throughputs), as was emulsifier type and concentration. The addition of different hydrocolloids to the aqueous phase was also investigated, in order to create mechanical rigidity of the droplets, and prevent water loss on fracture. This process allows small droplets to be formed (typically 5 μm), and can be used to temper the cocoa butter. Crystalline shells were observed (using cryoSEM) at the droplet interface, suggesting that fat crystals were behaving as Pickering particles, sintering together at the interface, stabilising the emulsion. This work highlights the potential for cocoa butter emulsions to result in fat reduction in chocolate, using a one step process to achieve emulsification and tempering.

Optimisation of chemical composition and sonication for air filled emulsion production

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Air filled emulsions are a new and promising area for food calorie reduction. They can be produced, mixed and stabilized for use within standard oil in water emulsions, whilst leaving texture, taste and visual appearance unaffected. Currently the only known production method uses ultra-sonication, which is power intensive and therefore would prove expensive for scale up. Therefore optimization of the ultrasonic stage of the process is a key step towards minimizing the cost of producing large volumes of air filled emulsions. However, optimization of the whole formulation is also important so that process variability does not produce a disconnection between the energy input and chemical make-up of the process materials. Different concentrations of egg white proteins and bovine serum albumin with various sonication times, intensities and frequencies were tested to identify the optimum conditions for air filled emulsion production. The possibility for the recycling of un-reacted proteins back into the process is also considered for cost minimization.

Shrinking drops: Interfacial rheology versus compositional ripening in oil-in-water emulsions

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Oil-in-water emulsions stabilized with biopolymers or particles are important in foods, consumer products and pharmaceuticals. Often, the oil droplets are not perfectly hydrophobic, but are mixtures of compounds with a wide range of partition coefficients and volatilities. Due to concentration gradients and to gradients in the chemical potential such oil droplets are prone to compositional ripening, associated with dramatic changes in the morphology and size distribution of the emulsion. A competition arises between the effects of bulk mass transfer and interfacial viscoelasticity [1 - 3]. Here, we study emulsion drops of model volatiles subjected to mass-transfer driven shrinkage for various classes of physically different interfacial adsorption layers: (i) low molecular weight surfactants; (ii) dense interfacial adsorption layers with pronounced interfacial viscoelasticity; (iii) flexible biopolymer adsorption layers; and (iv) solid-stabilized (Pickering) drops. We combine video microscopy, pendant drop tensiometry, and interfacial shear and dilatational rheology to quantify and visualize the compositional ripening processes in emulsions with either simple or complex interfaces, and we discuss the role of these effects for flavor delivery systems and emulsion formulation.

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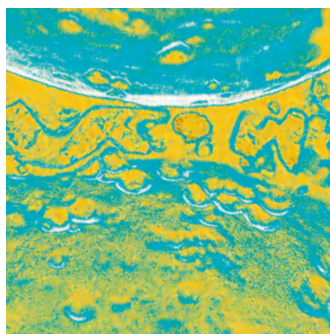
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Semi-Solid Foods

Steady shear properties and crystallisation phenomena in edible organogels

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The aim of the present work is the rheological and microstructural analysis of olive oil/monoglycerides/cocoa butter organogels as a potential oil phase of structured food emulsions. Organogels are organic solid-like systems produced with either low molecular-weight organic gelators (LMOG) or polymeric gelators, both able to self-assemble and form anisotropic structures which can join in three-dimensional networks, stabilizing the solvent. Step temperature ramp tests at different shear rates and controlled cooling conditions (5°C/min, starting from the initial temperature of 70°C) were adopted to evaluate the onset of crystallization temperature (T_{co}) and viscosity of different samples. In order to study the crystallization phenomena potentially occurring in adopted raw materials, a preliminary characterization was carried out on pure and blended fats (olive oil and/or cocoa butter), evidencing a relevant effect of adopted shear rate on T_{co} . Afterwards, the influence of monoglycerides on organogelation of either pure or blended fats was investigated, increasing the organogelator from 0.5 % (w/w) up to 60 % (w/w) and observing the increase of T_{co} with its concentration from $17.7 \pm 2.7^\circ\text{C}$ up to $64.5 \pm 1.0^\circ\text{C}$. Experimental results showed that T_{co} is only determined by the organogelator concentration, and it does not depend on the shear rate and fat source. On the other hand, sample viscosity depends on both the amount of organogelator and the oil phase composition, increasing with saturated fat concentration. The obtained results allow the fat to be designed with the desired properties: its crystallization point and rheology can be predicted on the basis of the amounts of the organogelator and the saturated fats, respectively.

Rheological response of protein-high sugar matrices – effect of protein composition

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Confectionary products such as caramel, toffee and fudge are complex food systems consisting of protein aggregates and fat droplets in a highly concentrated sugar matrix. For the consumer, sensory properties such as stickiness and hardness are important characteristics whereas for the industry also apparent yield and flow behavior are key with regard to processing and shelf life stability. In order to tune product properties with regard to altered processing or reformulation, proper quantification of texture and quality related parameters is required. Rheology is used to unravel these key aspects. The protein composition is varied to create different microstructures and hence different flow behavior. Apparent yield and flow behavior of viscoelastic materials such as structured protein-high sugar matrices can be determined by a creep flow test at a relevant shear stress range. The total compliance J_{tot} of a viscoelastic material shows a transient flow behavior going from rest into continuous flow, and consists of an elastic component J_{el} and viscous flow (creep viscosity η_{crp}). Both protein content and the amount of whey protein affect the flow behavior and the extent of elastic contribution to J_{tot} . We show that the build-up of an elastic network is essential for yield behavior (stand-up) and related to the value of J_{el} . From dynamic oscillation measurements information on the microstructure and structure breakdown is retrieved. It is shown that by increasing the whey protein: casein ratio both the hardness (G') and the extent of structure breakdown decrease. In relation to sensory properties, G' was found to linearly correlate with sensory hardness. The effect of protein composition on both flow behavior and hardness was found to relate to the extent of protein aggregation and the ability of protein aggregates to trap fat droplets into a continuous network, and both are affected by the presence of sugar. Light scattering and microscopy revealed that increasing the whey protein:casein ratio decreased the amount of aggregates resulting in a less coherent network and more flow behavior. In summary, creep tests, dynamic oscillation and light scattering, provide quantitative key parameters relevant for consumer quality attributes and industrial processing conditions of structured protein-high sugar matrices.

An investigation into the mechanisms driving structural change in Mozzarella and cheddar cheese during maturation

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Cheese undergoes significant structural rearrangement during maturation. At different stages in the maturation process different mechanisms are more prominent than others in driving structural change. These changes in structure have a direct effect on the functionality of the cheeses. The objective of this investigation was to identify the drivers of structural changes occurring in both Mozzarella and cheddar cheeses during maturation. Relationships between these structural changes and functional properties were also investigated. Fresh samples of both cheddar and low moisture part skim Mozzarella were collected from a commercial production line and assessed on a regular basis to identify changes occurring during maturation. A number of techniques were used to assess the changes in structure and movement of components within the cheese including NMR spectroscopy, dielectric spectroscopy, urea PAGE gels and scanning electron microscopy. These measurements were carried out in unison with measurements looking at changes in the functionality of the cheese. Significant changes in the structure of both Mozzarella and cheddar were identified over the maturation period investigated. Immediately after manufacture we observed changes occurring in the structure of both cheeses as the free water was absorbed into the protein matrix. This was observed both visually and in terms of the mobility of components in the cheese. After these initial changes due to water movement, the structure of the cheeses continued to change. These continued changes were observed through a number of the measurement techniques. As the structure and component mobility varied, so did the functional properties of the cheeses. At different stages in the maturation profile of both cheeses, different mechanisms had a more pronounced impact on structural change. These structural changes were found to have a significant impact on the functional properties of both cheeses.

Gluten free bread based on cereal protein melts

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The wheat protein gluten is the origin of the excellent elastic properties of wheat dough responsible for its good baking properties. Gluten forms a network which has high extensional viscosity and is strain hardening thus stabilizing and trapping air bubbles to give the desired porous structure typical of white bread. There are prolamin proteins corresponding to wheat gluten in all cereals, however, few of them have the required properties for baking, i.e. they especially lack the extensional rheological properties necessary for foam stabilization under normal baking conditions. There is still a desire to bake gluten free bread both due to the demand from gluten intolerant Europeans and due to high wheat prices in e.g. Africa. Gluten-free products are necessary for those suffering from coeliac disease caused by wheat gluten, which is a well-recognized problem in Western Europe and USA where about 1 % of the population is suffering from it. Wheat-free bread has been produced by utilization of the maize prolamin zein mixed with starch in proportions similar to flour. The protein was firstly processed over its T_g to form a melt where after starch and other components were added. The zein based dough was finally baked into porous bread. Dough based on maize flour was used as a reference. The extensional rheology of the dough was determined by Hyperbolic Contraction Flow and was shown to well predict final bread volume. The foam formation during baking involves high extension at low extension rates. The relevant rheological methods for predicting foam stability of dough must involve extension. The microstructure of the flour based dough revealed that the protein was trapped in its physiological structure and the amount insufficient. A protein content of 15 - 20 % was needed to form a good bread structure, and foam formation was facilitated by addition of plasticizing components and hydrocolloids such as beta-glucan rich bran and modified cellulose.

Rheology of starch-gluten systems - unusual biopolymer systems with a hierarchy of interactions

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Wheat dough shows a unique combination of material properties, owing largely to the specific storage proteins of wheat - the gluten. This macropolymer is able to form a network upon hydration and energy input (kneading), into which starch granules are embedded as filler particles. The resulting viscoelastic composite material is highly complex and the underlying molecular origin of its properties is still not fully understood. Since dough is a material in which structures at length scales spanning several orders of magnitude (starch granules, mesostructure of gluten macropolymer, structure of individual protein molecules) play a role, it is of interest to look at its elastic behavior at different deformation amplitudes to learn about the contributions of short and long ranged interactions. The easiest way to picture dough microscopically is that of a continuous protein phase with embedded starch granules, optionally including also a water and/ or air phase. One may thus identify three contributions to the overall elastic properties of dough, resulting from starch/ starch, starch/ protein and protein/ protein interactions. Given the very large contact area between the starch granule surfaces and its surrounding gluten matrix, it is expected that the starch granules' surface properties and their interaction with the proteins contribute to the overall rheological behavior of the dough, rather than them acting as inert filler particles. A method to separate the influence of the different interactions is the use of controllable model dough systems consisting of starch and gluten, in which the relative importance of them is varied by the respective amounts of starch and protein. Apart from the molecular properties of the individual components (e.g. development stage of gluten network, adhesion of gluten to starch surface) the microstructure of the material, such as the distribution of the starch granules, also plays an important role for dough rheological properties. This microstructure as well as the gluten structure and possibly also the interactions between starch granules as well as between starch and proteins are changed during the mixing process presumably on different time scales. Thus dough made from various starch/ gluten mixtures at different stages of development is investigated.

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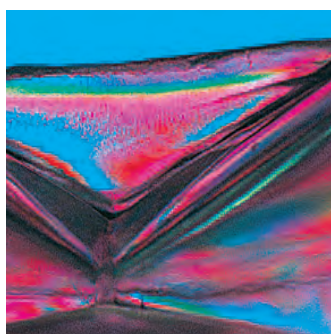
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Rheological Methods

Improved modeling of texture perception of semisolid milk products by friction tests

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Texture perception of food products in the mouth is attributed to a combination of rheology and friction forces generated between the tongue and palate, and the presence of particles. Lubricational properties are commonly characterized by means of a tribometer which measures the friction force between two bodies in rubbing motion. In this study, a tribometer accessory attached to a conventional rheometer is used for performing friction tests. The setup of the accessory follows a ball on pyramid principle. A steel ball representing the palate is pressed with a defined normal force on three inset plates as contact zones which are set in an angle of 45°. The first approach of our study was to examine the lubricating properties of elastic pads covering different intrinsic roughness and deformability scales of the human tongue surface as a function of lubricants. Our secondary objective was to investigate the hydrodynamic and friction forces exerted by a set of fatty yogurt products in a tribosystem in order to assess the relationship between friction data and well-established physical parameters (rheology, particle size). Lubricational behavior of tested elastic pads placed into the tribosystem was significantly affected by the patterning of the pads' surface, deformability was secondary. However, an increase of fat content of the lubricants involved a decrease in coefficient of friction for all tested elastic pads. A detailed, combined sensory and tribological investigation showed that friction data relates directly to creaminess. By including friction data into the regression model, the goodness-of-fit for the textural mouthfeel attributes viscosity and creaminess were improved. This study reveals an improved model for describing textural in-mouth sensations of yogurt products based on rheological, frictional and particle size measurements. This approach outlines a tribosystem equipped with suitable elastic pads that emulate human tongue properties as a promising tool to investigate frictional properties of semisolid food products.

Rheological evaluation of flow properties of large particulate-liquid model food systems

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Rheological characterization and evaluation of flow properties of large particulate-liquid model food systems was attempted by using the ball measuring system (BMS). The large particle-liquid model soup systems were composed of alginate gel particles (~ 10 mm) and starch solution as a liquid phase. The effects of particle concentrations (0 to ~ 60 %v/v), particle shapes (cube, sphere, rod and disc) and starch concentrations (3 and 5 %w/w starch) were investigated. The overall rheological behavior was characterized by the interactions between shear stress, shear viscosity and shear rate. The power law model was applied to further characterize the flow properties and obtain the consistency K and power law index n , which can link the BMS rheological data with the pipe flow properties of the system. The K increased and n decreased with increasing particle concentration at all particle shapes at 3 % starch in the liquid phase. The particle effect is further analyzed by means of the Krieger-Dougherty model to characterize the present systems by means of the maximum packing fraction ϕ_m of the particles and the intrinsic viscosity $[\eta]$. The ϕ_m depended on the particle shape, where the highest $\phi_m \sim 0.62$ was obtained in the systems with spherical particles at 3 % starch in the liquid phase, while the lowest $\phi_m \sim 0.54$ was obtained in the system with rod shaped particles at 3 % starch in the liquid phase. The obtained $[\eta]$ value depended on both particle shape and starch concentration and was largely in the order of 4.04 (cube), 3.28 (disc), 2.56 (sphere) and 2.32 (rod) at 3 % starch, where the order is in good agreement with reported small rigid particle systems (< 1 mm). The $[\eta]$ was 1.1 and relatively small in the 5 % starch spherical particle system, suggesting less effect of particles at high starch concentration. The present study suggests that the BMS technology is useful to evaluate the flow properties of large particle-liquid model food systems, and therefore, to estimate the pipe flow behavior using relatively small scale experiment (~ 0.5 L).

Industrial adaptation of the ultrasonic UVP-PD in-line rheometer system

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The new in-line concept for enhanced tube viscometry combining the Doppler-based Ultrasound Velocity Profiling (UVP) technique with Pressure Difference (PD) measurements was investigated for the first time over a decade ago. However, there still exists no commercial UVP+PD system on the market due to the industrial requirements and the complexity of real industrial fluids, such as non-Newtonian, non-transparent, high concentration of solids. A complete UVP+PD system with a new generation of sensors, electronics and software that meets the industrial requirements has recently been developed at SIK. The updated UVP+PD methodology and system developed at SIK has now been successfully installed in industry for different industrial applications. The new industrial version of the UVP+PD system can, for the first time, be used to monitor several industrial unit operations in real-time. Examples include; fat crystallization, heat treatment, injection grouting, rapid start-up or shutdown of the process, liquid displacements during rinsing or product change and in-line mixing. Measurements have been carried out in industrial process lines or pilot plants under true realistic processing conditions. The UVP+PD methodology and system developed at SIK has been successfully applied to a range of model and industrial fluids and suspensions, including fluids containing large particles and fibers, such as real foods, complex fat blends, paper pulp, mineral suspensions and cement based grouts. In addition, simultaneous measurements of the attenuation of transmitted ultrasound and changes in sound velocity provide an interesting option for determining particle concentration (e.g. solid fat content) in-line.

Rheological study of bubbly liquids with non-equilibrium deformation using ultrasonic velocity profiler

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Rheological properties of bubbly liquids produce unexpected behavior in liquid food, chemical solutions, and even in simple air-water two-phase flows in engineering. Our aim of the study is to elucidate mechanisms of frictional drag reduction by injecting bubbles on the wall turbulence. After hundreds of researches carried out in the last 35 years, one of most experimentally consistent hypothesis that has been survived is bubbles' drastic increase in local viscosity, which can suppress preferentially the turbulent eddies in boundary layers. Modification of the effective viscosity of spherical bubble mixture and that of deformable bubbles were already given. Nevertheless, fluctuations of the effective viscosity caused by non-equilibrium bubble deformation in transient flows have not been investigated. In this research, to understand the mechanism of changing effective viscosity with non-equilibrium bubble deformation, rotating cylinder filled with mixture fluid of silicone oil and bubbles is used for the setup. The cylinder rotation is controlled by a motor which can provide stepping drives for spin-up flows and sinusoidal motions for oscillating flows. In the cylindrical system, it is possible to observe the process of bubble deformation due to the fluid shear, i.e. recovering its original spherical shape via shear deformation, because the strain rate becomes zero in steady rotations. Since it is difficult to measure the velocity distribution of high void fraction fluids by optical methods such as particle image velocimetry (PIV), we adopt ultrasonic velocity profiler (UVP), which can measure even inside opaque fluids. To capture the deformation of bubbles, a high speed video camera is used. The temporal-spatial velocity distribution is measured by using UVP in both single-phase and two-phase conditions with bubbles of 1 mm diameter. This result shows that the wave shapes of velocity fluctuations is different between single-phase and two-phase conditions. FFT analysis on the temporal-spatial velocity distribution obtained by UVP shows the difference on spatial distribution of power spectrum of the velocity fluctuation between single-phase and two-phase conditions. From these data, we discuss change of the effective viscosity due to non-equilibrium bubble deformation.

Characterisation of viscoelasticity and dynamics in polymers and proteins using DLS microrheology

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The ability to precisely detect the onset of protein aggregation and draw insights into microstructural characteristics of the evolving microstructure plays a critical role in a variety of applications, including food colloids. We propose the application of a method based on optical microrheology for the detection of the onset of protein aggregation and for developing an understanding of the nature of the evolving network structure. Optical tracer microrheology relies on determining the behavior of tracer particles embedded in the sample of interest, and originates, with the seminal work of Phillies, and has evolved significantly over the last decade. The tracer microrheology approach overcomes the limitations of mechanical rheometry, as measurements can be effectively carried out on comparatively dilute samples and require only small sample volumes. The technique relies on the determination of the mean squared displacement of the tracer particles, from which several properties of the sample can be determined, such as viscosity, complex viscosity, the exponent of the tracer mean squared displacement power law fit and the viscoelastic modulus. Sample viscosity can be directly obtained from the slope of the mean squared displacement using the Stokes-Einstein relation in the Newtonian regime. For non-Newtonian samples the frequency dependent viscoelastic modulus and complex viscosity can be calculated using the Generalized Stokes-Einstein relation. The elastic modulus G' emerges as a key parameter in the detection of the on-set of protein aggregation and in understanding the nature of the evolving microstructure. Choosing the correct probe chemistry and size is highlighted as an important part of the experimental design. In this work we present applications of the technique to the determination of the viscoelastic modulus of PEG model solutions. We also use the tracer microrheology technique to determine the transition from purely viscous (Newtonian) behavior to viscoelastic behavior in a range of concentrations of BSA, as well as to track the gelation process during thermal denaturation of BSA.

Rheo-imaging to understand the flow of food emulsions

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For the fabrication of low fat, emulsified foods it is important to reduce the amount of fat while maintaining the same perception or mouth feel. The contribution of oil droplets to emulsion viscosity needs to be understood and enhanced for low fat food to maintain the same perceived creaminess. To this end we are interested in new ways to influence the flow properties of emulsions. The mechanical properties of liquid liquid interfaces depend strongly on adsorbed layer structure, composition and the type of stabilization. To demonstrate this we designed model emulsions with interfaces stabilized by surfactants, proteins, denatured proteins and colloidal particles. We have carefully ensured that the refractive index of all constituents is identical and fluorescently labelled them to allow for good imaging. We combined a fast confocal microscope mounted on an inverted frame with a rheometer to image directly the flow of these model emulsions in three dimensions in a controlled shear environment while we simultaneously measured the bulk rheology. We show that the type of stabilization indeed determines the mechanical properties of the interface which in turn affect the bulk mechanical properties of the emulsions. We characterized flow profiles, droplet interactions, rearrangements and deformations from the imaging and correlate these with bulk properties such as the dynamic viscosity and the yield stress obtained from the rheological analysis. This work will help in the design of model foods for planned sensory studies to determine the impact of interfacial design on sensory perception, and will contribute to the design of a new generation of better quality, reduced fat foods.

Online rheometry of complex process fluids

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The potential for using rheology in process industries for improving performance and product quality is well recognized. Typical industrial practices involve: i) Sampling small amounts of process fluids for rheological analysis with laboratory instruments. This can involve a significant delay between sampling and analysis - increasing processing time and consumption of resources. In addition, there is a risk that a single sample weighing only a few grams may not be representative of the bulk. Furthermore, many materials have a time- and shear history-dependent structure and delays, sample handling and environmental conditions to which the material is exposed in transit from pipe to lab can impact on the veracity of the laboratory measurement apropos the in-pipe rheology. ii) On-line techniques, usually torsional measurements on a vibrating probe, provide a single-point measurement in the high shear portion of the material flow curve. These measurements are usually treated as a QC number and monitored for deviation because it is difficult to confirm the accuracy of the measurement. Risks with these measurements include the difficulty in predicting fluid behavior from a single, high shear measurement, often of unknown accuracy. In this presentation we introduce an On-Line Rheometer (OLR) that is capable of providing continuous rheometric information, on-line and in real time. In- or on-line measurements remove many of the risks associated with delays, sample handling and environmental exposure. The OLR can provide viscoelastic data (G' , G'' , η^*) for the material in the pipe, providing a characteristic curve over a wide range of frequencies (1-100 Hz). The accuracy of these measurements can easily be compared to those measured in the laboratory. We use a 2.5 % solution of carboxymethyl cellulose (CMC) as a test fluid and obtain its linear viscoelastic characteristic while it is circulated in a pipe-loop equipped with the OLR and compare the performance of the OLR with those obtained using a laboratory rheometer as well as with other typical online instruments. The OLR performs well with respect to comparisons with laboratory measurements and may be used for QC in a similar way to traditional in-line monitoring, though clearly the OLR measurements are more powerful because a characteristic curve is generated.

Fourier Transform Rheology as a noninvasive morphological characterization technique for emulsions

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Emulsions consist of two liquid phases and cover a wide range of applications in daily life, like e.g. body care products, wall paints and food products. These products are of strong interest, due to their ability of combining two immiscible fluids as polar and non-polar substances, like for example water and oil. The emulsion properties like volume average radius and polydispersity have an influence on the texture of such systems and their rheological behavior [1]. Fourier Transform Rheology (FT-Rheology) is used to understand the relation between shear induced structures of dispersions and the non-stationary mechanical non-linear properties [2, 3]. Within this work the application of FT-Rheology as a noninvasive morphological characterization technique for emulsions shall be introduced. Non-linear simulations with the model of Maffettone and Minale in combination with the Batchelor theory (MM-B-model) yield a universal emulsion value E_0 at small strain amplitudes [4]. This universal value contains the nonlinear rheological parameter, $I_{5/3}$, as well as emulsion properties like volume average radius, interfacial tension and matrix and dispersed phase viscosity, and is a useful tool for emulsion characterization. Additionally, polydispersity is tackled as well as emulsions with a high volume fraction, like commercial samples. First results are established for dilute polymer blends with variable distributions and interfacial tensions, as well as for commercial samples with a high volume fraction [5].

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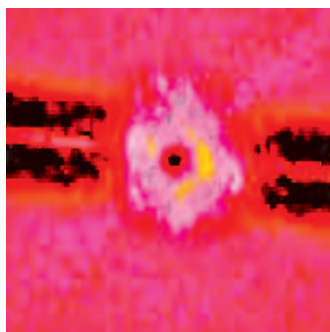
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Rheological Modeling and Numerical Methods

Rheology of concentrated suspension: Simulation, model system and industrial products

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In chemical, cosmetic, pharmaceutical, and food industries, the rheological properties of the products are important parameters to control for both their process and application. A significant part of these products consist in suspensions, colloidal or not, of solid particles, which are poly-disperse, polymorphic, physico-chemically heterogeneous and dispersed in a fluid matrix reactive or not. The link between formulation and material properties is then only empirical where, in concentrated suspensions, only small modifications have a huge impact on the flow properties. Despite the complexity of the formulations, the rheological flow properties obey to universal empirical laws, as these fluids may be ranged in a few categories of power laws fluids with or not a yield stress. Nevertheless, the understanding of these laws requires a description of the organization of the particles, as a function of the volume fraction, the shear rate and the inter-particle interactions. A better understanding between microscopic interaction and macroscopic properties would have a direct impact on the improvement of products formulation in term of quality but also development. Experiments and dissipative dynamics simulations of the flow of concentrated suspensions, colloidal or not in Newtonian or non-Newtonian fluids have been performed to quantify the impact of the structure on the macroscopic properties and thus from the start of the flow to the steady state. The simulations are, in a first step, validated with different model systems in order to study the impact of the volume fraction on the rheological properties. As a second step, simulations are used to quantify the local stress and their spatial organization or correlation. We show, at the onset of the flow, that the stress concentrated in the compression direction, where, in the steady state, the stress distribution is strongly anisotropic, oriented parallel to the flow direction. Finally, the effect of the solid volume fraction on the flow properties is revisited.

Fast and non-contact rheology of foodstuff using the foodtexture puff device and partial least squares regression

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For many foodstuffs, the rheological behavior is a crucial factor determining the quality. However, determining rheological properties is not always straightforward and in any case it is a time consuming process impeding to be used on a large sample size or even in line. Recently, a novel method called Foodtexture Puff Device (FPD) was developed that allows for a fast characterization of rheological properties. Its principle is based on deforming the surface of the product to be analyzed by means of a short air pulse, and to record the deformation at a high sampling rate as a function of time. Key parameters derived from the acquired waveform can then be related to rheological properties, or to a reference measure in general. However, for complex materials, the characteristic values such as maximal or residual deformation are insufficient to fully describe the underlying behavior. In our contribution, the goal was to relate the waveforms as a whole to reference rheological measurements. Based on two case studies, one on yoghurt samples and one on oils, we developed calibration models that relate the waveforms generated by the FPD to the rheology of the products. For this purpose, Partial Least Squares (PLS) regression was used. It tries to maximize the covariance between the inputs (the waveforms) and the output (reference measurements). It is shown that for the considered case studies, the waveforms hold relevant information so that quantitative information about the rheological properties can be derived. We also show the added value of using PLS calibration when compared to relating the reference measures to the maximal and/or residual deformation.

Digital bread crumbs and bread texture

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The mechanical behavior of bread crumb and consumer sensory perceptions of bread have been shown to go hand in hand. The driving factor behind high quality bread has generally been considered to be the loaf volume, which is analogous to porosity. Our recent publication [1] has shown that it is not only porosity that is important, but also how the porosity is distributed. Breads contain both open and closed pores. Breads are unique in that one large interconnected pore comprises 99 % of all total porosity. However, closed pores are also important, with larger closed pore volumes resulting in breads with higher Young's moduli, meaning a firmer texture. Qualitatively, it was also found that larger closed pore volumes lead to crumblier textures while smaller volumes lead to more pliable ones. The main challenge to delineating the effects of porosity on bread texture is creating samples with specific porosities, which is an immensely difficult task through experimentation. In rock mechanics, methods to study and predict the behavior of materials through the use of digital environments have been well established. Here we apply the knowledge previously developed for rocks to breads, by imaging the microstructure using x-ray microtomography, characterizing the pore distribution through statistical analysis, and testing for porosity and pore structure effects on mechanical behavior by applying homogenization techniques.

[1] Wang et al., (2011): It is a maze: the pore structure of bread crumbs, J. Cer Sci., 54(2), 203-210

Simulation of non-newtonian flow in collapsible elastic tubes

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Many biofluid mechanical processes encountered in the human body can be modeled as the flow of a non-Newtonian fluid in a deformable or collapsible elastic tube which is subjected to external and/or internal forces. Examples include the transport of food or digestive liquids in the esophagus, stomach and intestines, and the flow of blood through veins, capillaries and arteries. This is an inherently multiphysics problem, involving the deformation of an elastic tube and the flow of a liquid within the deforming tube. Specifically, as the fluid flows through the tube, it produces surface forces on the internal tube wall, which may cause the tube to deform. This change in the tube's shape causes a change in the flow field and hence the surface forces on the tube, which again affects the tube's deformation. Moreover, there may be large additional external forces applied to the outer wall of the tube which further cause the tube to deform, thus contributing to the complex interplay between the flow field and the tube deformation. Under many relevant conditions, the tube experiences very large deformations and may approach complete collapse. In this study, numerical simulations are used to investigate the flow of non-Newtonian fluids in deforming elastic tubes, and comparisons are made with experiments. The open source software package, OpenFOAM, is used as the computational platform. Specifically, the finite volume method is employed to solve the conservation equation of mass and momentum for velocity and pressure, where a Carreau-Yasuda viscosity law is used to model the non-Newtonian fluid. Two flow cases are considered. In the first case, the steady flow of a non-Newtonian fluid in a deformed tube is calculated. The computational domain in these simulations is constructed from the experimentally determined tube shape at various cross sections. Comparison of the computed velocity field with the steady-state velocity field measured in the experiments using UVP show overall good agreement, as does the pressure drop along the tube's axis. In the second case, an external force is applied to deform the tube in such a way as to mimic peristaltic motion. As in the experiments, this is achieved in the simulations by moving a roller along the exterior of the tube. Again, comparisons between the simulated and measured velocity fields are made.

A composite model to predict cheese sensory texture

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Many foods are bio-based materials that consist of structural elements spanning a wide range of length scales. The food sensation or sensory texture of these materials depends strongly on the structural arrangement, mechanical behavior, and interactions of the structural elements. In most food materials a full understanding of the relation between microstructure and even the basic mechanical aspects of sensory texture is still lacking. As a consequence, no systematic strategies towards influencing the sensory texture have been explored. In this study the relation between microstructure and mechanical properties (e.g. sensory texture) of semi-hard cheese will be established. At the micrometer scale, cheese is modeled as a composite material, in which fat particles are dispersed in a protein-water matrix. In order to clarify the separate contribution of fat and protein material properties and the composite morphology to the overall mechanical properties a hybrid experimental and computational procedure is adopted. A Representative Volume Element (RVE) is defined in which filler volume fraction, size distribution and spatial distribution is prescribed. For given values of these parameters a 2D filler composite morphology is created automatically and subsequently translated into an element mesh. Elastic properties of the fat particles are taken from literature and the constitutive behavior is described with a neo-Hookean law. A linear viscoelastic model for the water-protein matrix is created by fitting storage and loss modulus data from oscillatory shear experiments on a multimode Maxwell model. With the morphology of the composite and the material properties of its constituents defined, a virtual stress relaxation experiment is simulated with Finite Element Analysis. At different levels of fat concentration, this is compared with a stress relaxation experiments on real cheese samples. By systematically changing morphology of the virtual composite and comparing simulation results with experiments the contribution of structure to overall mechanical properties is elucidated. In future work, non-linearity will be introduced into the constitutive equations and large deformation experiments will be performed. Furthermore an extra length scale will be introduced in the structure-property model by describing the protein-water matrix as a network of sponge-like particles. Constitutive laws at this length scale will be derived using micro gel suspensions and Brownian dynamics simulations.

Droplet production from micro-pores into shear flow fields

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Immiscible multiphase fluid systems are relevant in many applications in industries such as the food, pharmaceutical, and polymer industry. However, the efficient and reliable production of these fluid systems with desired properties remains a challenge. One approach used to create droplets of one liquid in another liquid relies on forming and detaching droplets from pores. Specifically, a dispersed phase fluid is transported through a pore and into a channel containing a continuous phase fluid. Droplets of the dispersed phase are then detached and transported through the channel by the flow of the continuous phase. Devices that take this approach include the rotating membrane and T-cell devices. The former consists of two concentric cylinders, in which the inner cylinder is composed of a membrane containing many small pores, and one or both of the cylinders rotate to produce the continuous phase flow field. In the T-cell, a rectangular or cylindrical pore opens into a rectangular channel where the continuous phase flow is pressure driven. In this study, the formation and detachment of droplets from micron-sized pores into shear flow fields is investigated using numerical simulations. Newtonian and non-Newtonian fluid phases are considered. The open source software, OpenFOAM, is used as the basis for the simulations. The numerical algorithm employs the finite volume method for solving the mass and momentum conservation equations with a volume-of-fluid approach for capturing the fluid-fluid interface. In addition, a contact model is used to account for the interaction between the fluids and the walls of the channel and pore. Various geometrical, flow, fluid and interfacial parameters govern the characteristics of droplet production from pores, such as droplet size and detachment frequency. One focus of this study is the effect of the flow type in the channel on drop characteristics, specifically the shear flow fields that are encountered in the T-cell and rotating membrane devices. In addition, the effect of the channel height relative to pore width is investigated, as well as the effect of the contact model and the fluid rheology. Comparisons are made with experiments when possible, where initial comparisons generally show good agreement.

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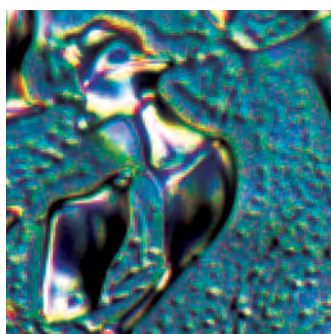
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Biopolymer Solutions and Gels

Controlling the shear thickening behaviour of mamaku polysaccharide with cation valency and concentration

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A novel polysaccharide with unusual rheological properties has been extracted from the fronds of a native New Zealand black tree fern or Mamaku in Mori. The polysaccharide in its native form possesses shear thickening, anti-thixotropic behavior at intermediate shear rates of 10 - 100 1/s (from 5 % w/w concentration), and Weissenberg effect [1]. Researchers have found remarkable physiological effects of the Mamaku extract on antrocorporal (stomach wall) contractions during digestion. The unique rheological behavior of such a biomaterial could potentially impart satiety to the consumer via an unconventional mechanism [2]. The Mamaku extract contain a high mineral content of potassium (6 % w/w), sodium (1 %), calcium (0.2 %), magnesium (0.1 %) and aluminum (0.1 %) in its native form. Upon removal of these minerals and low molecular weight sugars via dialysis, the shear thickening effect is lost. This is however reversible by adding of metallic chloride salts back into the dialysed solution. The valency and concentration of the cation is a crucial factor in determining: i) zero-shear viscosity, ii) critical shear rate (or shear rate at onset of shear-thickening), $\dot{\gamma}_c$, and iii) the extent of shear-thickening of the solution. At a similar salt concentration (0.05 M), the monovalent cations i.e. Na^+ and K^+ had the least reduction in zero-shear viscosity, and both the lowest $\dot{\gamma}_c$ and degree of shear-thickening. The effects show an opposite trend with increasing cation charge. In addition, the rheological behavior was highly similar for cations of equal valency e.g. between Na^+ and K^+ ; Ca^{2+} and Mg^{2+} . This study presents an opportunity to control the rheology of the polysaccharide by manipulating the valency and concentration of the added salt.

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Starch gelatinization in carrageenan solutions: Exclusion, adsorption, or absorption of carrageenan chains by starch granules?

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Starch-hydrocolloid combinations are widely used in food products. Hydrocolloids influence the texture of starch-containing media, indicating possible interactions. However, the mechanisms are not yet elucidated. One of the proposed (but not established) mechanism suggests exclusion, at least partly, of the hydrocolloid by starch granules during gelatinization and thus an increase of hydrocolloid concentration in the continuous phase [1]. The objective of this work was to investigate the behavior of κ (Ck) and ι (Ci) carrageenans during gelatinization of modified waxy maize starch for different carrageenan/starch ratios. Two experiments were done: one with increasing starch concentration (from 0.5 to 4 %)/carrageenan (0.2 %), and the other with starch (2 %)/increasing carrageenan concentration (0.1 to 0.4 % Ck and Ci). The starch dispersed in carrageenan solutions was gelatinized in non destroying conditions (up to 85°C, 10°C/min) and separated from the suspensions by centrifugation (10000 g/5 min). The carrageenan concentration in the supernatant was evaluated with the help of Ck and Ci reference curves connecting the apparent viscosity measured at 20°C to the concentration of carrageenan. This was compared to values calculated using two extreme assumptions: (H1) carrageenan penetrates into the starch granules driven by solvent and (H2) carrageenan does not penetrate at all into them (total exclusion). A percentage of carrageenan that is trapped by starch granules (% $C_{trapped}$) was then evaluated. The % $C_{trapped}$ was higher for Ck (~ 80 %), than for Ci (~ 30 %). However, neither the increase of starch nor carrageenan concentration in the mixtures affected the % $C_{trapped}$ (Ck and Ci). In order to understand the mechanism of trapping of carrageenan by swelling starch granules, the influence of the contact moment between starch and carrageenan (before and after gelatinization) was studied. Similar % $C_{trapped}$ was obtained, independent of whether the contact moment was before or after gelatinization. Confocal laser scanning microscopy visualizations of the blends clearly showed carrageenan adsorbed on starch granules. As a conclusion, carrageenan chains were found to be partly excluded, and partly adsorbed by starch granules. The proportion of each phenomenon depended on carrageenan types, but not on the mixture ratios.

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Rheology of beta-lactoglobulin and hen egg white lysozyme nanofibrils

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Many proteins and peptides of diverse sequence and structure self-assemble into morphologically similar fibrillar aggregates. These nanofibrils are remarkably ordered and stable and they exhibit unique mechanical properties. Therefore, they can be useful as building blocks for protein-based functional materials in food applications. The goal of our work is to elucidate the relation between the structure and mechanics of nanofibrils and networks thereof, which is an essential step towards their applications. We will show rheology and laser tweezer microrheology measurements of the viscoelastic properties of nanofibrils of two food-related proteins: β -lactoglobulin (β -lg) and hen egg white lysozyme (HEWL). Since the macroscopic rheology of the nanofibril networks will depend both on the stiffness of the constituent fibrils and on the energetic of their interactions, we investigate for the same system both the micromechanics of individual fibrils and the emergent mechanics of networks on the mesoscopic scale. We will show that the mechanical properties of the fibrils and their networks sensitively depend on the beta-sheet content of the nanofibrils, probed by Vibrational Sum Frequency Generation spectroscopy.

The processing-structure-functionality relation in whey protein nanofibril dispersions

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Several globular food proteins form nanofibrils when heated at low pH and low ionic strength, including proteins from soy, egg, fish and milk. Protein nanofibrils enhance viscosity more efficiently than other types of protein aggregates due to their extreme aspect ratio, which allows them to form physical entanglement networks. A range of fibril morphologies can be obtained, and their rheological properties differ, but in ways that are not always obvious. Here we survey the relationship between processing variables (pH, temperature, added salts), structure and rheological functionality for whey protein nanofibril dispersions. The bulk viscosity of whey protein fibril dispersions depends on the volume fraction of fibrils, but in many cases the length, flexibility, shape and alignment of fibrils have more influence than simply the amount of fibrils present. Adding salts modifies the kinetics of whey protein self-assembly as well as transforming morphology, and bulk viscosity varies with salt concentration in a concentration-dependent but nonlinear way. Heating temperature affects the length and stiffness of fibrils, with consequences for bulk viscosity. The economic feasibility of using fibrils as food ingredients probably relies on being able to maintain functionality after drying and rehydration. Freeze-drying fibril dispersions appears to fracture individual fibrils, resulting in decreased viscosity after rehydration. However the size of the viscosity decrease is very different for different types of fibrils, and this disparity can be understood by considering the processing-structure relationship.

General self-assembly mechanism converting hydrolyzed globular proteins into giant multistranded amyloid ribbons

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Most proteins can form supramolecular fibrillar aggregates in vivo or in vitro, leading to neuronal and systemic pathologies and suggesting a common tendency of polypeptides to form amyloids. Hen egg white lysozyme (HEWL) and β -lactoglobulin (BLG) are both globular proteins forming amyloid fibrils in vitro but they differ in their molecular weights, primary to quaternary structures and physical properties, hindering any direct comparison in their amyloidosis pathway. In the present work, however, we intend to draw general conclusions on their fibrillation process, showing a cross-sectional growth in both multistranded protein systems, progressing up to unprecedented levels. The number of filaments per aggregate increases with incubation time and goes up to 17 filaments per fibril for HEWL (173 nm wide) and 16 filaments for BLG (160 nm). For both proteins, the formation of such similar large multistranded twisted and helical ribbons is concomitant with the hydrolysis of the pristine protein into very short fragments (< 6 kDa) and is associated with further structural changes. These results demonstrate that a complete hydrolysis of the protein at long incubation time leads to the newly reported massive ribbons. These results open new perspectives in the understanding of fibrillation processes in globular proteins and amyloids polymorphism. Experimental results all provide convincing evidence that protein unfolding and complete hydrolysis are essential for the formation of large laminated aggregates and that small peptides fragments participate in the formation of these fibrils, irrespectively of the initial size of the protein. The exceptional similarities in the fibrillation steps, building blocks and final amyloid structures for globular proteins as diverse as BLG and HEWL and the direct analogies with aggregation processes in short synthetic polypeptides, further support a possible general common unfolding-fragmentation and aggregation mechanism for amyloidosis in globular proteins.

Do exopolysaccharides from lactic acid bacteria enhance textural properties of milk gels when added prior to acidification?

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Exopolysaccharid (EPS)-producing lactic acid bacteria (LAB) are widely used in the manufacture of dairy products to improve texture and to decrease syneresis without the use of other hydrocolloids. Although texture-enhancing effects of the in situ produced heteropolysaccharides are generally acknowledged, little is known on the relevance of the continuously changing environment during fermentation and the exact mechanisms of the structure-function interactions between EPS and milk proteins. The aim of our study was to evaluate whether the addition of purified EPS prior to acidification results in milk gels with similar enhanced rheological properties. After optimization of batch fermentation to gain higher yields of EPS from *Streptococcus thermophilus* ST143 (Chr. Hansen A/S, Horsholm, Denmark) and *S. thermophilus* DGCC7710 (Danisco A/S, Niebüll, DE), EPS were isolated and purified. Defined amounts of lyophilized EPS powder were dissolved in skim milk, and subsequent acidification was performed either with D-glucono-d-lactone (GDL) or single strains of lactic acid bacteria. A non-EPS producing strain was used as a reference for fermentation experiments. Fermentation with single strains of *S. thermophilus* resulted in a gel stiffness (G'_{max}) of 304 and 258 Pa for ST-143 and DGCC 7710, respectively, compared to 154 Pa with the reference strain. However, time to reach pH 4.6 varied considerably and was 705 min (ST-143), 488 min (DGCC 7710) and 787 min (reference). In model gels that were acidified with GDL for harmonization of gelation time, corresponding values of G'_{max} were 300 Pa (EPS from ST-143 was added to the milk prior to acidification), 320 Pa (EPS from DGCC 7710 was added) and 180 Pa (reference, no EPS added). When EPS were present, the onset of gelation ($G' = 1$ Pa) took place at higher pH, independent whether they were produced in situ or added to the base milk. Our data clearly show that the external addition of purified EPS results in a similar impact on shear viscosity and structure stability of stirred gels. Further experiments, that concentrate on the importance of fermentation parameters are under progress.

Engineering of heat-induced milk protein particles for acid gels

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The heat-treatment of skim milk at 90°C/10 min has long been reported to improve its acid gelation; hence its wide application in yoghurt manufacture. These changes have been attributed to the formation of heat-induced whey protein/kappa-casein complexes that interact with the casein micelles on heating, or shortly on acidification. Through this interaction, the complexes are believed to functionalize the casein micelles as to provoke their early destabilisation on acidification, to increase their bridging connectivity and to enhance their ability to engage stronger interactions on formation of the network. However, the exact nature of the colloidal attractive/repulsive interaction forces that drive gelation and are affected by the complexes is not known. In the seek for a comprehensive view of the acid gelation process in heated milk, a range of artificial protein complexes were produced, having one candidate property largely modulated. The success of the modification was assessed through the complete characterisation of the artificial complexes. The complexes were then introduced into a model milk system, and the acid gelation of these systems was studied. Examples are shown on repulsive electrostatic and attractive hydrophobic interactions.

Rheological and electron-microscopical studies of structure formation in milk during acidification by GDL

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Glucono-Delta-Lactone is a common tool to simulate the slow acidification of milk by microorganisms. Milk samples were acidified by GDL at varying concentrations and temperatures. The decrease of pH was recorded. It was found that the acidification rate follows a first order reaction and depends only on GDL concentration and temperature but not on fat content and thermal pre-treatment. The size of casein micelles and particles in skim milk during acidification was measured using laser diffraction (Beckmann Coulter). The size of the casein micelles in UHT milk is about $0.13\ \mu\text{m}$. Particle size is constant during the first phase of acidification independently of the GDL concentration. Only the temperature affects the growth of the particles but not the GDL concentration and the acidification rate. At 23°C the start point of aggregation is at pH 5.0. At 42°C the aggregation starts at pH value 5.3. It was observed that these particles become bigger. For the investigation of the gel formation oscillation rheometry was chosen. The rheological measurements were carried out using a Physica UDS 200 (plate-plate measuring system). Storage modulus (G'), loss modulus (G'') and loss angle ($\tan\delta$) of tempered and inoculated milk were recorded at a frequency of $1\ \text{s}^{-1}$ and a deformation of 1 %. As it is state of knowledge the gels develop after a phase of pre-acidification. G' and G'' increase and $\tan\delta$ decreases. The point of maximum, both of G' and G'' , is reached at the same pH value (4.6) At higher GDL concentration this point is reached earlier. During the initial phase of acidification the viscosity was measured by rotational mode (cone-plate system) since it is more sensitive. A slight decrease of the viscosity during this phase is an indicator for changes in the micellar structure. At a pH value of 5.2 the viscosity increases until the cone starts to slip due to syneresis. The gels were characterized by uniaxial compression (Texture Analyzer). The gels become stronger at higher acidification rates and temperatures. TEM micrographs show the conformational changes of the casein micelles during acidification.

Alkali-extracted hybrid carrageenans from *Mastocarpus stellatus* seaweeds grown in an IMTA: A palette of gelling properties.

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Mastocarpus stellatus are red seaweeds which can be exploited to produce κ / ι -hybrid carrageenan (KI) [1]. The increasing demand of such additives in a wide range of industrial applications is gearing the need for a sustainable production of this natural gelling agent. Following a recent study on the seasonal variation of KI in the seaweed [2], *Mastocarpus stellatus* seaweeds were collected on the Portuguese coast and then used as water cleaning agents in a fish farm, in order to produce an Integrated Multi-Trophic Aquaculture (IMTA) system [3] allowing the sustainable production of carrageenophytes. After a month in IMTA, seaweeds were alkali treated with NaOH or KOH and KI were extracted. Here we report on the effects of the alkali concentration, alkali treatment duration and type on the KI chemistry and gel properties in the presence of NaCl or KCl. The originality of this study lies in the choice of seaweeds from which KI is isolated, namely *Mastocarpus stellatus* grown in an IMTA, and also in the systematic comparison between the chemistry of the seaweeds assessed with DRIFT and CP-MAS NMR and the chemical structure of extracted KI. Gels of 1 %wt KI in 1M NaCl or 1M KCl solutions were rheologically tested with SAOS. The thermo-rheological characteristics of gels will be presented and compared to the chemical structure of extracted KI and of polysaccharides contained in the seaweeds.

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Impact of dextran and dextran sulfate on the structural and rheological properties of acid milk gels

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We used a model acid gel system to investigate the impact of adding uncharged and negatively charged dextran to milk at different pH levels. Two concentrations (0.075 and 0.5 %) dextran (mol wt: $2.0 \cdot 10^6$ Da) or dextran sulfate (mol wt: $1.4 \cdot 10^6$ Da) were added to cold milk ($\sim 0^\circ\text{C}$) that had been acidified to pH 4.4, 4.6, 4.8 and 4.9. Acidified milks were heated in the rheometer or microscope slide at a rate of $0.5^\circ\text{C}/\text{min}$ to 30°C (which induced gelation), and held at 30°C for 17 h. Microstructural and rheological properties of gels were determined by fluorescent microscopy and small amplitude oscillation rheology, respectively. Microstructural analyses indicated that gels made at pH 4.4 with the addition of 0.5 % dextran exhibited extensive phase separation, while gels made with 0.075 % dextran or the control had homogeneous structure. At higher pH values, gels made with 0.5 % dextran did not exhibit obvious phase separation. At all pH values gels made with 0.5 % dextran sulfate showed larger pores in the matrix than control gels. Gelation temperatures (during warming of acidified milks) and gel stiffness after 17 h at 30°C were similar for control or gels made with 0.075 % dextran. Addition of 0.5 % dextran at pH 4.4, 4.6 and 4.8, resulted in gelation at significantly lower temperature than the control gels or gels made with dextran sulfate. In acidified milks at pH 4.9, gels made with 0.5 % dextran sulfate had lowest gelation temperature. Stiffness of gels made with 0.5 % dextran sulfate at pH 4.4 was approximately 1.5 times higher than the control or gels made with 0.5 % dextran. This study demonstrated that low concentrations of uncharged dextran did not impact rheological properties; high concentrations gave earlier gelation, which might be due to depletion induced attractions between casein micelles. At pH values below the isoelectric point negatively charged dextran produced stiffer gels, possibly due to attractive interactions with caseins. Further investigations of the effects of phase separation in these systems are underway.

The use of tribology as an in-vitro method to study model food structures

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A variety of methods and equipment have been explored that characterize and attempt to predict in-mouth behavior. Better understanding of material performance has become increasingly significant in the efforts to design new food products. Tribology, the study of thin film and lubrication behavior, is one technique that has seen growing interest, and is often considered as an extension to rheological measurements, but at a smaller scale. Previous tribology work has investigated the development of equipment configuration to relate to the in-mouth environment. It has focused on gaining absolute, and often time independent, data for structures, similar to viscometry measurements. The development of tribology equipment has been undertaken to assess its ability to characterize structures, but also to extend this to study more dynamic measurements, which could be better associated with oral processing. In this work two case studies are presented. Firstly, the measurement of inhomogeneous samples using tribology, where lubrication response depends on the mixing properties of the sample with water. Secondly, dynamic measurements of temperature induced biopolymer ordering, where lubrication is a function of the extent of ordering experienced. This ordering process is compared to viscometry measurements. Results show that for inhomogeneous samples the lubrication behavior varies. The tendency of a structure to mix with water determined the speed at which an introduced sample affected lubrication. This behavior would suggest that in-mouth, the inherent lubrication properties of structures would be affected by how completely it had mixed with saliva in the mouth. Further to this, dynamic lubrication measurements of biopolymer ordering show definitive differences at each stage of the ordering process, both in tribology and viscometry. However, the patterns of lubrication recorded for different sample concentrations show different trends between samples to that of viscometry. Tribology can provide information on model food systems. This technique presents different trends and more dynamic behavior of structures when compared to other methods such as viscometry.

Gel particulate rheology and tribology as a function of volume fraction and particle stiffness

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We produce gel particulate thickeners by applying a shear force to kappa carrageenan and potassium chloride solutions as they undergo gelation on cooling. During this process, the gelled nuclei that form during the onset of spinodal decomposition are segregated by shear forces that restrict the molecular ordering to within discrete particles, rather than macroscopically as in quiescently cooled gels. The resultant fluid gels have considerable potential to be used as particulate thickeners in fat-reduced food products. The bulk rheological properties of the fluid gels are largely influenced through volume fraction and particle elasticity (controlled through dilution and carrageenan concentration, respectively). We show the effect of these structural parameters on rheological properties (small and large deformation) and tribological response. Tribology (thin-film rheology) concerns lubrication, friction, contact mechanics and wear, and has shown to be incredibly valuable in relating analytical data with in-mouth textural attributes. In this work, tribology is investigated using a tribometer with a ball-and-disc made from PDMS (polydimethylsiloxane) surfaces that are aimed to resemble oral surfaces in terms of elasticity. The purpose of this work is to understand the structural dependence of fluid gels on rheological and tribological behavior in order to optimize their use as fat-replacers in soft solid and liquid foods. Zero- and infinite-shear viscosities are compared with values predicted by models provided by Krieger-Dougherty and Batchelor-Einstein, for high and low volume fractions respectively. We also report the G' and phase angles as a function of volume fraction and particle elasticity, and use these, together with microstructure analysis, to explain the deviations from hard-sphere behavior. The lubricating abilities of such systems is shown to be dependent on both particle stiffness and packing behavior, whilst seemingly independent of viscosity.

Rheology of gelatin–polysaccharide food gels

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Mixed biopolymer systems can be considered as a basis of production of the new biopolymer products with interesting peculiar properties that differ considerably from the properties of individual components. Modification of gel-formation properties of protein and, therefore, composition, structure, rheology and other physical and chemical properties of gels on its basis can be provided by complex formation of the protein macromolecules by polysaccharides. The complexes of gelatin with ionic polysaccharide can be considered as a new type of gel formers and the base for food gels with various properties. The aim of the work reported was to carry out a rheological study of biopolymer aqueous mixtures (sols and gels) containing gelatin and anionic polysaccharide sodium alginate, carrageenan and cationic polysaccharide chitosan. Polysaccharide has been introduced into mixed systems at the level at which one itself has no gelling properties but where it has a positive synergistic effect on the gelling behavior of the mixed system. The effect of component concentration (mass ratio), pH, temperature and time-dependence has been studied. The results of rheological measurements within the non-linear viscoelasticity range have been fitted using Hershel-Bulkley model and Casson model. The analysis of the rheological curves allows characterizing the gelatin gels of low concentration with polysaccharide as viscoelastic-plastic material. Ionic polysaccharide additions give non-monotonous change of the rheological parameters (plastic viscosity, the yield stress, index of flow) with clearly expressed extreme points for the multicomponent gels in comparison with the gelatin gels; velocity of three-dimensional structure formation increases, the yield stress rises. Using the methods of light dispersion and thermochemistry interactions between gelatin and ionic polysaccharide in aqueous systems has been investigated that result in microphase separation – formation of high-dispersed particles of the interpolymer complexes. The maximum strength of gelatin-polysaccharide gels, apparently, is connected with formation of both the gel network by the complexes of gelatin with anionic polysaccharide and the maximum number of salt bindings. Relationship between rheological behavior and structural features (method of scanning electron microscopy) are being studied for the mixtures of protein and polysaccharide. This work is supported by the Russian Foundation for Basic Research (project 10-03-00310-a).

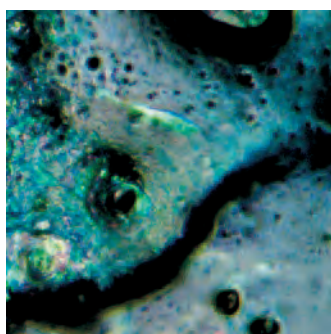
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Colloidal
Systems

Edible supramolecular chiral nanostructures by self-assembly of an amphiphilic phytosterol conjugate

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Phytosterols are sterols extracted from plants, and have structures similar to that of cholesterol. They have been proposed as a natural source for the reduction of both cholesterol in blood plasma as well as cholesterol low-density lipoprotein, and recommended as a dietary supplement. Besides regular phytosterols, conjugated phytosterols are also present in plants as fatty acids, acylated glycosides or glycosides derivatives. In this work, we discuss for the first time the spontaneous self-assembly behaviour of a conjugated glucose-sitosterol (β -sitosterolin), synthesized in our laboratory, and analogue to that found in several natural products. We rely on small and wide angle X-ray scattering (SAXS and WAXS) and on atomic force microscopy (AFM) for the study of the complex architectures of the colloidal objects based on supramolecular β -sitosterolin self-assembly. The self-assembly of a phytosterol conjugate (β -sitosterolin) has been investigated in bulk, in molecular solution and in the presence of a selective solvent. Depending on the quality of the selective solvent, the molecule aggregates into molecular bilayers, in which the moieties facing the solvent can be either the glucose or sitosterol units, depending on whether water or alcohol is used as solvent, respectively. In the first case (water), platelet-like structures with thickness equal to an integer multiple of the bilayer period are formed, which upon dilution transform into left-handed helical ribbons, whose supramolecular chirality is induced by packing of the sitosterol moieties. In the second case (alcohol), spherical-like objects much smaller in size are formed, as a consequence of the high volume fraction of the sitosterol moieties as opposed to that of glucose. These findings may both contribute to the understanding of supramolecular chirality in the self-aggregation process of chiral molecules, as well as to provide an appealing new system for the design of edible fibrils with desirable nutritional components.

The effect of heat induced denaturation of whey proteins on the rheological and colloidal behavior of concentrated milk

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During heat treatment, the whey proteins denature and aggregate with each other and with κ -casein. The pH of the milk before heating is an important factor determining the type and composition of the heat-induced complexes in the serum phase. The objective of this work was to determine if changes in the serum composition because of heating will affect the colloidal behavior of the casein micelles in concentrated milk. To better understand this behavior will allow for a better design of concentration processes. Milk was concentrated using osmotic stressing, as this non-invasive method allows investigating the effect of casein micelles volume fraction without altering the ionic balance in the serum phase and without applying shear to the protein particles. The viscosity of casein micelles was measured as a function of volume fraction. In unheated milk, the increase in viscosity with volume fraction was well predicted by the Eilers equation. On the other hand, the viscosity of the heated milk varied significantly from this semi-empirical model. The radius of the casein micelles, measured by light scattering, varied depending on the initial pH of the milk at heating. Diffusing wave spectroscopy parameters, namely the diffusion coefficient and turbidity parameter ($1/l^*$) clearly showed that in unheated milk the casein micelles behaved as hard spheres up to a volume fraction of about 0.32. At higher volume fractions, their mean squared displacement and $1/l^*$ indicated arrested motion and increased interactions. The critical value of volume fraction was lower in the case of heated milk. The behavior of heated milk is ruled by the presence of heat induced aggregates and it is dependent on the ratio of the aggregates present in solution or on the surface of the casein micelles. This research brings new insights on the interactions occurring in milk proteins during concentration, and on the effects of processing history on structure-function of casein micelles.

Monitor kinetics in soft porous heterogeneous materials using FRAP

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Diffusion is vital for many food properties such as propagation of water into pasta during cooking, protection of sensitive food substances like vitamins, antioxidants and minerals during storage, oral taste release and water management in pastry products. These examples show that it is important to have good control over the diffusion properties in order to obtain desired functionality. Therefore thorough understanding of structure - mass transport relationships and good measurement techniques are essential. In this talk, the coupling between structure and diffusion in soft porous heterogeneous materials will be exemplified and discussed. Confocal laser scanning microscopy (CLSM) in combination with Fluorescence recovery after photobleaching (FRAP) is a versatile method to determine quantitative diffusion properties locally directly in the microscope. It can be used in many types of soft porous homogeneous and heterogeneous biomaterials with complicated structures and many length scales present. In FRAP, part of the fluorescently labelled molecules of interest are deactivated in a certain region using photobleaching. Directly after the bleaching, unbleached molecules from the area outside the bleached region start to diffuse into the bleached region and simultaneously, bleached molecules start to diffuse out. The rate of intensity recovery is proportional to the diffusion rate. If appropriate experimental conditions and models are used, qualitative data such as the diffusion coefficient and interaction parameters can be estimated. Recently three new advanced FRAP models [1-3] have been developed based on a new pixel-based framework that effectively utilize all available spatial and temporal data in the FRAP data. This new framework opens up possibilities to estimate the diffusion coefficient with good precision and error estimates in arbitrary geometries with low amount of bleaching. Here, the new pixel-based models will be presented. Food properties changes as a function of time and surrounding conditions. CLSM-FRAP combined with stages to control the surrounding conditions like temperature is a powerful technique to monitor the effect of kinetics on the diffusion properties. In this talk, the possibilities of CLSM-FRAP and the new pixelbased models to quantitatively monitor diffusion during fat crystallisation in chocolate [4], gelatine gelation [5] and swelling of superabsorbent polymers [1] will be shown. It has been found that tempering and solid particle additions have a large impact on the time-dependent fat migration in chocolate model systems⁴. CLSM-FRAP combined with rheology and experimental design has shown strong indication of formation of transient structures during gelation of gelatine gels [5].

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Understanding and tailoring the flow properties of biopharmaceutical formulations: The influence of the interface

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Monoclonal antibody (mAb) therapeutics for subcutaneous administration are often formulated as highly concentrated solutions (> 50 mg/ml). Under such molecularly crowded conditions limited stability and pronounced viscoelasticity can arise, constituting challenges for both manufacture and drug delivery. This project aims to better understand how molecular interactions in such protein solutions govern their bulk flow properties. In particular we will consider the often neglected impact of the air-water (a/w) interface on these properties and hence the ability to form a viable formulation. We have investigated flow properties of β -lactoglobulin (β -LG) solutions as a model surface active biomacromolecule. Interfacial and bulk rheology data have been acquired for β -LG solutions (0.1 - 60 mg/mL, at pH 6.0, 10 mM Histidine), and related to surface tensiometry data. Preliminary results indicate that the pronounced viscoelasticity observed at low shear rates (< 1 1/s) arises from its adsorption at the a/w interface. This influence can be related to the molecule's stability in solution. Ongoing studies are exploring the effects of adding a range of surface active brush-like polymers based on poly(poly(ethyleneglycol methacrylate)s to the β -LG solutions, as possible excipients to reduce viscosity to tractable values at both low and high shear rates.

Surface rheological properties and adsorption behavior of protein fibrils and protein-polysaccharide complexes at liquid-liquid and solid-liquid interfaces

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Protein fibrils and protein-polysaccharide complexes are surface active, and can be used as emulsifiers, foam stabilizers, or encapsulating materials. Knowledge of the adsorption behavior of these materials on solid-liquid and liquid-liquid surfaces is important for developing new applications such as highly stable emulsions or encapsulation systems. In this study we have investigated the adsorption of two types of fibrils from lysozyme (long semi-flexible fibrils and short rigid fibrils), fibrils from ovalbumin (short and flexible), lysozyme-pectin complexes, and ovalbumin-pectin complexes, at oil-water interfaces, using ellipsometry. We have also characterized the surface dilatational and surface shear rheological properties of these interfaces, using an automated drop tensiometer, and a stress controlled rheometer with biconical disk geometry. We have also studied the multilayer adsorption of these materials at solid-liquid interfaces, to characterize their effectiveness as building blocks for multilayer encapsulation systems. The properties of the adsorbed layers (thickness, density, and distribution) were determined using reflectometry, ellipsometry and AFM. We will discuss the effect of the properties of the fibrils (length, flexibility), and complexes (size, charge distribution) on structure and rheological properties of the interfaces, and on their effectiveness as stabilizers for emulsions, foam, or encapsulation systems.

Influence of the counter-ions sodium or calcium on the rheology of caseinate suspensions

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The viscosity and frequency dependent shear moduli of dense calcium caseinate suspensions were investigated as a function of the concentration and the temperature at pH 6.7. The results are compared with a similar study on sodium caseinate. Both systems form viscoelastic suspensions with a viscosity that increases strongly with increasing concentration and decreasing temperature. An important effect of the counter-ion on the viscoelastic properties was observed, which is attributed to the specific interaction of Ca^{2+} with casein.

Probing the diffusional and structural properties of casein micelles dispersions through Fluorescence Recovery After Photobleaching (FRAP): From the dilute to the highly concentrated/crowded regime

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Casein micelles are one of the most important products in human diet. These roughly spherical protein assemblages of ~ 120 nm contain ~ 80 % of water and share many similitudes with 'conventional' synthetic microgel particles. SAXS/osmotic pressure and rheological studies have reported on the rheological and phase properties of micelle dispersions at extremely high concentrations [Bouchoux et al. 2009 & 2010]. In this paper, we investigate the diffusional properties of similar dispersions and we attempt to correlate those properties with features of the micelle internal structure. FRAP was used to measure the diffusion of tracers of different sizes and shapes (globular (FITC-proteins); 'reptoidal' (FITC-dextrans)) in dispersions of 'native' casein micelles of concentrations 150 - 450 g/L, i.e. from the liquid to the solid-like state. In order to investigate the effect of structure and interactions on diffusion, experiments were also performed at high ionic strength and with caseins, not organized into micelles (caseinate). FRAP was appropriate for the measurement of diffusional properties of concentrated casein dispersions. In the 'native' state, the decrease in the tracer's diffusion with casein concentration follows three regimes:

- 150 - 210g/L, before micellar close-packing: diffusion takes place only in the space between micelles. Diffusion depends solely on the size and not on the shape of the tracers.
- 210 - 330g/L, at close-packing: there is no more space between micelles and diffusion takes inside them. The tracers show a behavior that can be described through the Rouse/reptation models; meaning the dextrans are able to deform to go through the casein network, while the proteins (much less deformable) are progressively blocked into it. In this regime, the equivalent porosity and tortuosity of the system as a function of concentration can be estimated through the diffusion data.
- 330 - 450g/L, micellar squeezing: diffusion still takes place into the micelles, but the network is now so tight that no correlation between tracer's size and diffusion exists. Comparison with data obtained with caseinate in the same concentration suggests that diffusion is now governed by the internal dynamics of the micelle.

Our results bring new quantitative information on casein gels structure and further substantiate the on-going discussion of the diffusion driving parameters in very concentrated systems.

Characterization of emulsifier's adsorption on solid particle surfaces in lipophilic suspensions by AFM

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Chocolate masses are examples of confectionery suspensions where polar solids like sucrose, milk powder and cocoa particles are dispersed in a lipophilic phase of cocoa butter. In these suspensions, sucrose is of special interest because sugar crystals have areas of different surface polarities due to grinding in waterless environment. These are caused by amorphous and crystalline structures. Free surface energy contributes to aggregation of solid particles and may induce phase separation of the suspension. Part of the continuous phase is immobilized in interparticular spaces between aggregated particles resulting in increased viscosity and a yield value can be detected. With respect to chocolate processing, the suspension's flow behavior is of outstanding interest. Besides agglomeration, flow properties are also affected by multiple influencing factors like volume fraction, shape, surface topography of solids and their energetic interactions with the continuous liquid phase. To control these interactions, emulsifiers are added. They enhance wettability of the dispersed particles and facilitate their distribution in the continuous phase. The effects of emulsifiers in lipophilic suspensions have been well-known for years. It is assumed that they cover particle surfaces and reduce immobilization of cocoa butter at the solid surfaces. However, considering the differences in polarity of the sucrose particles after grinding, the spatial distribution of emulsifiers on sucrose particles and its effect on suspension properties is not known so far. More detailed information about the interactions of emulsifiers and sucrose surfaces can be obtained by atomic force microscopy (AFM). Therefore, AFM-techniques were applied to investigate different surface properties before and after adsorption of phospholipid fractions. In particular, topographical imaging, force-distance-curves and surface potential measurements were carried out. In addition, adsorption of fractions of phospholipid blends in these suspensions was quantified and related to the sugar surface area. In this presentation, results concerning possible relations between surface polarities of solid particles, emulsifier's adsorption in lipophilic suspensions and effects on flow behavior will be shown and discussed. The AFM-data will be correlated with results from rheological measurements considering particle size distribution. In this way, microstructural properties can be transferred into macroscopic scale.

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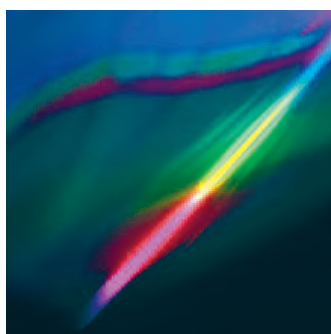
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Rheo-SALS, SANS, SAXS

Combined methods in Rheology: Rheo-SAXS, Rheo-NMR, and Rheo-Dielectric to bridge length and time scales

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Rheology as the science of flow and deformation of matter measures in general forces (torque and normal forces) and displacement of materials. Regularly rheology wants to establish the interplay of molecular structure and mechanical properties. Both quantities often differ in their internal length scale starting from 10^{-9} to 10^{-2} m and time scales covering 10^{-8} to 100 s. Furthermore mechanical deformation e.g. shear can lead to oriented structures or crystallization if non-linear shear is applied. Consequently there is a need to conduct in-situ molecular characterization techniques during rheological measurements if non-linear shear, e.g. large amplitude oscillatory shear (LAOS) in combination with FT-Rheology is applied. Within this presentation we would like to present three recent developments within our group to investigate short length scale dynamic measurements ($< 1 - 2$ nm) via rheo-NMR, molecular size (R_g) measurements (ca. 10 - 50 nm) and structural correlations via Rheo-SAXS measurements. The experimental realization and first experiments will be presented in detail.

Shining light on the microstructure of frozen food: Using X-ray phase contrast tomographic microscopy on ice cream

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The sensory perception of food is determined by its microstructure which in turn is influenced by its ingredients and their processing. The interactions within this complex system are not yet completely understood. This is due to the currently used imaging techniques that cannot provide direct information on the microstructure without destroying the sample. Synchrotron tomographic microscopy can fill this gap and provide three dimensional information in a direct and non-invasive way at a high spatial resolution. Propagation based phase contrast imaging (PCI) allows the differentiation of similarly absorbing materials like sugar solution and ice crystals without the addition of contrast agents. A new sample environment makes in-situ experiments under controlled thermal conditions in a subzero degrees Celsius regime possible and for example allows for time lapse studies or the investigation of heat shocks. Here we present how PCI can non-destructively reveal internal microstructures of ice cream and pave the road for a better knowledge of the fundamental physics behind coarsening and other microstructure evolutionary effects.

Stabilization of emulsions and foams: Probing interfacial properties by small angle scattering

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Adsorption of proteins at interfaces is important for the stabilization of emulsions or foams but is detrimental for pharmacological formulations. The amount and nature of the protein adsorbed to an interface is decisive for the interfacial film properties [1, 2]. In this study we focus on a set of recombinant proteins called designed ankyrin proteins (DARPs) [3, 4]. We aim to establish the link between protein structure and the resulting morphology and mechanical properties of the protein adsorption layer. The modular construction of the DARPs allows a polymer-like extension of a protein with the same building block and a controlled adjustment of its bulk and interfacial properties. With small angle scattering techniques and interfacial rheology we want to correlate protein size and stability to the resulting adsorption layer morphology, layer viscoelasticity, and single capsule mechanics.

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Viscous heating in a mini-Couette cell used in Rheo-XRD and Rheo-NMR research with a non-Newtonian standard fluid

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A mathematical model is proposed to describe the temperature increase due to viscous heating in a mini-Couette cell used in Rheo-XRD and Rheo-NMR experiments on crystallization of triglycerides. The mini-Couette cell cannot satisfy the small gap approximation, due to the small radius and the need to have enough sample in the gap to measure its XRD and NMR signals. The nanocrystalline suspensions formed during crystallization are non-Newtonian and shear thinning within the ranges that have been so far explored. The model was tested using a standard non-Newtonian rheological oil, NIST standard reference material (SRM) 2490. The model was developed using the shear rate dependent Cross constitutive viscosity equation of the oil, modified to be a function of temperature. The constitutive equation was used in the momentum transport differential equation. It was also used in the heat transport equation in the viscous heat generation term. The model was cast into functions of the non-dimensional temperature and non-dimensional velocity of the oil, and their derivatives. Numerical solutions for the four variables were obtained for the boundary conditions of the mini-Couette cell. A first set of solutions were sought where the heating effect could be considered negligible and the fluid remained isothermal. The temperature and viscosity behavior of the oil was then analyzed under high shear rate, to study the viscous heating effect of the oil. The experiments were conducted in the cell driven by an Anton-Paar DSR 301 rheometer, at rotational frequencies between 0.05 and 50 rps. At low rotational frequencies a narrow zone of fluid near the shaft accounted for most of the dissipated energy, whereas the external region remained quasi-stationary. As the shear rate was increased, the inner sheared region extended farther from the shaft. For a particular rotational speed, the fluid displays a broad range of shear rates. In the case of large rotational frequencies, the temperature at the shaft is considerably higher than at the external wall. The model developed in this work is essential for the interpretation of viscometric experiments done under shear flow in small-enclosed cells with high curvature of non-Newtonian materials. It also helps with the understanding of the crystallization behavior of mixtures of triglycerides, and with the calibration of the rheometer under high shear rate conditions, as well as for the design of industrial shearing crystallizers.

New insights into starch gelatinisation: Simultaneous small-angle neutron scattering and rapid-visco analysis

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The technique of Rapid viscoanalysis (RVA) is widely used in the grain industry as a standard method for determining the pasting properties of starches; however little is known about structural changes that occur during various standard protocols. Small angle neutron scattering (SANS) has several key advantages over x-rays for characterising food systems at nanometre scales, such as high penetration through dense preparations and sample environments as well as the ability to limit radiation damage. Simultaneous SANS and RVA measurements were performed on the SANS instrument, Quokka, at ANSTO on a variety of commercial starches. A commercial RVA instrument was modified to include suitable apertures for the neutron beam through the heating block and a modified paddle assembly. Samples for RVA were made up at a composition of 3 g starch in 47.8 g of deuterated water. A standard 13 minute profile was used with increased paddle speed to avoid settling of the mixtures. SANS measurements were made at 1 minute intervals throughout the RVA process across a q range of 0.018 - 0.2 Å⁻¹. In each of the starches, the well-known lamellar structure is observed up to the point at which the viscosity begins to increase markedly. At this stage, the lamellar structure transforms almost instantaneously, the scattering patterns changing to that of a large scale structure with no apparent semi-crystalline properties and whose structure is analysed in terms of a fractal like gel. The basic building blocks of this gel, under the assumption that they are spheroids, appear to have dimensions of ca. 1 nm across all the starches tested. The sizes of the clusters they form are several times larger, vary across the time course of the experiment and have aggregate sizes showing considerable botanical variation.

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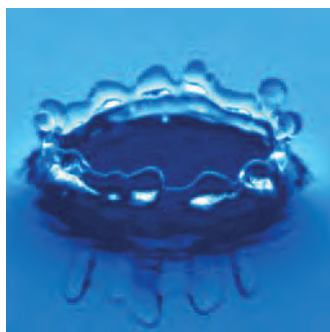
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Influence of Processing on Structure and Rheology

Potential of food structuring to improve texture of high-protein food and to initiate novel food structures

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Whey protein is often considered as an important protein source to develop high-protein foods. Some studies indicated that a higher intake of protein might maintain muscle mass in elderly (Campbell & Leidy, 2007; Kurpad & Vaz, 2009) and facilitates body weight control (Leujene, Kovacs & Westerterp-Plantenga, 2005). Unfortunately, a product that contains a high concentration of protein is often too firm, has a rubbery mouth feel, and hardens in time. A possible solution could be to use dispersed whey protein in high-protein food. This can be obtained by structuring the protein with a microparticulation process, resulting in whey protein microparticles. The process starts with heating while mixing a 40 % w/w whey protein isolate (WPI) dispersion, which leads to fractured gels. These gels are dried, giving protein granules which are milled to the desired particle size. Replacement part of WPI by WPI microparticles resulted in a softer protein gel at the same total protein concentration. A dispersion that only contained WPI microparticles did not form a gel upon heating. Flow-induced structuring was used as a tool to further structure WPI microparticles aiming at novel, anisotropic structures. A dispersion of 5 % w/w WPI-microparticle in 0.5 % w/w locust bean gum - 0.5 % w/w xanthan gum mixture was subjected to simple shear flow generated by an in-house shearing device. This closed system was operated at 80°C. After quenching the temperature to 10°C, the particles showed a certain degree of alignment with the flow. The reason for this alignment is not yet clear, however, it could be speculated that particle interactions or depletion interactions might play a role. In conclusion, structuring of protein into microparticles is a promising method to develop high-protein foods with an altered texture. A further structuring step of protein with shear flow is still in its infancy though it gives promising results. Therefore, a further exploration is needed, especially at high protein concentration. Nevertheless, this step might lead to creation of novel food structures for e.g. meat-like structures.

Effects of high pressure on the rheology of mozzarella type curd made from buffalo and cows' milk

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Effects of high pressure (HP) treatment on the rheological behavior and physico-chemical properties of mozzarella type curd made from skimmed buffalo and cows' milk were studied. The curd firmness and gel strength were decreased with increase of the pressure treatment for both milks. The concentration of denatured β -lactalbumin (β -la) and β -lactoglobulin (β -lg) were increased with increasing the pressure in both bovine milks. This may affect the firmness and gel strength of mozzarella curd made from both pressure treated milk samples. This may be due that the denatured whey proteins attached to the casein micelles which resulted in hindering of the rennet induced curd formation process. The concentration of denatured β -lg A was found to be higher than that of β -lg B in high pressure treated cows' milk samples, while buffalo milk contained single variant of β -lg protein. The casein micelles sizes were also reduced with the increase of pressure treatment in both milk samples. Therefore, the curd rheology was adversely affected at higher pressures due to the disruption and deformation of casein micelles. The optimum curd firmness and gel strength was obtained at the pressure of 50 MPa in both curd samples. Ionic calcium was found to be not significantly different in all high pressure treated milk samples. The overall dynamic curd rheology and physicochemical changes were found to be more pronounced in cows' curd than those made from buffalo curd at similar pressure treatment. This may be due to differences in sizes of casein micelles, casein and whey protein fractions of both milk samples.

Characterization of the resistance of a Swiss-type pressed cheese under stress as regards to the organization of the curd grains

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The objective of the present study is to follow the evolution of a Swiss-type cheese's paste when put under stress up to rupture, from both a microstructural and a rheological point of view, in order to achieve a better understanding of the cracks' formation in Swiss-type cheese during ripening and storage. Binocular observations (x5) and some confocal laser scanning microscopy (protein and fat labeling, X 10 and X 63) were carried out. Cheese paste showed an anisotropic organization of the pressed curd grains depending on their position regarding to the press axis. They had an ovoid shape with a principal axis orientated perpendicularly to the press direction. Borders of the curd grains showed a locally higher protein concentration. That is why their mechanical properties are probably different from the ones of the protein network inside the curd grains. Stress-strain curves of cheese's samples from small to high level of strain were carried out. Samples of ripened commercial Swiss-type cheese (fat/dry 47 %, water content 42 %) were studied using a MCR 301 rheometer (Anton Paar, Austria) used in a texturometer mode. The crosshead was driven in a decreasing speed mode ($dH/Hdt = \text{constant}$) in order to ensure a constant biaxial extensional rate in the cheese sample. These experimental conditions added to the lubricated conditions of the tests represented a Lubricated Squeezing Flow. It was chosen in order to mimic the real conditions of eyes growth in cheese. Tests led at different constant biaxial extensional speeds from small to high level of strain allowed the determination of threshold values on the stress/strain curves. These two noticeable points were the yield stress and the rupture point, where values of stress and strain could be found. They were used as rheological indicators in order to test the cheese paste anisotropy effect on its rheological properties at large deformations. It appeared that the rupture strain depended on the direction of sampling, in good accordance with the anisotropic organization of the curd grains in the cheese.

Impact of different technological treatments on structural and rheological properties of a cream cheese model

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Cream cheese is an oil-in-water emulsion, usually with high-fat content (33 %), acidified and then textured by heat treatments and homogenization. The aim of this study is to investigate the impact of different heat treatments, acidification kinetics and homogenization pressures on structural and rheological properties of cream cheese. Two heat treatments were studied to assess the impact of whey proteins aggregation on the structural and rheological properties of the emulsion and the final cream cheese. A high heat treatment (94°C/40 s) resulting in a high whey protein aggregation (55 % aggregated whey proteins) causes an increase in the emulsion viscosity. On the contrary a low heat treatment (72°C/20 s) does not lead to an aggregation of the whey proteins (< 5 % of aggregated whey proteins) and to an increase of the viscosity. The thermo-denatured whey proteins aggregates bind to fat globules which apparent volume increases, resulting in an increase of emulsion viscosity. In addition, two acidification kinetics were studied to assess the impact of the final pH on the structural and rheological properties of the acidified emulsion. Emulsions acidified until pH 4.9 tend to be more viscous than those acidified until pH 5.2. The decrease in pH induces demineralisation of casein micelles and thus casein dissociation and leads to the formation of protein network. This promotes an increase in emulsion viscosity. Moreover, acidified emulsions beforehand treated at 94°C are firmer than those treated at 72°C. Aggregated whey proteins participate in the protein network and strengthen it. Finally, four homogenization pressures were applied to the acidified product: 0, 5, 20 and 60 MPa. The increase in homogenization pressure induces decrease of fat globules size. The increase of fat surface promotes interaction with the protein network and leads to an increase the final cream cheese firmness. However, high homogenization pressures reduce firmness differences beforehand induced by the other technological treatments (heat treatment, acidification).

Rheological characterization of Mozzarella cheese curd with a capillary rheometer

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Mozzarella cheese production is estimated to be 2 million tones per year. Production equipment is continuously evolving to satisfy the increasing demand. For proper equipment design rheological characterisation of the cheese curd is important. At the processing temperature of 55 - 65°C the mozzarella curd is a viscoelastic material. Capillary rheometers have been already discussed in the literature to define the mozzarella cheese rheology [1, 2]. In our study we applied a capillary rheometer to determine the properties of mozzarella cheese curd. We observed reproducible flow curves at low shear rates. At higher shear rates a novel, to our knowledge not yet described in food science, flow property was observed. Surface instabilities were observed when exceeding specific shear rates dependent on temperature. Periodical pressure oscillations were measured and sharkskin formation (melt fracture) has been observed at the die outlet. This effect is already known in polymer science, and is related to a transition phase between wall adhesion and wall slip. In our case it was not a transition phase. Increasing the shear rate excessively did not result in a smoothening of the measured pressure. No steady state could be reached with wall slip. The aligned protein fibers are destroyed with the abrupt flow changes and an unwanted rough surface is created. This fact has to be considered when designing mozzarella production lines. A critical shear rate should therefore not be exceeded to provide the process-structure relationship. At elevated temperatures also an excessive separation of fluid from the mozzarella curd was observed. The fluid generated pockets, which disturbed the measurements, are in accordance with the literature where increased temperatures are associated with higher fat losses. The capillary rheometer was shown to be a useful tool to study mozzarella cheese curd under manufacturing conditions.

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Thermo-rheological analysis of protein-fat systems by applying temperature sweep experiments to grated and non-grated hard and semi-hard cheese varieties

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Meltability is a functional property whose valuation is essential for the use of cheese as ingredient in cooking meals and on which consumers preference and acceptance depend to a large extend. The temperature-sweep experiments have been shown to be a reliable method in terms of obtaining thermo-physical data of protein-fat systems and to predict melt behavior of cheese. However, methods to gain thermo-physical properties of ready-to-consume grated cheese, which has become a popular convenient food and can be found in frozen pizza and pasta dishes, are scarce and we are not aware of a study whose aim the analysis of grated cheese samples by temperature-sweep experiments was. Thus, objective of this study was to adapt the temperature-sweep method to the matrix of the protein-fat system grated cheese. Therefore, grated cheese was melted in a home-made device at 50°C. Subsequently, the so formed sample was placed in the rheometer and analyzed by temperature-sweep experiments. For purposes of comparison each cheese (three hard cheeses and three semi-hard cheeses) was analyzed in grated and non-grated form. The temperature-sweep method seems promising in terms of providing reproducible thermo-rheological data of grated cheese varieties, even though significant differences between some of the grated cheese samples were observed. Furthermore, the sample preparation (melting and slight pressing) had significant impact on the rheological properties in comparison with the non-grated cheese samples. The storage (G') as well as the loss (G'') modulus at 20°C decreased significantly for the grated cheese samples. Moreover, the softening point was reached at a significant lower temperature. During sample preparation, the cheese fat melted ($> 40^\circ\text{C}$) and coalescence of the fat globules occurred which, after resolidification, led to a less homogenous cheese body. As a consequence the protein network lacked the structural support of the fat globules and faster softening at lower temperatures occurred. The structural alterations during sample preparation and resolidification were confirmed by confocal laser scanning microscopy (CLSM). Further research in terms of correlation the rheological data with the chemical composition might clarify the occurred differences between the grated cheese samples during thermo analysis.

Dense whey protein particles for development of high protein foods

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Recently it has become clear that food products with high protein content offer various health benefits and give rise to a stronger feeling of satiety. However, in developing novel high protein food products it has been found that maintaining an optimal food structure is difficult, mainly as a result of protein aggregation. We investigate one possible route of uncoupling protein content from its effects on food structure: the use of prefabricated high protein structure elements with controlled interactions. We developed a robust procedure for preparing dense protein particles having an average size of a few microns, and report on the heat stability of concentrated dispersions of these particles. The particles, with a high internal protein content (~ 20 %w/w), were formed through emulsification of whey protein isolate solution in sunflower oil containing an oil soluble emulsifier, Polyglycerol Polyricinoleate. The emulsion was heated to induce gelation of whey protein inside the particles, and oil was removed in a series of washing steps. Protein particles were dispersed in different aqueous phases (solutions of sodium caseinate, whey protein isolate or gum arabic, volume fraction of particles approx. 35 %). We have analyzed the flow behavior, microstructure and particle sizes after heating the dispersions for 30 min at 90°C. All dispersions were liquid-like and no significant changes in the microstructure of the particles were recorded after heat treatment. A small shift in the particle size distribution towards larger sizes was noticed in some of the dispersions. When 1 %w/w gum arabic was used as stabilizer no change in the viscosity was observed after heat treatment, whereas when Na-caseinate or whey protein isolate solutions were used the low-shear viscosity of the particle dispersions increased and shear-thickening was observed in high shear regime. In conclusion, the use of prefabricated protein structure elements, in this case dense protein particles, has been shown to be a useful strategy to combat aggregation problems in high protein foods.

Microfluidization of milk significantly improves creaminess perception in yoghurt

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The impact of microfluidization of milk on microstructure, rheology, particle size, water retention and sensory properties, in particular creaminess, of yoghurt was studied. Microfluidization (25 - 150 MPa) of low-fat (1.5 %) milk produced yoghurts with improved water retention, texture (viscoelastic properties and viscosity) and creaminess compared to those made with conventionally homogenized (20/5 MPa) milk. A negative effect on sensory quality was seen in yoghurts made with microfluidized non-fat milk, indicating that some fat is essential for improved texture and perception of creaminess. Low-fat yoghurts with creaminess and texture superior to those of full-fat (3.5 %) conventionally homogenized yoghurts were produced by microfluidization at pressures ~ 50 MPa. Descriptive sensory analysis of the yoghurts indicated that the most important of a total of 33 descriptors for creaminess were: cohesiveness, thickness, and oral and spoon viscosity. Confocal microscopy of yoghurts made with microfluidized milk indicated a major reduction in size ($< 0.5 \mu\text{m}$) of fat globules, which were homogeneously incorporated within the protein aggregates. High-resolution scanning transmission electron microscopy of these yoghurts revealed the presence of distinct rounded particles or nanocomposites approximately 50 - 300 nm in diameter, consisting of extremely small (10 - 100 nm) fat globules encapsulated in thin layers of protein. These nano-composites of fat and protein were not seen in the conventionally homogenized yoghurt and it is hypothesized that these nanostructures may promote creaminess, possibly due to substantial increase in numbers of small fat globules that increase lubrication and viscosity, while creating a mouthcoating effect. In conjunction with microfluidization, creaminess and textural properties were further enhanced by heat treatment of milk. Prediction models based on multivariate analysis revealed that the desirable texture and creaminess in yoghurt was influenced by fat content and microfluidization pressure.

Particle formation of whey protein by extrusion cooking

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Whey proteins can be specifically modified to change their functional properties by controlled denaturation and aggregation. This particle formation can be achieved by mechanical and thermal treatment. There are different options to realize this combined heating and shearing process: in a scraped surface heat exchanger or in a tubular heat exchanger followed by homogenization or by extrusion. Using extrusion for the thermomechanical modification of the proteins has a series of advantages against other, e.g. the possibility to alter the process parameters in a wide range and to implement higher viscosities and therefore higher protein concentrations (up to five-fold). In the presented work, the influence of process parameters on both size and structure of the particles is investigated. Those process parameters are mainly the temperature, the residence time, the shear rate, the feed rate and the protein concentration. The size of the aggregates is measured by laser diffractometry, the degree of denaturation by RP-HPLC and the structure is investigated by optical microscopy and serum binding ability. RP-HPLC methods show that the combination of temperature and residence time determines the degree of denaturation of the whey proteins. The temperature has a strong impact on the denaturation kinetics. High temperature and longer residence time cause higher degrees of denaturation and larger particles. The feed rate has a strong impact on the heat transfer in the solution and the residence time distribution. Thus, the degree of denaturation increases with decreasing feed rate. The protein concentration determines the rheological properties of the solution and significantly influences the residence time and the shear rate. Since shear also depends on the screw configuration and the screw speed it cannot be investigated separately. By rotational measurements in a rheometer, the impact of shear rate and heating time was therefore investigated separately. The formation of the protein aggregates can be followed by the change of the viscosity during the heating and shearing of the protein solution. Depending on the protein concentration and the shear rate, either an increased aggregation due to increased collision rate or disaggregation is the main determinate of the process.

Characterization of the structural properties and stabilizing forces in heat induced gels of whey protein fractions in presence and absence of high shearing

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Gels from 10 % protein solutions of isolated α -Lactalbumin (a-La) and β -Lactoglobulin (b-Lg) as well as native mixtures (ratio a-La:b-Lg of 20:80) of both proteins were produced after heating the solutions at 90°C in absence and presence of shearing until a denaturation degree higher than 90 % was reached. Gel properties (G' and G''), serum binding and textural properties were analyzed in samples obtained without shearing after variation of lactose level (1 - 13 %) and pH (3 - 8). Weaker gels were formed in solutions containing a-La as well as low pH and low lactose concentration. Serum binding was higher as the lactose levels increased. A complete characterization of the binding forces (covalent, hydrophobic, electrostatic, calcium and hydrogen bonds) was conducted, showing that stronger gels with lower serum binding were obtained in conditions with dominating covalent bonding (i.e. higher pH and presence of b-Lg). The influence of shearing was studied using the same solutions and heating regime in a scraped surface heat exchanger in order to determine the role of binding forces and composition in the formation of microparticles of denatured whey protein with defined particle sizes and viscosity. High lactose contents and low pH were required in order to become microparticles with $d_{50,3} < 20 \mu m$ and high creaminess independent of the whey protein composition. The results are of high relevance for the production of gels with tailored properties as well as highly functional microparticles from heat denatured whey protein with excellent sensorial properties.

Comparisons between membranes for use in cross flow membrane emulsification

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Membrane emulsification is a technique that is generally seen as a method of producing low polydispersity emulsion droplets, however in the area of food production perhaps its main advantage is low processing shear. A series of experiments were undertaken to compare the performance of membrane emulsification with different membrane materials and morphologies. The effects of the different properties of the membranes were investigated in a cross-flow membrane emulsification device when making simple oil in water emulsions. The differences in the performance of four membrane materials (porous glass, ceramic, polymer and stainless steel) were investigated, and the effects of the various operating variables of the emulsification system were determined. The membranes used were all tubular and of 10 mm outside diameter, allowing their use in the same membrane emulsification system and with the same operating geometry. The shear produced by the flowing continuous phase of the emulsion detaches the forming droplets of dispersed phase from the surface of the membrane to form an emulsion. Under different hydrodynamic conditions the prevailing detachment mechanism can be due to the cross flow shear, or the dispersed phase pressure. The morphologies of the different membranes cause variations in the droplet detachment regime; the support material of the ceramic membrane causes the droplets size to change less with changing shear, and more with changes in pressure than with the porous glass membrane, for example. The low shear nature of the resultant emulsification process lends itself to the rapid production of carefully controlled emulsion structures depending on the materials being emulsified. Structures such as double emulsions where the internal dispersed phase is kept separate from the external continuous phase throughout processing has useful applications within the food industry in areas such as flavor release and encapsulation of healthy ingredients. This effect is shown using confocal microscopy to dye the internal dispersed phase of a double emulsion. The resultant emulsion shows no un-dyed droplets in the internal dispersed phase, and no detectable dye in the external continuous phase. The performance of the membranes making gelled particles was also investigated, and the stainless steel membrane showed the most favorable results as the pores are straight and perpendicular to the surface they are less likely to foul during gelation.

Concentration of milk by membrane filtration modifies the acid induced gelation properties of casein micelles

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Membrane filtration is a widespread unit operation in dairy technology, and little is understood on how concentration by ultrafiltration and diafiltration (which implies the addition of water to the retentate) may change the physico-chemical properties of the casein micelles. In order to provide important insights into a comprehensive understanding of the effect of concentration by ultrafiltration or ultrafiltration and diafiltration, this study investigated the colloidal properties and behavior of concentrated milk during acidification. The results indicated that during concentration, the amount of colloidal calcium increased but not proportionally to the volume fraction of concentration, suggesting that at the high concentrations (4x) the colloidal calcium phosphate in the micelles decreased. This was consistent with the observations in the buffering capacity peak by titration with HCl. The research also brought evidence of compositional changes in the serum fraction during diafiltration. This serum fraction contained more soluble protein, with important consequences to the functionality of the concentrated milk during acid induced gelation. During acidification, the gelation pH was affected by the extent of concentration, due to a reduction in the interparticle distance and increased collision frequencies, but also by the changes in functionality of the micelles. Concentrated samples formed significantly stiffer gels than control milk because of an increased amount of linkages in the network. Diafiltered milk showed a significantly higher gelation pH compared to the ultrafiltered milk at the same volume fraction. And this cannot be explained only by the decrease in the interparticle distance. This work provides important insights into a comprehensive understanding of the effect of membrane filtration on physico-chemical functionality and properties of casein micelles in the concentrated milk, possibly allowing improvement of processing efficiency.

Effect of xanthan gum addition on food processing and storage stability of fruit juice based beverages enriched with dietary fibers.

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Fruit based beverages represent a good vector to deliver functional ingredients. Dietary fibres have several health attributes such as contribution to digestive health, control of glycemic index. Several health effects have been related to increased viscosity of added polysaccharide. Guar gum (G), β -glucan (Bg) and konjac mannan (K) are examples of functional PS added to food products to provide some health benefits. However, the effect of processing and storage conditions on PS stability is unknown especially in liquid products. Recent works reported a synergistic effect between xanthan gum (X) and oat β -glucan on viscosity of heat-treated fruit juice based beverages. Therefore, the objective of this study was to determine if X has a protective effect on stability of three fibers (G, Bg, K) in a fruit juice based beverage during processing and storage. Ratio and final concentration of fibres and X have been chosen to reach a common viscosity at 30 1/s and are compared at this shear rate value. Statistical analyses were done to study the effect of pasteurisation, time (0 to 4 months) and storage temperature (4 or 20°C). Pasteurization stabilized viscosity and turbidity of Bg and Bg-X beverages as compared to unheated beverages over a one week storage period. Viscosity of pasteurized beverages enriched in G increased (18 %) while the K beverages decreased (13 %). Addition of X has prevented the viscosity changes observed on fiber K (K-X) but not on fiber G (G-X). Viscosity and turbidity of beverages enriched in β -glucan (Bg, Bg-X) significantly increased with time ($p < 0.01$); up to 300 % for Bg and 200 % for Bg-X after 16 weeks. Bg-Bg interactions were favored during storage. However, viscosity of beverages enriched in K and G decreased over time, probably due to depolymerisation reactions. Addition of X protected K against degradation at 4°C but had no effect on G. Viscosity and turbidity changes are faster when beverages are stored at 20°C. Therefore, processing and storage treatments have a major impact on fiber's stability. In many cases, addition of X can have a protective effect. Formulation is crucial to ensure technological stability and physiological effect of dietary fibers.

Effects of oxidation on physical and mechanical properties of ethyl cellulose oleogels

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Ethyl cellulose (EC) oleogels have great potential to be used as a fat mimetic to reduce saturated and trans-fats in food formulations without compromising quality or textural aspects. These gels are formed through the physical entanglement of EC polymer strands which form a network supported by hydrogen bonding. Within this matrix liquid oil droplets become entrapped, acting as a passive filler; that is, they do not chemically interact with the polymer network [1]. It has recently been demonstrated that the mechanical properties of these oleogels are highly dependent on the fatty acid composition of the oil phase [1, 2]. However, to induce the gelation process, EC must first be heated above its glass transition temperature. The entire sample volume must therefore be heated to temperatures in excess of 140°C, which can have a significant impact on the unsaturated fatty acids present in the solvent. In this work, we have demonstrated that heat induced auto-oxidation of the oil also plays a significant role in determining the mechanical strength of the resulting oleogel. Back extrusion was used to assess the mechanical properties of 10 cP EC oleogels prepared using increasingly oxidized canola oil. The mechanical strength of the resulting gels were observed to be positively correlated with the extent of oil oxidation (measured via peroxide value), suggesting that an increase in polarity of the solvent enables it to interact with EC and provide structural support to the gel network. This hypothesis is also supported by the observation that pre-oxidized canola oil produces a much more homogeneous gel structure. It has been discovered that oleogels prepared with higher viscosity formulations of EC (20, 45 cP) tend to fractionate, producing a two-phase gel with a firm exterior and a significantly softer core; an effect which is exacerbated in the presence of the plasticizing agent sorbitan monostearate. The complete absence of this 'jelly donut' effect from oleogels prepared with pre-oxidized oil suggests that oxidative degradation results in improved EC solubility. This work provides insight into the molecular interactions involved in the stabilization of EC oleogels and will help improve the quality of vegetable oil-based EC oleogels for future applications in food systems.

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Properties and structure of functionalized citrus fibers obtained by high shear and alcohol treatments

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Fibers from CWM of citrus are prepared according to a specific process that includes some stages of high shear treatment followed by dehydration into alcohol. These specific treatments allow the fibers to be dispersible and hydratable after drying even under low shear conditions. Therefore, it constitutes a new range of texturizers based on plant cell wall materials. The functionality of these fibers is described in terms of rheological behavior and ability to build viscosity in various media and conditions. It is observed that they have a much better ability to develop texture under low shear conditions than for instance microfibrillated cellulose or other related fibers. The effect of solid concentration is approached in terms of critical concentration, for which values of 3 % weight concentration were found. Based on these properties this new texturizer has been compared with other fibers and microfibrillar cellulose. Some elements of structure investigation are given. Chemical analysis and microscopy can help understanding a specific ultra- and microstructure of these products. Thus cellulose, hemicellulose, pectin and protein content are measured. Microscopy shows different structure than this of classical microfibril of cellulose. It is emphasized that these fibers based on CWM do not have the structure of microfibrillated cellulose and therefore behave differently. This brings up to new application opportunities in both food and non food domain.

Instrumental and sensory analysis of the firmness of vacuum-infused strawberries

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Most fresh fruits are very perishable and therefore heating or freezing processes are used to extend their shelf life. Due to these processes, the firm texture of the fresh product is lost, and a soft, undesirable product is obtained. In recent years, considerable research has shown that the firmness of processed strawberries can be improved, by using vacuum infusion as a pre-treatment to incorporate Ca^{2+} -ions together with exogenous pectinmethylesterase (PME) in the fruit [1, 2]. PME de-esterifies pectin and the resulting low-methoxylated pectin is linked in a network with Ca^{2+} as a Ca-pectate gel. This significantly increases the firmness, even after subsequent processing including heating, freezing and high pressure processing [1, 3]. The present work further elaborates on this, by incorporating sensory analysis of the firmness of the processed strawberries (evaluating the impact of freezing, heating and high pressure treatment). Two strawberry varieties (Elsanta and Albion) were analysed instrumentally with two different probes (5 mm cylindrical probe; Ottawa cell, TA.XT II+, StableMicroSystems) and by a sensory panel (15 trained panel members). The strawberries were analysed fresh (with and without vacuum infusion), and after freezing and thawing, heat processing (for 5 and 20 minutes) or high pressure treatment. The increased firmness, shown with both probes, was confirmed by the sensory panel, for both varieties and for all treatments. All data were lumped together to identify the relationship between the sensory and the instrumental data. A better correlation was found for the sensory results and the results obtained with the cylindrical probe as compared to the results obtained with the Ottawa cell. However, in an experiment where only fresh strawberries (15 different varieties) were evaluated, a better correlation between the Ottawa cell and the sensory analysis, as compared to the cylindrical probe results was obtained. The pectin content, degree of methylesterification of pectin and the Ca^{2+} -content of the strawberry samples were analysed as well. The increased firmness of the vacuum infused samples could be clearly ascribed to the lower degree of esterification (from 70 to 45 %), in combination with the increased Ca^{2+} -content (3 - 4 times higher), compared with the fresh samples.

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Controlled fracture behavior of field peas via the state diagram

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Field peas (*Pisum sativum*) and other pulses exhibit highly organized cotyledon tissue structures consisting of starch granules embedded in a protein matrix [1]. Fine milling of peas results in a powder mixture of which small particles ($< 20 \mu m$) have a higher protein content. Air classification is applied to produce protein-enriched concentrates up to 56 w/w % protein from the fine fraction. Although this dry fractionation route provides a functional protein concentrate, its purity is relatively low. During milling, cotyledon material is stressed by the action of mechanical moving parts (e.g. pins). When the local strain energy exceeds a critical level, fracture occurs along lines of weakness, and stored energy is released. Ideally, fracture of the material leads to complete disentanglement of starch granules and protein without damaging the starch granules. The mechanical properties of the cell constituents determine to a large extent fracture behavior and vary with moisture content and temperature [2]. This can be understood from the different structural states (i.e. glass or rubber) of starch and protein domains under varying conditions as represented in a state diagram. This work presents a state diagram for the cotyledon tissue of field peas based on Differential Scanning Calorimetry (DSC) and Thermal Mechanical Compression Test (TMCT) analyses of pea starch and pea protein. The state diagram of pea starch is compared to the state diagrams of other starches and their respective prediction based on Flory-Huggins free volume theory [3]. Controlled breakage experiments of single peas, in which the structural state of protein and starch is varied, will be done using a texture analyzer. These experiments are meant to identify resulting fracture lines through the cotyledon tissue and could provide an indication to what extent protein matrix and starch granules are disentangled. The latter will be confirmed by Scanning Electron Microscopy (SEM) pictures. Finally, the ambition is to translate the single pea breakage results to pilot-scale milling. The novelty of this work is that mechanical properties of the individual cell constituents are studied and controlled to provide optimal breakage behavior resulting in complete disentanglement of starch and protein.

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Separation and hydrolysis of wheat gluten using extreme low moisture concentration

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The objective of this project is to investigate the effect of lowering the moisture concentration on separation and subsequent enzymatic hydrolysis of wheat gluten considering the full process chain from wheat flour to the final product. Wheat gluten, the major protein of wheat, is mainly used as an additive to improve the baking quality of flours. As a by-product of the wheat starch production, its world production has strongly increased in the last years. However, its lack of some desirable functional properties, such as its water insolubility at neutral pH, limits its application. Enzymatic hydrolysis is a mild and selective method to modify the protein structure and to improve its functional properties. The two process steps of separating starch and gluten and at a later stage the hydrolysis of gluten are currently performed with an excess of water. However, a high water usage and the resulting higher energy consumption are undesired from an environmental point of view. Some years ago, a new separation principle was introduced for wheat flour (Peighambardoust et al., 2008), the so called shear-induced separation. By exposing wheat flour dough to a curvi-linear shear field it can be separated at low water concentrations owing to differences in the rheological properties of starch and gluten. More recent, it was found that this separation consists of three steps being gluten aggregation, subsequent grow to gluten domains and migration to the cone. The final step was found to be strongly dependent on the device geometry. To further increase the protein purity or even lower the moisture content, the influence of xylanase on the shear-induced separation was investigated. Xylanase catalyzes the degradation of arabinoxylans which are present at 1.5 - 2.5 % in wheat flour, which could be favorable because arabinoxylans negatively affect gluten agglomeration and have a high water-holding capacity (van der Borgh et al., 2004). Hence, the water content was further reduced up to 32 % wb. Under these conditions the separation process altered, amongst other due to changes in the rheological behavior of the dough. The reduced water content led to increased viscosities which influenced the migration step. Furthermore, the protein content of the gluten patches could be increased up to 55 % using xylanase compared to 40 % for non-enzyme samples.

Development and optimization of centrifugal impact milling for preparation of biopolymer powders for food applications

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Galactomannan polysaccharides are long-chain macromolecules found at high concentrations within the endosperm tissues of seeds of several legumes such as guar (*Cyamopsis tetragonoloba*), tara (*Caesalpinia spinosa*), and carob (*Ceratonia siliqua*). Beneficial uses of these water-soluble polysaccharides have been found by the food, paper, and oil industries, primarily as efficient thickeners and stabilizers, binders, and as fibers for the human diet. During conventional production the legume seeds are usually undergoing several harsh and damaging processing steps. The plant seed coat is removed by roasting or acid peeling, followed by grinding the endosperm to a fine powder. These processes cause irreversible changes in the structure of the biopolymer molecule due to thermal and mechanical treatments. Thus, the potential functionality of the product as a thickener is diminished. Although the production process used can alter the properties of these galactomannans significantly, only limited scientific data are available. Therefore a detailed study of milling such endosperms was conducted. Due to a specific milling procedure the powder properties as well as the properties of resulting solutions can be markedly enhanced. The solution viscosities were increased up to 5 times compared to conventional commercial materials. This increase was most pronounced at colder dissolution temperatures of 5 - 45°C. In addition, the newly developed process leads to an exceptionally increase of the solubility of 25 %. Hence, the new process preserves the polymer structure naturally present in the plant endosperms, while leading to a better accessibility of the polymer chains during dissolution. Besides, the typical random coil behavior of the galactomannan solution could be preserved. The detailed knowledge of the relevant parameters influencing the process combined with the knowledge of the endosperm morphology and galactomannan molecular structure enables us to tailor the process for specific flour attributes such as particle size, dispersing, and wetting properties, or enhanced solubility and molecular weight. Thus, it is possible to control the desired powder properties as well as the rheological behavior of the resulting galactomannan solutions.

Flour quality and dough sheetability

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The conventional dough rheology tests, as practiced in the cereal industry for assessing end use applications of wheat, are performed with doughs that are mixed to optimal water absorption set at 500 BU in a Farinograph mixer. Dough consistency corresponding to 500 BU is considered acceptable for handling and the rheology tests are used to further delineate between flours for end use applications - breads, cakes, feed etc. In addition, flours that require a higher proportion of water to form 500BU doughs are more profitable for product manufacturers. As a result, lower protein flours, which require less water to form optimal doughs, are discarded immediately for bread making applications. However, this paradigm changes when the method for dough processing is sheeting, in which wetter, low protein doughs can be handled. High quality breads have been made from these flours. The question becomes how to characterize flours for dough sheetability. Chakrabarti-Bell et al. [1] showed that dough's elasticity dominates dough behavior during sheeting by incorporating a rheological model for dough into finite element simulation of sheeting. Therefore, to develop a method for characterizing dough sheetability, a project was carried out using six different flours sourced around the world, varying in both protein content and water absorption. Each flour was mixed at three different flour-water ratios and then sheeted using an instrumented single roll sheeter. True rheological properties were also measured and the data fitted to a modified Bergstrom-Boyce model for dough [1]. Good agreement was observed in terms of dough sheet exit thicknesses and roll forces. The dataset highlight a simple relationship between roll forces and exit thickness providing definitions and measures for dough sheetability. The procedures for deriving parameters for the BB model for dough are described, and the importance of performing virtual sheeting tests for accurate estimation of dough elasticity and sheetability is highlighted.

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Confocal microscopy of bread dough under controlled thermo-mechanical treatment

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The rheology of bread dough is known to be complicated. However, it was extensively studied using both empirical and fundamental rheology characterizations. Many studies proposed correlations between rheological properties and macroscopic behavior such as elastic recovery after shaping or alveolar structure after proofing and cooking. The protein gluten network is recognizing to play a major role on the rheology of the dough. On the other hand, dough microstructure was observed at different scales using microscopy techniques such as CSLM. However, to our knowledge, no study exists on the direct observation of the evolution of the gluten organization at microscopic scale under shearing and heating.

Using a home made rheo-optic device mounting on a confocal microscope, bread dough for different composition (water, sugar and oil contents) under continuous or oscillating strain was studied to find the optical signature of microstructure changes in relation with shear-thickening effect previously measured. This device allows controlling the strain and the shear rate but also the temperature of the dough, all parameters causing changes in gluten microstructure. It was shown that the higher the water content or the sugar content, the stiffer was the gluten strands but the less continuous the network before shearing and the more reinforced were the gluten strands under shearing. Moreover, the oil could be observed in the form of droplets located at the dough/bubble interface and in the form of gluten/oil co-localisation areas around the bubbles. Shearing the dough using an oscillatory strain signal led to an increase of the co-localisation areas. Finally, by a controlled increase of temperature, the growth of an air bubble in bread dough containing yeast was followed during proofing. The influence and the disposition of fat globules at the bubble air-protein interface along this growing process were followed.

In- and off-line determination of the viscosity functions of starch melts

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In food technology cereals are produced during extrusion cooking in twin-screw extruders. Starch is plasticized due to mechanical and thermal energy input. The energy input during extrusion cooking may change the molar mass distribution of the starch, thus alter in its rheological properties. Accordingly the rheology of starch melts not only depends on formulation (kind of starch, solids and moisture content), but also on the process parameters (temperature, pressure, screw speed). Up to now it was not possible to determine rheological properties of starch melt with sampling during extrusion and subsequent analysis in lab, because the melt only flows at similar temperature and pressure conditions like in the cooking extrusion process. Therefore, the rheological measurements of starch melts were done in-line in capillary rheometers directly attached to the extruder exit. These in-line-measurements suffer from the limited shear rate range. The specific mechanical energy input changes with variation of flow rate and accordingly the starch properties may change. Therefore, measurements are often done at single shear rates. The latter issue makes the correlation of single measurements nearly impossible. With a new developed in-line capillary rheometer, that allows to vary the geometry and therefore the shear rates in operation at constant volume flow and specific mechanical energy input, the viscosity function could be determined at a wide shear rate range. In parallel the generated starch-extrudates were characterized rheologically under pressure in a commercial high-pressure cell (Anton Paar) in a laboratory rheometer (MCR501) at process-relevant temperatures. The in- and off-line determined viscosity functions are compared and described with the Carreau model. The dependence of the Carreau parameters on the moisture content and specific mechanical energy input are discussed. Furthermore, the extrudates were analyzed with respect to the change of the molecular structure and the viscosity functions are correlated to the molecular degradation and the specific mechanical energy input.

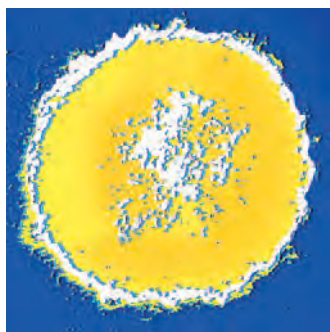
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Encapsulation

pH-responsive lyotropic liquid crystals for controlled drug delivery

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We present a food-grade lyotropic liquid crystal system, capable to respond to pH-variations with a reversible switch in both the structure and physical properties. The system, which is composed by monolinolein and linoleic acid (97:3 wt % ratio) in presence of excess water at 37°C and 150 mM ionic strength, is specifically designed to reversibly change from a reverse Im3m bicontinuous cubic phase to a HII reverse columnar hexagonal phase, when changing the pH from neutral (pH = 7) to acidic (pH = 2) conditions, to simulate intestine and stomach conditions, respectively. The pH-responsiveness is provided by the linoleic acid, which being a weak acid (pKa5), is essentially in the de-protonated charged state at pH 7 and mainly protonated and neutral at pH 2, imposing changes in the critical packing parameter (CPP) of the lyotropic liquid crystal. The use of this system as an efficient controlled-release delivery vehicle is demonstrated on the hydrophilic drug phloroglucinol, by both release and diffusion studies at different pH, as followed by UV-Vis spectroscopy. The Im3m cubic phase at pH 7 is shown to release four times faster than the HII phase at pH 2, making this system an ideal candidate for oral administration of drugs for targeted delivery in intestine or colon tracts.

Biopolymer-based core/shell capsules: colloidal interactions, interfacial physics and barrier properties of complex coacervate delivery systems

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Core-shell capsules with (bio)polymer walls are relevant in a many applications including flavors, perfumes, pharmaceuticals, inks, or pesticides. For flavor applications, the choice of wall materials is naturally restricted to edible ingredients in line with legislation constraints; self-assembled, biopolymer-based capsules are among the most important delivery systems in this field. Encapsulation systems that combine a label-friendly wall chemistry and ease of applicability or low material and processing costs are particularly interesting for flavors and fragrances. Complex coacervation is a liquid/liquid phase separation occurring in colloidal systems, resulting in the formation of two liquid phases: a hydrocolloid-rich phase (coacervate phase) and a dilute continuous phase. The coacervate appears as amorphous liquid droplets exhibiting affinity for interfaces due to specific interfacial properties. Formation of microcapsules by complex coacervation typically involves the steps of (i) phase separation and formation of coacervate nodules; (ii) emulsification/dispersion of the active ingredient; (iii) deposition and coalescence of coacervate nodules onto the active/continuous phase interface, (iv) physical gelation and, optionally, (v) covalent crosslinking of the wall. Whereas step (i) has been covered in much detail in the literature over the last decades, there is far less quantitative knowledge available regarding the engineering/processing of the (micro)capsule genesis described in the steps (ii)-(v). In this contribution, we provide a comprehensive discussion of complex coacervation processes from the perspective of delivery systems for active ingredients, focusing on the intimate coupling of phenomena including colloidal interactions and phase diagrams, the hydrodynamics of core formation, and quantitative analysis of the wall materials, in particular the role of crosslinking in gelatin and coacervate materials. Methods include microscopy, dynamic and static light scattering, rheology, and micro-DSC.

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[2] P. Bouquerand, G. Dardelle, P. Erni, V. Normand, in: *Encapsulation technologies and delivery systems for food ingredients and nutraceuticals*, Garti N and McClements D. J. (Eds.), Woodhead Publishing Ltd., Cambridge UK (2012, in press)

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The incorporation of polyphenols in casein micelles – encapsulation behavior and effect of processing

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Casein micelles are porous protein assemblies about 200 nm in diameter often described as one of nature's solution for delivery of nutritional compounds. Polyphenols are important food components with recognized bioactivity. The objective of this study was to determine differences in the affinity of casein micelles for model polyphenols. Caseins have been shown to be avid binders of polyphenols when the proteins are present in monomeric form, but not all these bioactive molecules will have the same affinity to the casein micelles. Curcumin, resveratrol and EGCG (green tea catechin) were tested as model molecules. Fluorescence quenching experiments clearly showed differences in the affinity of casein micelles for the polyphenolic compounds. Curcumin, the most hydrophobic amongst the molecules tested showed a 1:1 quenching of tryptophan fluorescence. On the other hand, resveratrol and EGCG, more hydrophilic, showed limited access to the inner core of the casein micelles, with resveratrol being the least effective in quenching tryptophan fluorescence. The association of polyphenol molecules with casein micelles affected their rennet induced gelation. In all cases the gelation time and the formation of the gel network were inhibited, but the extent of the inhibition depended on the nature of the polyphenols. EGCG, which has been reported to specifically bind to the proline residues of the casein micelles, at saturation concentration showed inhibition of the primary stage of casein micelles aggregation. Resveratrol and curcumin showed inhibition of the secondary stage. A better understanding of the effect of the presence of polyphenols when encapsulated in casein micelles is critical in order to deliver these bioactive molecules through dairy products.

Microencapsulation of probiotics in milk protein matrices

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Probiotics are defined by the WHO as “live microorganisms which, when administered in adequate numbers, confer a health benefit on the host. For an effective application in functional food it is therefore necessary that the bacteria reach their destination with high viability and functionality. Major issues are therefore the protection of the probiotic cells during processing, adverse milieu conditions such as low pH or oxidative environment in the host product, which carries the probiotics, longer periods of storage under such conditions, and gastric transit as well as the controlled release in the human gut. Microencapsulation offers a possibility to improve the survival of the bacteria along the whole chain until reaching the intestine by enclosing the bacteria with a coating or in a matrix. A novel method for the encapsulation of probiotic cells in food-grade protein matrices based on spray drying was developed. The process is based on rennet induced hydrolysis of the κ -caseine in skim milk concentrate before spray drying. In contrary to existing encapsulation methods based on spray drying, this process offers the chance to produce water insoluble microcapsules in a large scale. Besides this advantage, the protein matrix shows better barrier effects in comparison to alginate capsules because of the high gel network density. In this study the influence of dry matter content of the skim milk concentrate and drying temperature on the survival of the bacteria and matrix properties was investigated. Regarding the survival, survival rate increases with decreasing solid concentration and increasing drying temperatures. Regarding the matrix properties, protein composition and solubility of the spray dried capsules were studied depending on rehydration temperatures by SDS-Page and BCA assay. It has been shown that for higher rehydration temperatures, water insoluble spherical capsules with a volume-based median in the size-range between 50 and 350 μm were obtained. The size and shape of the capsules after rehydration was dependent on the dry matter content, way of atomization and the process parameters during the drying process. The capsule matrix composition includes to a large extend caseins but also denatured whey proteins.

Oligofructose fatty acid esters create air/water interfaces with extremely high moduli

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Two main classes of currently used food-grade emulsifiers are proteins and low molecular weight surfactants. Low molecular weight surfactants lower the surface tension to a greater extent than proteins. Proteins, however, tend to form a viscoelastic network at the interface leading to a high dilatational modulus while low molecular weight surfactants generally create interfaces with a low dilatational modulus. Inspired by amphiphilic block oligomers, oligofructose was esterified to a fatty acid in an attempt to create a food-grade surfactant that is capable of lowering the surface tension considerably while also creating an interface with a high dilatational modulus. In order to create molecules with different functionality the fatty acid chain length was varied: butyric, caprylic, capric, lauric, palmitic and stearic acids were used. Surface tension and surface dilatational modulus were determined using an automated drop tensiometer, while the surface shear modulus was determined using a rheometer equipped with a double wall ring geometry. The results were compared to those obtained with sucrose esters of fatty acids. The extent to which the surface tension is lowered depends on the length of fatty acid: the longer the fatty acid the lower the surface tension. Palmitic acid and stearic acid esters are particularly efficient in lowering the surface tension. Esters (with either sucrose or oligofructose as the hydrophilic head group) of shorter fatty acids (lauric acid or smaller) give a low dilatational modulus while esters (both oligofructose and sucrose) of longer fatty acid chains (palmitic acid or stearic acid) give an extremely high dilatational modulus. The shear modulus of esters of shorter fatty acids is very low. When studying esters of palmitic acid and stearic acid there is a clear difference between sucrose esters or oligofructose esters. Oligofructose esters do give a high shear modulus while moduli of sucrose esters were very low. Thus, with the palmitic acid and stearic acid esters we were able to synthesize molecules that considerably lower the surface tension of an air/water interface and give an extremely high dilatational and shear modulus.

Alginate capsules gelled with CaCl_2 nanoparicles

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Alginate is a well-known food grade polymer in microencapsulation technologies. It can be gelled using divalent cations such as Ca^{2+} . It is non-toxic, low in costs, and can be used to trap bacterial cells and other compounds. Many methods have been developed for encapsulation in alginate capsules, which can generally be divided into internal and external gelation. Most of the methods result in capsules larger than 100 microns. This size is however undesirable if the alginate capsules should be added to food products and should be undetectable in the mouth. Capsules smaller than 25 microns are then required. We developed a new method for producing alginate capsules smaller than 25 microns. An alginate solution, containing bacterial cells or other compounds, is emulsified in an oil continuous phase forming a water-in-oil emulsion. Separately, we have developed a method to produce food grade CaCl_2 nanoparticles (120 nm). The nanoparticles are prepared through evaporation of ethanol from an ethanol-in-oil emulsion, whereby the CaCl_2 is dissolved in the ethanol phase. The method was evaluated with two ethanol soluble salts (CaCl_2 , MgCl_2), which are also water soluble. The formation of CaCl_2 nanoparticles was ascertained by scanning electron microscopy and by dynamic light scattering. The CaCl_2 nanoparticles are added to the oil continuous phase containing the alginate emulsion droplets. The CaCl_2 nanoparticles will then adsorb on the surface of the alginate emulsion droplets and start to dissolve into it, gelling the droplets. The gelled alginate droplets function as matrix for holding the encapsulated matter. The conditions inside the alginate capsule can be optimized to the needs of the encapsulated compound. The gelled capsules can be separated from the oil and additional coatings can be applied on the alginate capsules to obtain desired stability properties.

Emulsification using EDGE (Edge-based Droplet Generation) devices

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Monodisperse emulsions with sizes of 0.1-100 μm have several applications in both science and industry. Important characteristics of emulsions (rheology, appearance, chemical reactivity, physical stability etc.) are determined by droplet size and size distribution. Traditional equipments, having poor control over droplet size, produce highly polydispersed emulsions with coefficient of variation (CV) of around 40 % which makes them intrinsically unstable. Several micro emulsification systems (T- and Y- junctions, co-flow systems, flow focusing devices, microchannels etc.) have been introduced in the past 20 years. These systems have excellent control over droplet size and produce highly monodisperse emulsions. However, range of their practical applications is limited due to low throughputs and complications related to their scale up. We have recently introduced a new emulsification system called EDGE (Edge-based droplet generation) with which multiple monodisperse droplets can be produced simultaneously from a single droplet formation unit (DFU). An EDGE-unit is a slit like structure called plateau located between an oil and a continuous phase channel. Monodisperse droplets are successfully produced at several locations along the entire length of the plateau. The system is robust and stable under acceptable pressure range. Investigations on an up-scaled version (chip of 1.5 cm^2) showed that all the DFUs were active. Double emulsions, which are used as templates for encapsulation systems, have also been successfully produced from practical food grade ingredients. Further scale-up is currently being investigated and for this, amongst others, metal versions of EDGE systems are considered. Our initial results with metal EDGE systems are quite promising.

Flow and diffusion in heterogeneous hierarchical model materials studied by Micro-PIV

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Much of the physical properties of soft biomaterials are derived from the microstructure, i.e. the size, shape, and spatial distribution of their components. This includes mass transport ability; that is the diffusion and flow properties. In structures having pores at nm scale, mass transport by diffusion is dominant, and no flow can be pushed through the structure. By increasing the structure dimensions from nm range to μm range, the mass transport via convective flow gets increasingly important. However, not much work has been done to reveal the structure-flow relationships in heterogeneous porous materials at the micrometer level and where the transition between flow and diffusion occurs. Here, we provide an experimental method for characterization and understanding of flow in heterogeneous porous structures. For this, we have established a method for flow measurements in microfluidic systems through tailor made model material in PDMS, using soft lithography. This material can be chemically modified, providing a wide range of surface properties displayed for the flowing media. The structures, having a Sierpinski carpet pattern or a pattern that mimics random heterogeneous materials, are characterized by confocal microscopy, and the flow is monitored by μ -PIV. Results on particle flow and diffusion behavior in these model materials show good agreement with predictions from Lattice-Boltzmann modeling, which are performed in geometries constructed from 3D-CLSM images of the actual PDMS structures. Given this, mass transport behavior may be predicted even for more complex structures, like gels or composite material, in e.g. food or biomaterials. This is a step in the directions towards predictive science with regards to tailoring mass transport in soft biomaterials.

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Macromolecular Assemblies

Characterization of complexation and coacervation between beta-lactoglobulin and soluble fraction of gum tragacanth: Approached by spectroscopic, rheometric, and light scattering methodologies

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Over the last couple of decades, a great interest in development and optimization of the multi-functional systems by interactions between macroions, especially proteins and polysaccharides, has been appeared. It is well-known that characterization of the phase behavior of non-cognate protein-polysaccharide systems is essential for providing a foundation for their possible applications such as protein purification, the design of food analogous and microencapsulation technology. Gum tragacanth, a branched heterogeneous anionic polysaccharide, is obtained as a dried exudate from the stems and branches of different species of *Astragalus*. This highly acid-resistant gum, which has been accepted since 1961 as GRAS at the level of 0.2 - 1.3 %, consists of two major fractions: tragacanthin (water-soluble fraction) and bassorin (water-insoluble but swellable fraction). Beta-Lactoglobulin, the major protein of bovine milk whey, is a small globular protein of 18350 Da. This protein has a quite well known structure containing mainly beta-sheets, some beta-turns, and one alpha-helix; therefore, it can play the role of an in situ molecular marker in the study of the formation and structure of complexes and coacervates. In the present study, we utilized the coupling of slow in situ acidification of β -lactoglobulin/tragacanthin mixture and rheometry to monitor online the structural changes that occur during the assembly process. In parallel, spectrophotometric and light scattering measurements were done, as complementary methods. The results obtained by this multi-methodological approach allowed us to parameterize the associative phase separation process in terms of a set of characteristic pH values at which critical structural changes took place. Rheological measurements provided interesting insights into the phase behavior of the system and the results were in good agreement with quiescent pH-induced phase transitions established by two other methods. The formation of soluble interpolymeric complexes caused an increase in the viscosity of the system, likely, due to the solvent reorganization and the ordering of water molecules at the complex interface. As the complexes initiate aggregation, accompanied by the liberation of water molecules from the particles, the viscosity decreased over time (or decrease in pH), particularly below pH where phase separation began. Subsequently, coacervate formation became evident by the appearance of an identifiable peak in the mechanical spectrum.

Comparison of viscoelastic properties of gluten obtained from einkorn, emmer, durum, spelt, and common wheat

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Primitive wheats, einkorn (*Triticum monococcum* ssp *monococcum*), emmer (*T. turgidum* ssp *dicoccum* (Schrank ex Schubler) Thell.) and spelt (*T. aestivum* ssp. *spelta* (L.) Thell.) receive significant interest from the part of organic agriculture and as the crops that after reintroduction into modern agriculture could help maintaining biodiversity of the sustainable agricultural environment. Gluten proteins complexes of these wheats represent natural model systems composed of genetically significantly differentiated sets of gliadins and glutenins enabling deeper insight in molecular basis of structure formation and fundamental rheological properties of the gluten matrix. In this paper, protein fractional composition and rheological properties of gluten samples isolated from einkorn, emmer, durum, spelt and common wheat were studied. Gluten samples were isolated from einkorn cultivars Tifi, Terzino, Svenskaja (Germany) and Monlis (Italy), emmer Rossorubino and Zefiro (Italy), durum wheat Iride and Levante (Italy) spelt Alkor (Switzerland) and common wheat cultivars of contrasted technological value (Begra and Wilga, Poland). Protein fractional composition of flour and gluten samples was determined according to a three-step protein extraction procedure and capillary electrophoresis method. Gluten samples for rheological studies were prepared by rehydrating freeze-dried gluten in an excess of solvent. For rehydration, a 0.1 M N-ethylmaleimide (NEMI) solution was used to prevent chemical ageing of the samples during the rheological measurements. The controlled stress rheometer Rheometric SR 500 was used in a cone and plate geometry. Mechanical spectra were registered over frequency range 0.001 - 200 rad/s and strain amplitude was kept within 3 %. Retardation tests were determined for studied gluten samples. Cole-Cole fit parameters: plateau compliance (J_{N0}), plateau modulus (G_{N0}), loss peak characteristic frequency (ω_0) and spread parameter n were calculated from gluten mechanical spectra. Retardation test parameters (steady state compliance (J_e) and steady state viscosity (η_0), were determined. The method of Kaschta was used to calculate the discrete retardation spectrum from creep recovery data; the spectrum was then used to convert recovery data into storage and loss compliance and modulus. This approach enabled extension of frequency window of gluten mechanical spectra down to 10^{-6} rad/s. Width of viscoelastic plateau I was calculated from the composite mechanical spectra. Multiple regression analysis of dependencies between gluten protein fractional composition and gluten rheological parameters was performed.

Milk proteins under extreme conditions

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Structural and functional studies of proteins are well established in solution and crystals, but still challenging at interfaces or under high pressure. With an increasing degree of oligomerization of proteins there is a disproportional increase in the complexity of the matter considered and in the experimental effort. The first system we considered was the casein micelle, a macromolecular system with sizes distributed around 160 nm. We show how casein micelles behave in membrane-near deposits when subjected to fluid flow fields. In detail, we focus on shape changes of the originally spherical micelle in dependence of the deposit height and the trans-membrane pressure applied. For the studies we used both grazing incidence small angle X-ray scattering (GISAXS) and atomic force microscopy (AFM) for the surface-sensitive measurements and static and dynamic light scattering (SLS/DLS) for the characterization in solution. In contrast to AFM, which provides a top-view of the deposited casein micelles, GISAXS allows also resolving shape changes perpendicular to the membrane surface. For the filtration experiments we used SiXNiY-micro-sieves characterized by smooth surfaces needed for surface-sensitive experiments. As a second system we investigated β -lactoglobulin (BLG) under high hydrostatic pressure. The protein is the main whey protein in bovine milk with a molecular mass of 18.3 kDa. At ambient pressure and pH 4.6, the protein exists as a mixture of octamers and dimers. We show how the oligomeric state changes with increasing pressure, temperature and salt concentration. For the experiments we used static and dynamic light scattering in a temperature-controlled high pressure four windows optical cell. The measurements allowed us to estimate variation in molecular weight and hydrodynamic radius in dependence of the milieu and physical conditions. With the oligomeric state, the protein may change also its structure-functional properties, which will be considered in the future.

Relation between particle properties and rheological characteristics of carrot-derived suspensions

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Nowadays, there is a vast consumption of liquid vegetable-derived products such as purées, baby food, soups and sauces. These are produced by blending, mixing and homogenizing raw vegetables resulting in suspensions consisting of plant-tissue-based particles in a continuous serum phase with (among others) pectin, sugars and organic acids solubilized in it. The sensory properties of processed foods (in particular texture, flavor and mouthfeel) as well as the bioaccessibility of micronutrients from such foods are determined by their microstructure and the related rheology. Hence, understanding the relations between microstructure and rheological properties of these products is needed to allow tailoring of their quality characteristics. The rheological properties of food suspensions are known to be determined by both the particle properties of the dispersed phase and the properties of the serum phase (especially of the solubilized pectin). In the present work, the effect of particle type, size, polydispersity and concentration on the rheological behavior of carrot-derived suspensions was investigated systematically. Hereto, a range of relatively monodisperse suspensions, containing either carrot cell fragments or cell clusters with average cluster sizes ranging from 70 - 370 micrometer, was prepared by reconstitution of carrot tissue particles in water. All the carrot-derived suspensions, with pulp content from 30 to 65 wt%, are non-Newtonian liquids exhibiting a yield stress. For all reconstituted suspensions, the value of the dynamic yield stress was approximately 75 % of that of the static yield stress. The undisrupted network structure of all suspensions could be described as a weak gel with a rather low critical strain (of the order of 1 %). Particle concentration, size and type appeared to be key structural properties controlling the rheological parameters of these carrot-derived suspensions. Increase in yield stress and storage modulus with particle concentration could be fitted to a power law model. In addition, a unique linear relation was found between the yield stress and the plateau modulus, independent of particle size and type. When comparing the rheological behavior of reconstituted suspensions with that of the original carrot purée of similar average diameter and pulp content, the network structure in carrot purée was weaker, which may be attributed to the broader particle size distribution.

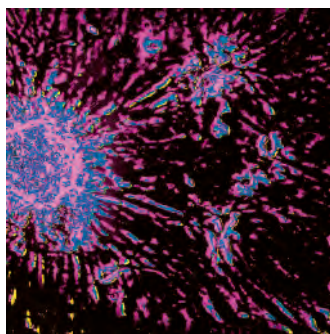
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Structure, Nutrition and Health

Structure and digestion of plant cell wall materials: Does size matter?

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Plant cell walls are the major structural component of fruits and vegetables. They are broken down to small particles during food processing and/or after oral mastication. As there are no enzymes within the human stomach or small intestine that are capable of hydrolysing the main components of plant cell walls, they pass along the gastrointestinal tract without being digested, absorbed or metabolized, until they reach the terminal ileum and colon where they are utilized as fermentation substrates by the gut microbial community. It has been demonstrated that the nature and chemical structure of individual polysaccharides, and the matrix structure and the physical properties of the fibre can significantly affect how materials are fermented. However little is known about how the size and morphology of cell wall particles may influence the fermentation kinetics of cell wall materials. In addition, the break-up of cell walls and cell membranes allows the release of micronutrients such as vitamins and minerals, polyphenols and carotenoids, etc which are otherwise encapsulated. However, the release of micronutrients may be hindered by their interaction with cell wall components. Thus, the physical and physiological functionality of plant cell wall materials have a major impact on the nutritional properties of fruit and vegetable based foods. In this work, a range of thermal and mechanical processes were applied to create cell wall particles with different structural morphologies from carrot root tissues. The dispersions cover a range of particle size and morphology from clusters of cells with an average particle size ($d_{0.5}$) of 298 and 137 μm , respectively, to single cells of 75 μm or cell fragments of 50 μm . In vitro studies were carried out to determine the release kinetics of carotenoids, polyphenols and anthocyanins under simulated gastric stomach and small intestinal conditions, and the fermentation kinetics of plant cell wall particles using porcine faeces as an inoculum. The results showed that the highest release of carotenoids, polyphenols and anthocyanins was obtained from the smaller particles, but in contrast, the fastest fermentation occurred for larger particles. Larger cell clusters were more rapidly fermented and produced more SCFA and gas than smaller single cells or cell fragments, particularly between 8 and 20 h. Confocal microscopy suggests that the junctions between cells provide an environment that promotes bacterial growth, outweighing the greater specific surface area of smaller particles as a driver for more rapid fermentation. These studies demonstrate that it may be possible, by controlling the size of plant cell wall particles, to design vegetable-based foods to enhance micronutrient bioavailability and for fibre delivery to promote fermentation that acts throughout the colon to maximize potential for human health.

Can changes in rheology contribute to the understanding of starch digestion?

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Although not a classical rheological tool, the Rapid Visco-Analyser (RVA) is widely used in the starch and associated industries to investigate pasting properties of food and feed. The shear rates of RVA rotational speeds have been estimated, and with its paddles minimizing sedimentation and moisture loss during experiments, it can be a useful starch (empirical) rheological tool. Changes in the RVA viscosity of potato and waxy maize starches during digestion were studied using two amylases (amyloglucosidase and α -amylase) at three enzyme concentrations (60 - 1700 U), and up to three solids contents (3 - 20 %) of the starch dispersions in a replicated randomized experiment. The starches were completely gelatinized in the RVA, stored at about 4°C for 18 hours before digestion at 50°C at a calculated shear rate of about 17 1/s (50 rev/min) for 5 minutes. Control dispersions effectively did not change (< 10 %) in viscosity with time (a time-independent behavior). With the treated gels, irrespective of the starch, enzyme and concentration, the RVA viscosity reduced with time due to the production of low molecular weight products (dextrins and/or glucose). A first-order kinetic model ($RVAV_t = RVAV_0 + RVAV_{\infty-0} [1 - \exp[-K_{VIS} t]]$) suitably described ($r^2 > 0.9$) the starch viscosity-digestograms, and the rate of change in starch viscosity, K_{VIS} (an index of the rate of starch digestion) was significantly ($p < 0.05$) inversely related to the gel solids content, and directly related to the enzyme concentration. K_{VIS} of the waxy maize starch was higher than that of the potato starch, and a higher K_{VIS} was measured when the α -amylase digested the potato starch than when the amyloglucosidase was the digestive enzyme. Differences in potato and waxy maize starch structures, and specificity of enzymes, as well as enzyme:substrate ratios, are discussed to explain the results. Rheology is proposed as valuable in understanding starch digestion.

Engineering functional foods with high vegetable content

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The increase in the number of overweight and obese children has become a serious public health problem in many industrialized countries [1]. One of the possible solutions to the child obesity problem is to increase children's intake of vegetables [2]. Incorporating vegetables in a gelled matrix (i.e. starch or protein) to create pasta or noodle-like products may increase vegetable intake, as pasta is very appreciated by children [3]. But incorporating high volume fractions of dispersed vegetables in these products has a significant effect on their physical properties and stability [4]. For this reason we have investigated the rheological properties of gelled starch and protein matrices with high volume fractions of vegetables (up to 20 % dry basis) and compared the results with model systems consisting of fish gelatin and quartz beads. Rheological measurements can provide important information about the cooking properties and sensory perception of a product (i.e. hardness and elasticity) and can also provide insight on structure formation and processing conditions that will lead to optimized food products [5]. Our studies show that the swelling of the vegetable particles has a very significant effect on the rheology of the systems. Data were described well by mean field theories, based on a viscoelastic generalization of the Frankel and Acrivos model [6].

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Relations between bread structure, bolus formation and dynamics of salty and texture perceptions

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During food consumption, complex oral processing occurs to progressively transform a solid food into a food bolus, ready to be swallowed. The understanding of bolus formation is important because it prepares the food product for digestion, but also because it contributes to sensory perception. In this context, bread deserves a specific attention, due to the continuous effort of public policies to lower its salt content. So, taking bread as a model food, the aim of this work is to build relations between food breakdown in the mouth, salt release and stimuli perception. In this purpose, 3 model breads were processed in a home breadmaker, in order to control their formulation. They all contained the same salt concentration but varied in structure (breads 1 and 2 had same composition, but bread 2 was denser due to a different fermentation process (density 0.15 and 0.3, respectively)) or composition (bread 3 was made with the same process as bread 1, but with 2 % fat added). Sensory analysis was conducted with a 12-persons trained panel. The dynamics of perception in mouth was studied with the Temporal Dominance of Sensation method (TDS), performed on texture attributes and salty perception. Time Intensity (TI) method was also used to assess the evolution of salty perception during consumption. Eight panelists were asked to spit the bolus after 5 seconds, 10 seconds or just before swallowing and their boluses were characterized. Water content of bolus was determined and rheological properties of bolus were assessed by viscoelasticity measurements on a rheometer equipped with a vane geometry in the linear viscoelastic domain. The release of salt in saliva was measured during consumption, by recovering saliva on the tongue with a filter paper and quantifying sodium with a specific-sodium electrode. Despite inter-individual differences found in bolus properties, results show that breads display different dynamic sensory profiles. Structure and composition impact the evolution of perceptions, and particularly salty perception. Breads 1 and 3 appear to be more salty than the denser bread 2. For these 2 breads, in TDS, salty is a dominant sensation perceived at the end of the consumption sequence and this intensity (observed with TI) is higher. Bolus hydration increases slowly during consumption (~ 10 % wet basis). Both G' and G'' moduli decrease during oral processing by a factor of 3 between the beginning of mastication and swallowing point. Relations between data on salt release in saliva and salty perception are now studied in order to better understand salty perception.

Spatiotemporal mapping and modeling of the duodenum: A physiologically based fluid mechanical model of pendular activity

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The duodenum is an important site for the physical processing of digesta but its contractile behavior is poorly understood. After its transit through the pylorus, the pH of gastric chyme must be raised by mixture with pancreatic and small intestinal secretions to a level that is suitable for the efficient operation of the suite of intestinal enzymes. Further, the level of mixing must be sufficient to enable micelles in secreted bile to physically interact with and disperse contained fat droplets (Mu and Hoy, 2004). The objective of this study was to characterize contractile activity in the duodenum and model its consequences on fluid mechanics and mixing. We used high fidelity longitudinal and radial spatiotemporal mapping to measure contractile activity in the proximal duodenum of 17 rats when perfused with saline or aqueous suspensions of micellar decanoic acid. Its activity was characterized by stationary periodic longitudinal contract is occurring with a frequency of 36 ± 2 cpm and strain rate amplitude of 26.8 ± 8.0 % 1/s on 4-6 domains along the duodenum. At low rates of perfusion these contractions were uncoordinated between neighboring domains and were rarely accompanied by low amplitude circular contractions. Then, a fluid mechanical model was developed. The flow was solved using a Lattice BGK mod for incompressible Navier-Stokes equations (Guo et al., 2000) with moving boundaries. In a first step, the longitudinal and circular contractile activity of the duodenum idealized to study the influence of different parameters, as the Reynolds number ($Re < 16$) and the Strouhal number ($3 < St < 16$), on the flow conditions. In a second step, different sequences of longitudinal contractions measured by spatio-temporal mapping were directly used as boundary conditions to obtain a statistically robust description of the flow in the lumen. We conclude that the organization of longitudinal contractions in uncoordinated territories allow the generation of recirculation between each domain improving the mixing efficiency. Finally, the statistical evaluation of flow conditions allows variance between and within duodenal preparations to be assessed as well as sensitivity of this mixing to changes in viscosity of the luminal chyme.

In-vitro small intestinal non-Newtonian fluid flow characteristics using Ultrasound Doppler Technique

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The in vitro small intestinal non-Newtonian fluid flow investigation is important to biofluid mechanics encountered in human body (e.g. food flow in pharynx, esophagus and small intestine). The pulsed ultrasound Doppler based velocity profile (UVP) technique has been extensively used during steady and unsteady laminar flow of non-Newtonian fluids [1] and suspensions [2] mostly in non-collapsible tubes. In contrast, most of the fluid conveying vessels in the human body are elastic and deform under imposed flow condition [3]. The small intestine is mainly involved for absorption of all nutrients and more than 95 % water during digestion under peristaltic flow, which causes better mixing and enhancing the mass transport of nutrients from food matrix through gastrointestinal tract (GIT) membrane. Therefore, in vitro investigation of modeled small intestine is motivated by the fact that attempts to understand the physical and chemical processes of food undergoing in GIT by peristalsis. The previous study involved experimental investigation of flow behavior (velocity distributions) of non-Newtonian fluids by UVP in a collapsed elastic tube under various compressive transmural pressures using a Starling Resistor [4]. The results depict that the elastic tube collapses from elliptical to line or area contacted two lobes shape and the corresponding velocity profile changes from a parabolic to a bi-modal. It was also observed that shear thinning solution viscosity decreases as the cross sectional area reduces under different degree of tube deformation. In addition, the velocity field in an in-vitro small intestine (by squeezing of an elastic tube) has been investigated under different peristaltic wave speeds resulting in maximum velocity near the tube wall while lowest being at the tube center. The corresponding magnitudes of the velocities and the pressure gradient between crest and trough of a peristaltic wave increased, as the wave speed is faster. The crest showed a higher pressure compared to the trough.

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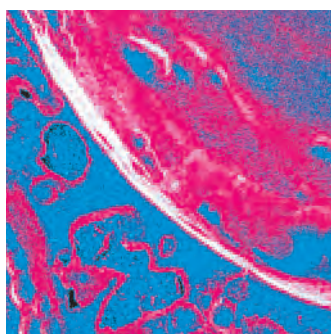
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Emulsions and Foams

Passive microrheology: Non-intrusive measurement of the emulsion stability

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This work presents a new technique of the passive microrheology for the study of the microstructure properties of soft materials like emulsions. Our technology uses Multi Speckle DWS (MS-DWS) set-up in backscattering with a video camera. It allows to measure the mean displacement of the microstructure particles in a spatial range between 0.1 and 100 nm and a time scale between 10^{-3} and 10^5 seconds [1, 2]. Different parameters can be measured or obtained directly from the Mean Square Displacement (MSD) curve like a fluidity index, an elasticity index, a solid-liquid balance, a macroscopic viscosity index, a relaxation time, a MSD slope. This technique allows to monitor the evolution of the microstructure, the restructuration after shearing, the variation of the viscoelastic properties versus temperature, pH, the physical stability of emulsion or suspension. This work focuses on the measurement of viscoelastic properties evolution of emulsions to follow their stability. The results will show the advantages of using a non intrusive method to detect nascent destabilization of the microstructure before rheology or visual method.

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Rheological characterization and stability of high internal phase water-in-oil emulsions

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High Internal Phase Emulsion (HIPE) is an emulsion containing more than 74 % of dispersed phase, resulting in droplets with a shape between spheres and polyhedral. The high viscosity and stability of HIPEs make them interesting for the development of products. In this work, water-in-oil emulsions at high water:oil ratio were evaluated regarding the kinetics stability, rheological parameters and optical microscopy. Emulsions were firstly prepared by adding deionized water to the mixture of n-hexadecane and surfactant polyglycerol polyricinoleate (PGPR) using a rotor-stator system at 14,000 rpm. After that, the emulsion was passed through a two-stage homogenizer at 15 MPa or 30 MPa in the first stage and 5 MPa in the second stage. The kinetic stability of the emulsion was evaluated by visual observation of phase separation during 14 days. The rheological measurements were carried out at 25°C in a stress-controlled rheometer one day after the emulsion preparation. Three steps up-down-up flow curves were obtained between 0 and 300 1/s and data were fitted to the Herschel-Bulkley model. The emulsions were observed in an optical microscope and the droplet size was measured using image analysis. The high-pressure homogenization effect was analyzed through fluorescence microscopy, dying the oil phase with Red Nile stain. The results showed that the emulsion stability increased with the water:oil ratio up to the maximum ratio tested (10:90, w/w) at 5 % PGPR (w/woil). In addition, the higher the water:oil ratio, the higher was the droplet size and the apparent viscosity. At 10:90 water:oil ratio, the emulsion presented many coalesced droplets and had an appearance of a strong gel. Furthermore, the use of high energy (15 MPa) in the emulsification generated smaller droplets and increased the apparent viscosity, but the excessive energy (30 MPa), mainly at high water:oil ratios, led to the emulsion destabilization and phase reversal, as confirmed by the fluorescence microscopy. In conclusion, HIPEs prepared in this study showed good stability due to their high viscosity, but they were susceptible to the application of high energy.

Comparing nano-emulsion formation with a high-pressure valve homogenizer and a Microfluidizer for the production of food-grade nanoemulsions

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The purpose of this work was to compare the production of oil-in-water nanoemulsions using a Microfluidizer and a high pressure valve homogeniser (HPH) for food emulsions. This work systematically compares the variables that control the energy dissipation rate and final droplet size of the emulsion, namely: operating pressure, residence time (passes) in the high turbulence zone, continuous and dispersed viscosities. Estimates for average energy dissipation rates for the Microfluidizer were $\sim 10^9$ W/kg, whilst the HPH was calculated using a method previously used and varied between 10^8 - 10^9 W/kg [1, 2]. The energy dissipation was larger for the Microfluidizer for the pressure range between 500 - 1500 bar, and the $d_{3,2}$ at all these pressures (110 - 120 nm) were smaller than the $d_{3,2}$ value at 1500 bar (128nm) in the HPH. According to turbulence theory, the smallest turbulent eddy size (Kolmogorov scale) has been evaluated for both systems. For the HPH with an emulsion of viscosity ratio 50 at 1500 bar, $K = 138$ nm, there was evidence after the first pass of turbulent inertial break-up. By increasing the residence time in the valve the emulsion saw more of the maximum shearing stresses and therefore after 5 passes the droplet size, $d_{3,2}$ reduced to 128 nm; just below the Kolmogorov length scale. The droplet break-up mechanism in the Microfluidizer is turbulent inertial under the same conditions, with the droplet size distribution after one pass being 10 nm below the space-averaged Kolmogorov length scale. This shows that the difference between the maximum energy dissipation and the average is larger within the HPH than the Microfluidizer. By changing the viscosity of the dispersed phase, to viscosity ratios between 10 - 110, the $d_{3,2}$ increased for the HPH with only turbulent inertial break-up. For the Microfluidizer at a viscosity ratio of 10 the $d_{3,2}$ was 95nm, however, from 20 - 110 the values of $d_{3,2}$ did not increase and ranged from 110 - 117 nm. Decreasing the viscosity ratio further from 10 - 0.1 led to an overall decrease in $d_{3,2}$ for both machines and compared to the Kolmogorov length scale (up to 10 μm) the droplet break-up should be dominated by turbulent viscous break-up. For decreasing viscosity ratios, the residence time in the turbulent chamber becomes more significant. This is the first attempt at comparing these two processes and this work provides a better understanding of the nano-emulsion formation in both the HPH and Microfluidizer with emphasis on the energy dissipation distribution and the droplet break-up mechanisms present.

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Emulsion based lipstick

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Lipsticks consist of 3 main ingredients: waxes, pigments and perfumes. The addition of water, into a water-in-oil emulsion could aid natural lubrication, preventing drying of lips, (i) directly delivering water to lips and (ii) droplet rupture results in micro pores allowing moisture from air or saliva to reach the lips. The aim of this work was to investigate the effect of 4 different emulsifiers (sorbitan olivem, PGPR, monoolein and lecithin) on droplet size (measured using NMR) and the effect of different wax concentrations (carnauba wax (CW), microcrystalline wax (MW), paraffin and performalene) on the melting and material properties using DSC, texture analysis and rheology. Results showed that PGPR produces the smallest droplets ($d_{3,2} \sim 2\mu m$). SEM images show wax crystals are formed at the interface during cooling acting as Pickering particles which creates long term stability (droplet size monitored over 6 months). Melting is affected by the different ratios of waxes. The combinations of both MW and CW have an appropriate melting profile (starting at $\sim 32^\circ C$) suitable for lipstick application. At $32^\circ C$ the wax crystals start to melt allowing the emulsion to spread across the lips. It was also observed that the introduction of water, results in an emulsion product with a lower point of fracture (0.0031 MPa (no H₂O) to 0.0012 MPa (20 % H₂O)), and thus indicating a softer product (measured using texture analysis). The addition of crystalline waxes (paraffin and performalene) was then investigated to consider the effect on material properties. Results showed that the point of fracture increased (0.0028 MPa (20 % H₂O)), this can be attributed to a more rigid wax crystal network being formed. Rheological data showed that the elastic modulus (G') decreases with increasing water content (From 3.97 MPa (10 % H₂O) - 1.95 MPa (40 % H₂O)) and the introduction of either paraffin or performalene increased G' (3.88 MPa (40 % H₂O)). Results also show that for all emulsions investigated (10 - 40 % H₂O), G' is always greater than G'' indicating the formulation is more solid than viscous. In conclusion emulsions made with a polymeric emulsifier (2 % PGPR) are the most suitable for lipstick application, emulsions containing 10 % MW and 5 % CW have the most suitable melting profile and the addition of crystalline waxes adds rigidity to the crystal network of the continuous phase.

Structural evaluation of ice cream produced with cupuassu fat through rheology

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Cupuassu (*Theobroma grandiflorum*) is a fruit that can be found in the Amazonian region and its seed is very rich in fats. The cupuassu fat has found applications in the pharmaceutical, chemical and food industries, for its interesting melting and solid fat content profile. This fat has characteristics similar to cocoa butter. Because of its characteristics, this fat is a strong candidate to be used in ice cream formulations, where for a long time hydrogenated vegetable fat has been used for the benefits it brings to the texture and cost of ice creams. Understanding the use of substitutes for hydrogenated fats and their effects on the structure of the ice cream are of great interest from the industry and the aim of this work. The rheological analysis will allow discussing the influence of ingredients on the structure and composition of the food. Two ice cream formulations were produced using the same methodology and ratio of ingredients where one was used hydrogenated vegetable fat and the other, cupuassu fat. Oscillatory rheological tests before and after the ageing process were performed in a MARS, Haake rheometer + optical microscopy, at a temperature of 10°C to simulate the temperature of the refrigerator used in the ageing process. The cone plate sensor (C35/1Ti polished), with a 0.024 mm gap was used. Analyses were performed at least in triplicate, with a stress amplitude sweep ranging from 0.01 - 100 Pa, with a constant frequency of 1 Hz and frequency sweeps varying from 0.1 -10 Hz, constant stress of 0.1 Pa. A melting curve was obtained by recording the weight of the dripped portion of 100 g of the ice cream samples placed on a metal grid, every 5 minutes for 45 minutes at room temperature. The rheological tests showed similar response from the ageing process to both formulations, and the melting tests showed a slower meltdown of the structure with the ice cream produced with cupuassu fat. The results obtained demonstrated that cupuassu fat is a good substitute for hydrogenated vegetable oil for using in ice cream formulations.

Structural evaluation of ice cream produced with palm fat through rheology

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The ice cream industry had used the hydrogenated vegetable fat in the manufacture of its products for the effects that provides to texture and its low cost. After trans fatty acids have been included among dietary lipids that act as risk factors for health, fat industry have sought to replace the use of hydrogenated vegetable fat. One candidate for that fat is the palm fat (*Elaeis guineensis*). Naturally refined it is free of trans fat. Understanding the use of substitutes for hydrogenated fats and their effects on the structure of the ice cream are of great interest from the industry and aim of this work. The rheological analysis will allow discussing the influence of ingredients on the structure and composition of the food. Two ice cream formulations were produced using the same methodology and ratio of ingredients where one was used hydrogenated vegetable fat and the other, palm fat. Oscillatory rheological tests before and after the ageing process were performed in a MARS, Haake rheometer + optical microscopy, at a temperature of 10°C to simulate the temperature of the refrigerator used in the ageing process. The cone plate sensor (C35/1Ti polished), with a 0.024 mm gap was used. Analyses were performed at least in triplicate, with a stress amplitude sweep ranging from 0.01 to 100 Pa, with a constant frequency of 1 Hz and frequency sweeps varying from 0.1 -10 Hz, constant stress of 0.1 Pa. A melting curve was obtained by recording the weight of the dripped portion of 100 g of the ice cream samples placed on a metal grid, every 5 minutes for 45 minutes at room temperature. The rheological and the melting tests showed a better response from the ageing process and a better formed structure with the formulation produced with hydrogenated fat. It is suggested that formulations produced with palm fat suffers a poorer partial coalescence by its crystallization profile and less membrane destabilization by the emulsifiers.

Stabilization of emulsions by sodium caseinate - κ -carrageenan interfacial complexes

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Proteins extracted from a variety of natural sources have been extensively used in food products due to their emulsifier properties and because of the growing interest in the use of these biopolymers as replacements for the synthetic emulsifiers. Nevertheless, the protein-stabilized emulsions are highly sensitive to environmental conditions, such as the pH, ionic strength and temperature. However, many studies have demonstrated that the stability of emulsions to environmental conditions can be improved by the formation of protein:polysaccharide complexes through covalent bonding or electrostatic interactions. In the latter, multilayer interfacial membranes are formed around the droplets by the addition of a polysaccharide to an emulsion stabilized by an oppositely charged protein, being called layer-by-layer electrostatic deposition technique. Thus, the aim of this work was to investigate the influence of pH and polysaccharide concentration on sodium caseinate/ κ -carrageenan stabilized emulsions in order to improve the emulsion stability by interfacial complexation. O/W primary emulsion containing 0.5 % (w/v) sodium caseinate (Na-CN) and 20 % (v/v) soybean oil was prepared at pH 7.0 by the homogenization using Ultraturrax and high-pressure homogenizer. Multilayer emulsions were then prepared by the mixture of primary emulsion with κ -carrageenan solutions with different concentrations and the pH was maintained in 7.0 or adjusted to 3.5. The emulsions were evaluated in relation to mean droplet diameter, microstructure, droplet surface coverage, zeta potential and rheological properties. The results showed that κ -carrageenan was little adsorbed onto the droplet surface at pH 7 and depletion flocculation was observed when polysaccharide concentration exceeded 0.5 % (w/v). On the other hand, at pH 3.5, emulsions showed bridging flocculation at lower polysaccharide concentrations (0.05 - 0.25 % w/v) and oil droplets completely covered by the polysaccharide from 0.5 % (w/v) κ -carrageenan due to the electrostatic interaction between them at this condition. Stable multilayer emulsions could be formed the highest κ -carrageenan concentration (1 % w/v) in both pH values (7.0 and 3.5) due to the increase of the continuous phase viscosity, maintaining the emulsions kinetically stable. Thus, stable emulsions were successfully produced by using protein-polysaccharide interfacial complexes and the oil diameter, zeta potential and rheological properties of these emulsions were not affected by pH changes.

High-throughput premix membrane emulsification by using metal sieves

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Emulsions are of great interest for many food and non-food products, and various emulsification techniques are applied in practice, although there is still a continuing quest for techniques that use less energy and give good control on droplet size (distribution). Here we present results obtained with premix membrane emulsification using nickel sieves, which have a uniform pore size (distribution), and can be used at relatively low transmembrane pressure. Oil-in-water emulsion was prepared using different sieves having rectangular and squared pores, and scaling relations were derived for premix emulsions consisting of 5 % n-hexadecane in water having 0.5 % (v/v) Tween-20 as surfactant. The transmembrane fluxes were rather high ($1000 \text{ m}^3/(\text{m}^{-2}\text{h}^{-1})$ at 1 bar applied pressure) while a reasonable span (around 1) of the droplet size was found. There was no indication of fouling in the process, which indicates that the process is tolerant to product and conditions. A master curve was derived for the Weber number as a function of the transmembrane pressure normalized on the Laplace pressure of the emulsion before emulsification. This curve comprises all process parameters and allows estimation of the emulsion droplet size. The results show that these metal sieves enable an appreciable reduction in droplet size, considerably smaller than the smallest dimension of the pores. Currently we apply the technology to food grade systems and evaluate whether the master curve can also be used to describe these emulsions.

Rheological properties and short-term stability of oil-in-water emulsions formed with milk whey proteins: Influence of ι -carrageenan and κ -carrageenan

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The stability of oil-in-water emulsions can be improved by polysaccharide molecules. Particularly, emulsions formed with carrageenan proved to be more stable compared with those stabilized with proteins alone and the stability can be enhanced considerably by adding ι -carrageenan rather κ -carrageenan. The influence of ι -carrageenan and κ -carrageenan concentration on the properties of oil-in-water emulsions stabilized by milk whey proteins was investigated by measuring the particle size distribution, the creaming stability and the rheological properties of the bulk and the interface, in order to determine the experimental conditions, where both polysaccharides can be used to improve emulsion stability. Emulsions were formed using 3 wt % of commercial milk whey proteins in the aqueous phase, to have a considerable amount of protein remaining in the aqueous phase after emulsion formation, whereas carrageenan concentration was varied from 0 up to 0.2 wt% in the aqueous phase to improve the stability. The flow behavior of the prepared O/W emulsions was highly influenced by the type of carrageenan present in the continuous phase but independent of their droplet size distribution. Increases in flow properties and the excellent stability towards phase separation found for the stored ι -carrageenan emulsion combined with the increase of the elasticity of the interfacial films of milk whey proteins/ ι -carrageenan adsorbed at the O/W interface than that of pure protein suggested that ι -carrageenan could retard the coalescence phenomena. Thus, the results support the ability of ι -carrageenan and the inability of κ -carrageenan in reducing partial coalescence either by providing a sufficiently thick continuous phase or by acting as a protective coating for oil droplets.

Influence of manufacture method, pH and carboxymethylcellulose on simulated digestion of O/W emulsion stabilized by whey protein

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Whey proteins are well known to facilitate emulsions formation and improve their stability by reducing the interfacial tension and by forming a protective membrane around fat droplets. Polysaccharides are often added to the oil-in-water emulsions to create a desirable texture and to stabilize emulsion droplets against gravitational separation. Emulsification of protein is one of processes that changes protein structure and it is highly likely to influence the way that the protein is digested. The aim of this study was to compare the influence of method of emulsion manufacture, pH and carboxymethylcellulose addition on the digestion of whey proteins stabilized emulsion. Five systems were studied: I type pH 5, II type pH 5, I type pH 7, II type pH 7 and control. I type of emulsion was made mixing appropriate amounts of CMC and WP solutions for 5 min at 24,000 rpm, the pH was adjusted to 5 or to 7 and mixture was stored for 1 hour. After 1 hour WP and CMC mixture was homogenized for 5 min at 24000 rpm with rapeseed oil. II type of emulsion was made homogenizing the appropriate amounts of WP solution and rapeseed oil for 5 min at 24000 rpm, the pH was adjusted to or to pH 7 and the mixture was stored 1 hour to help make an interaction between components. After 1 hour whey protein and oil mixture was stirred for 5 min at 24000 rpm with the CMC solution. Control emulsion prepared without addition of CMC. Emulsions were digested using pepsin under simulated gastric conditions (37°C, pH 2 with continuous shaking at approximately 100 rev/min for 2 h) and pancreatin under simulated duodenal conditions (37°C, pH 7 with continuous shaking at approximately 100 rev/min for 2 h). Digestibility rate of emulsion was characterized by droplet size distribution, protein hydrolysis rate, lipid hydrolysis rate, rheological characteristics of emulsions and simulated digestion fluids mixture. Higher amount of peptic hydrolysis products were observed digesting control emulsion. CMC saved its protective function -increase viscosity and reduce movement of emulsions compound. Increased viscosity reduced proteins availability to pepsin, oil droplets of emulsions with CMC had changed quite modest during the simulated digestion.

Comparing different commercial fenugreek galactomannans for the production of emulsions with high intensity sonication: Effect on physical stability and rheological properties

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In this research high intensity sonication (20kHz, 4min, 70 and 90 % amplitude) was used to prepare secondary whey protein model submicron emulsions (pH \approx 4). Primary/coarse emulsions prepared with a high shear device contained 2.7 wt% whey protein isolate (WPI), 20 wt% olive oil and four different types-fractions of commercial fenugreek gums (FGA, FGB, FGH and FGD) at 0.25 and 0.5 wt% concentration. In addition, emulsions containing commonly used galactomannans such as locust bean (LBG) and guar gum (GG) were prepared at the same concentrations. All emulsions were stored at 5°C and their physical stability was evaluated for 10 days by using a multiple-light scattering/optical/turbidometric method. Fenugreek gum fractions differed in galactomannan (FGH > FGD > FGA > FGB) and protein content (FGB > FGA > FGD > FGH). FGD is a debittered fraction, not exhibiting any spicy odor or bitter taste. The galactomannan content and viscosity of 1 % gum solutions for FGA, FGB and FGH followed the order FGH > FGD > FGA > FGB, while their protein content order was FGB > FGA > FGD > FGH. At low gum concentration (0.25 wt%) the stability of emulsions containing fenugreek gum followed the order FGB > FGA > FGH > FGD, while at higher concentration (0.5 wt%) the trend was changed to FGH \gg FGD > FGA > FGB. This suggests that at low concentrations the stability was primarily influenced by the gum protein content, whereas at higher concentrations stability was influenced by gum viscosity. This fact was confirmed by preparing 0.5 wt% gum emulsions in the absence of WPI which were observed for 20 min. FGB emulsions had higher back-scattering values that decreased in a lower rate with time in comparison to FGA and FGH. At 0.25 wt% gum concentration, FGD emulsions were the most stable of all (Serum index, SI = 17.6%), whereas at 0.5 wt% GG emulsions performed the best (SI = 2.8%). The effect of sonication on the viscosity of 1 wt% gum solutions was evaluated. Steady stress flow curves of sonicated gum solutions revealed that ultrasonic treatment significantly reduced their viscosity. Finally, the effect of gum type and concentration on oil droplet size is further discussed. This study aims at the potential substitution of common stabilizers (locust bean, guar) in salad dressing products with fenugreek galactomannans for nutritional and financial reasons.

Localization of particle in oil-water emulsion

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We investigated the behavior of heavy particle (particle-oil $\sim 0.7 \text{ g/cm}^3$) in oil/water emulsion. Drop of oil suspended with particle was generated in water. According to difference in chemical affinity, particle in oil drop was migrated into the other phase. In the case of hydrophilic particle, it was located in the interface and then they migrated to water. When it migrated to the other phase, the motion of particle along the interface induces its aggregation and the aggregated particle migrated to water phase. On the other hand, when hydrophobic particle was suspended in oil drop, it hardly migrated to water phase. Though hydrophobic particle was settled down near the interface, it did not migrate to the water phase in an observation time of 2 hours. The sediments of hydrophobic particle in an oil drop cause heterogeneity of density. The dense particle distribution changed the shape of oil drop. When the density of particle is similar to oil, the location of particle to the interface did delay due to Brownian motion between particles in an oil drop. The migration to the other phase was initially occurred by the particle near interface different with heavy particle.

Surface rheology of interfaces stabilized by different structures of BSA

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The interfacial rheology of three different structures of bovine serum albumin (BSA), i.e. BSA fibrils, soluble BSA pectin complexes, and native BSA, were investigated. At a bulk concentration of 0.05 % (w/w), there is only a small difference in the equilibrium surface tension of O/W interfaces stabilized with these three structures. However, there is a significant difference in the dilatational elastic and viscous modulus at low strain, which becomes less pronounced at high strain. Interfaces stabilized by BSA pectin complexes show the highest dilatational elastic and viscous modulus, followed by those stabilized by BSA fibrils and native BSA, for all strains measured (0.005 - 0.40). Frequency sweeps show that in the applied range of frequencies (0.005 - 0.1 1/s), the response of interfaces stabilized by complexes is significantly different from the response of interfaces stabilized by monomer and fibrils. The loss tangent of the former is considerably higher and decreases with increasing frequency, whereas the latter two display a loss tangent that is almost constant (~ 0.2) in the applied range of frequencies. The interfacial shear rheology of interfaces O/W interfaces stabilized by complexes, fibrils and native monomers is measured as a function of bulk concentration. For all concentrations (0.002 - 0.2 % (w/w)), the elastic and viscous modulus of interfaces stabilized by complexes is the highest, followed by fibrils and monomer. The elastic and viscous modulus of BSA fibrils and monomers increase as a function of concentration, up to a concentration of 0.2 % (w/w). However, the elastic and viscous modulus of the complex reaches a plateau at a concentration of 0.03 % (w/w), which suggest that at this point the interface is already saturated. This study has shown that O/W interfaces stabilized by BSA fibrils and BSA pectin soluble complexes have a surface rheology remarkably different from interfaces stabilized by native BSA. These aggregation states of BSA may find application as emulsifiers or foam stabilizers.

Influence of arabic gum on the stability of ginger oil-in-green tea extract emulsion prepared by ultrasound

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The effect of Arabic gum concentration (5, 7 and 10 %wt) on the stability and the rheology of Ginger oil (2 %wt) in Green tea extract emulsion in pH = 4.8 was studied. Mean droplet diameter, specific surface area, flow behaviors and Creaming stability were measured in 4 weeks. By increasing the gum concentration from 5 to 10 %. Mean droplet diameter ($d_{4,3}$) decreased and Creaming stability increased ($p < 5 \%$). The results indicated that along 4 weeks droplets size (d_{43}) increased but specific surface area and Creaming stability decreased ($p < 5 \%$). In emulsions with 5 - 7 % Arabic gum, Newtonian flow behaviors and in emulsions prepared with 10 % Arabic gum non-newtonian shear-thinning flow behavior were observed ($p < 5 \%$).

Easing formulation of emulsions and test for long term stability using bottom focused back scattering

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Selection of efficient emulsifiers, their optimum concentration and of the processing conditions is a demanding and time consuming process. Evaluation of emulsion stability can be eased when focusing on the changes in droplet concentration right at the bottom part of the emulsion samples. Furthermore the level of the back scattering signal is directly related to the dispersion degree. Thus the initial back scattering signal can be used to compare emulsification quality and to detect destabilization due to aging. A new multisample approach using light backscattering signals is presented. It allows for fast measurement of stability and/or demixing phenomena and separation kinetics of up to 8 different samples simultaneously, by monitoring the concentration change in a very small region of interest at the bottom of sample cell. The method was applied to trace the optimum emulsifier concentrations for silicone oil emulsions to evaluate the influence of processing time and intensity. Further the method was used to detect the very first changes of sample dispersity after accelerated aging.

Effect of stearic acid and glycerin on properties of caseinate-based edible films

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Edible films and coatings are used to improve appearance and prolong shelf life of food products. Hydrophilic coatings based on proteins and polysaccharides provide good barrier to gas transmission but a poor barrier to water vapor. In order to decrease moisture migration from surface of food products, hydrophobic substances such as waxes and fatty acids are incorporated in coating formulations. In this study, the effect of plasticizer and stearic acid concentrations on water vapor transmission rate and water vapor permeability of sodium caseinate-based edible films were determined by measuring the amounts of water vapor transmitted through the films. Film-forming emulsions were also tested for flow rheological properties and surface tension. The results revealed that change in concentrations of sodium caseinate, glycerin and stearic acid had significant effect on water vapor transmission rate ($p < 0.01$). Formulations containing 8.20 gr/100 gr sodium caseinate, 1.54 gr/100 gr glycerin and 2.05 gr/100 gr stearic acid show the lowest water vapor transmission rate of 0.69 g/hm². All film-forming emulsions designated non-Newtonian and shear thinning flow behavior. This study also indicated a significant interaction effect of sodium caseinate and stearic acid on the surface tension of the film-forming emulsions as well as water vapor transmission of films ($p < 0.01$).

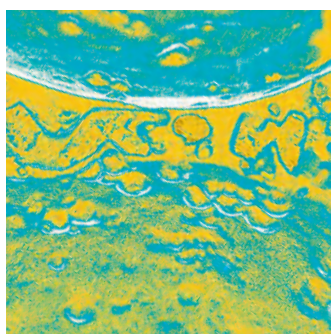
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Semi-Solid Foods

Physical and sensory descriptors of oral food breakdown pathways

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During the mastication, the physical and chemical properties of the food change until the formation of a food bolus suitable for swallowing. In the course of this process, the sensory perception evolves as well. The purpose of this work was to characterize the food bolus properties in the course of the mastication thanks to physical and sensory measurements, with a final aim to investigate the impact of the physical properties of the food bolus on the sensory perceptions. The mechanical (small and large deformation) and water content properties as well as the sensory texture properties of 5 wafer prototypes were investigated at different mastication times. The statistical effects of the subjects, the products and the mastication time as well as their interaction on the different physical and sensory parameters were calculated through ANOVA. Our results illustrate the main function of the mastication, i.e. to prepare a soft, cohesive and elastic food bolus that can be safely swallowed. The different products were broken down differently to reach similar physical properties just before being swallowed. While sensory attributes hard, gritty, crunchy and crispy evolved differently for different products until the point of swallowing, the attributes aeration, fluidity, melting and dryness reached similar intensities at the point of swallowing. The mastication time highly differed among subjects meaning that the mastication strategy to prepare a food bolus ready to be swallowed depends on the subject. Our results suggest that designing products with different moisture content and moisture uptake capacity throughout the consumption episode could be an option to modulate the dynamics of perceptions in the mouth. Furthermore, a better understanding of the physical changes of the food bolus needed to reach a swallowable stage will help design specific products adapted to the needs of specific populations such as children, elderly or dysphagic people.

A comparative study investigating the differences in component mobility during the initial stages of maturation of Mozzarella and cheddar cheese

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During the first few weeks following manufacture cheese undergoes significant changes in structure and arrangement of components. One of the key drivers involved in these changes is the hydration of the protein phase due to the binding of water. As changes in structure play an important role in the functional properties of cheese, an understanding of the mechanisms involved is critical. The first objective of this study was to identify the changes occurring in the structure and component mobility of both Mozzarella and cheddar cheese during the first few weeks immediately after manufacture. We also analyzed the differences between the cheeses and investigated potential mechanisms affecting the changes. We assessed fresh samples of Mozzarella and cheddar cheese over a number of weeks. The changes in component mobility were determined by assessing the T2 relaxation using NMR spectroscopy, dielectric properties over a frequency range of 20 MHz to 1.3 GHz, pH, assessing the quantity of expressible liquid as well as examining the structure using confocal microscopy. The experiments were all carried out at 20°C. During this period, there is significant movement of water within the cheeses from a state where it is more mobile to a state in which it becomes bound to the protein. This was observed visually where free water in confocal images on day one disappears as the protein swells to absorb it. This trend was identified using other measurement techniques that indicated that the water within the cheeses was able to lose a greater amount of energy as it became less mobile. The results from this study indicated that both cheddar and Mozzarella undergo significant changes in component mobility over the first few weeks following manufacture.

An investigation into the effect of maturation on the dielectric and rheological properties of Mozzarella cheese during heating

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The structure of low moisture part skim Mozzarella undergoes a number of major changes during the maturation process that impact on its functional properties. As Mozzarella is predominantly consumed in a molten state, it is important to understand how the functional properties of the cheese are affected by heat during the maturation process. The objective of this work was to investigate how the dielectric and rheological properties of Mozzarella change during its maturation and to relate these changes to the functional properties of the cheese. Relationships between dielectric and rheological properties during heating were also investigated. We measured the dielectric properties over a frequency range of 200 MHz to 1.3 GHz to assess the ability of the cheese to store and dissipate electrical energy. The rheological properties of the cheese were also assessed to identify the changes in the loss and storage moduli with temperature. Both the dielectric and rheological properties were assessed between the temperatures of 20 and 90°C. The melting behavior of the cheese was also assessed. Heating resulted in a shift in both the dielectric and rheological properties as the components within Mozzarella gain more energy and become more mobile. During the maturation process changes were observed in both the dielectric and rheological responses and how each responded to the heating profile. These changes were assessed against each other and compared to the meltability of the cheese. Both the rheological and dielectric properties of Mozzarella were found to change significantly during maturation. Relationships between the changes observed were identified and linked to the functionality of the cheese.

Oral processing of two milk chocolate samples

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Chocolate, from a food material science perspective, represents a fat continuous composite. The continuous fat matrix is predominantly composed of cocoa butter and emulsifiers such as lecithin. Other ingredients in a basic milk chocolate include sugar, milk solids and cocoa solids. This research concerns two milk chocolates which significantly differed in mouthfeel despite being of identical composition and manufactured to the same melt viscosity. Differences in mouthcoating and related attributes such as time of clearance from the oral cavity were most discriminating. This prompted an investigation into the oral processing of chocolate to understand the observed differences in mouthfeel. It was hypothesized that changes in the salivary properties of the panelists before and after eating these two chocolates may be observed depending on which chocolate was consumed. This was not the case. Upon ingesting chocolate, the continuous product matrix begins to melt and the mechanical action of oral processing disintegrates the chocolate. Presence of saliva facilitates phase inversion into oil-in-water emulsion microstructure. For the two chocolate samples investigated, their microstructure following oral processing was clearly different when evaluated by optical microscopy and particle size analysis. The sample found to be more mouthcoating appeared less flocculated than the other milk chocolate sample. Flocculation facilitates clearing of the mouth from food residues thus counteracting mouthcoating. The more mouthcoating chocolate also melted faster during consumption. The results led us to hypothesise that interfacial processes between the droplets of the phase inverted chocolate sample and the oral cavity drive mouthfeel perception. Involvement of interfacial and surface properties in narrow gap situations, as is the case between the tongue and upper palate when degree of mouthcoating is evaluated, implies that it may be useful to characterize the friction properties of chocolate. These were evaluated in-vitro at in-mouth temperature using a commercial tribology attachment on a rotational rheometer. Stark differences in the tribological behavior were found in the domain where the properties strongly depend on the characteristics of the friction surfaces and how they interact with the sample. In conclusion, novel correlation between a physical material property of a complex food composite and sensory behavior have been uncovered.

Quantitative analysis of cheese eye volume by means of X-ray computed tomography

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X-ray computed tomography (CT) is widely used in medicine but more recently also in technical applications such as structural mechanics, failure analysis or in archaeology. The CT system allows to distinguish materials of different density and therefore cheese eye volumes can be quantified without destruction of the cheeses. Eye formation is an important quality aspect of Swiss-type cheese and also of many semi-hard cheeses. The existing conventional (two-dimensional) X-ray system only allows a semi-quantitative determination of the eye-volume. To find differences in the activity of metabolic pathways with gas formation of different microorganisms in cheese for the development of eye forming cultures and to investigate other influences on eye formation, CT was applied to quantitatively determine the eye volume. A Philips CT system was used in collaboration with the veterinary hospital at the University of Bern (VetSuisse). To be able to quantify the eye volume, a specially designed software had to be developed. A CT system recognises the density difference between gas and cheese body, the total volume of the cheese as well as the total eye volume inside the cheese can be calculated. The CT system was validated with cheeses made with the inclusion of hollow polypropylene balls of a diameter of 10 and 20 mm, respectively. Six normally eyeless Gruyere type hard cheeses with an increasing number of plastic balls and hollow volumes from 0 to 33.3 ml and 0 to 309.1 ml, respectively, were produced and ripened for 30 days within a plastic film. The eye volume calculated with the CT system showed a good correlation ($R^2 > 0.991$) in comparison with the given hollow volume of the added balls. In the second part of the study, the CT system and the software were applied for the quantification of the eye volume in semi-hard and hard cheeses in a non-invasive manner. The influence of different microorganisms on the eye volume in cheese was investigated by CT [1 - 4].

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Stickiness of bread dough

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Sticky dough is of particular economic concern in industrial bakeries, with throughput of thousands of loaves an hour, due to the build up on machinery leading to loss of both time and materials. Stickiness is a combination of adhesion, the interaction between a material and a surface, and cohesion, the interactions within the material. It is therefore a result of a combination of surface and bulk rheological properties. There is no universal measure for stickiness within the food industry and reliable measurement is hard to achieve due to the complex viscoelastic nature of dough. Stickiness is usually determined subjectively based on the results of manually handling the dough. Any measurements that are done are generally empirical and limited in applicability to the conditions under which the test was performed. Therefore, a test to measure dough stickiness is being developed to overcome these problems and provide a quantitative measure of stickiness. Squeeze flow between lubricated and non-lubricated plates is being investigated as a means of testing for stickiness. The geometric orientation of a squeeze flow test ensures that there is continual generation of fresh material from the bulk at the interface so that the adhesion may be measured for a dough. Measurements using non-lubricated plates will show the results of both cohesive and adhesive forces. Such measurements are then compared to a lubricated sample (induced wall slip) to account for the cohesive aspect. Additionally, information about these cohesive forces within the dough can also be derived from this approach.

Structural investigation of crystallisation phenomena induced by organogelation in vegetable oils

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Solid fats are relevant from the industrial point of view because they can give the desired texture to the final foods and are suitable for the existing process conditions. Nevertheless the fats commonly used, i.e. saturated or hydrogenated fats, can have negative health effects (such as cardio-vascular diseases or increase in cholesterol levels) and, as a consequence, their consumption should be reduced. Potential replacers are based on the organogelation of liquid oils, rich in unsaturated fractions and therefore less dangerous for the human health. A deeper investigation of the microstructural effects of organogelators is, therefore, important to design novel products having the specific rheological properties. In this work a structural investigation of organogels, based on olive oil, cocoa butter and commercial monoglycerides of fatty acids (Myverol), was carried out by coupling rheological methods (i.e. small amplitude oscillations) with Differential Scanning Calorimetry (DSC) and Nuclear Magnetic Resonance (NMR), aiming at evidencing the crystallization phenomena and the change in material structure due to formulation and operating conditions. It was found that crystal formation is determined by the Myverol fraction and a crystallization temperature was estimated by a sharp change in dynamic moduli. It was observed that the transition temperature was function of the Myverol amount whereas the material consistency is affected by both Myverol and cocoa butter/oil ratio. Similar phenomena were identified by DSC and NMR methods and the transition temperatures, being approximately the same of the rheological ones, confirmed the observed crystallization process. The structural analysis evidenced, depending on the observed temperature range, the presence of different polymorph crystals that caused relevant changes in rheological parameters during the temperature ramp tests. The NMR techniques, adopted in the present work, gave a deeper understanding on the organogel structure confirming the results obtained through the rheological characterization.

Comparison of the structural behavior of fillings prepared with varied fat content of cupuassu fat and cocoa butter

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The chocolate is a product consisting of a chocolate mass or nucleus consisting of fillings made from fruit, fondant or fat, covered with chocolate. The use of lipid substances in the preparation of fillings should be done so as not to affect the integrity of the chocolate, the biggest challenge is to use a fat compatible with cocoa butter and that does not have a cost as high as the same. Cupuassu fat presents similarity with cocoa butter and can be used as an alternative in the production of fillings. The present work studied the structural behavior of chocolate fillings for prepared with different cupuassu fat and cocoa butter concentrations. Six fillings formulations were prepared with concentrate orange juice, white chocolate, fondant, fat base and ethanol 40°GL. The rheological behavior of the formulations was measured by oscillatory tests at the Haake MARS rheometer with the test taken in three steps: oscillation with a controlled tension (1 Hz, 0.01 Pa), rotational test with controlled shear rate (50 1/s) and oscillation with a controlled tension (1 Hz, 5 Pa). The samples show an elastic response, which may be related to the particle size of sugar (fondant) in the formulation. The sample with 10 % cupuassu fat showed higher viscosity values compared with the sample containing the same concentration of cocoa butter. The complex viscosity increases during the time analysis for all samples. For all samples, the shear time and/or the applied shear rate were not sufficient to let the viscosity reach at a constant level, suggesting the necessity of a long time to recover the structure completely. Thixotropic samples have a weak structure, which as the result of constant or dynamic shear may be temporarily destroyed resulting in a viscosity drop with time. It is characteristic for these thixotropic samples that can recover their original structure when left at rest for some time. Samples with cocoa butter regain most of its structure more quickly than the other. The general behavior was the same to all samples. Cocoa butter and cupuassu samples presented distinct behavior.

Addition of modified waxy maize starch accelerates milk fermentation and improves structure recovery after shear

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The objective of this work was to investigate the effect of modified waxy starch on the structure and physical properties of set and stirred yoghurt gels. Despite their widespread use in dairy products, very little is known on the effect of modified crosslinked starches on the structure and physicochemical properties of fermented milk gels. Milk or milk-starch mixtures (containing 1 or 2.5 % modified crosslinked waxy maize starch) were pre heated and homogenised at 55°C and then heated at 95°C for 5 min, using a pilot scale system. Milk was incubated with a commercial yogurt culture and fermentation was followed by dynamic oscillatory testing in rheometer at 40°C until pH 4.6 and after cooling down to 4°C. Frequency sweep tests at 40 and 4°C and strain sweep tests at 4°C were also performed. Although 1 % modified starch did not significantly alter the gelation time, increasing the starch concentration to 2.5 % induced gelation at a significantly shorter time compared to control. The storage modulus (G') at 40 and 4°C increased linearly with increasing the starch content, with the increase being higher at low temperature. However, the frequency and strain dependence of dynamic parameters of yogurt gels (i.e. G' and G'') were not affected by the presence of modified waxy maize starch. The final gels showed similar shear thinning behaviour with the milk gel containing 2.5 % starch having the highest viscosity. Fermentation was also carried out in a container and the yogurt gels were then broken and transferred to the rheometer to measure the recovery of the modulus (G') after shear at 500 1/s for 20 min. Milk gels showed the highest recovery of structure at 2.5 % modified starch followed by 1 % and control milk gels (G' values one hour after stirring cessation were 13.7, 8.7 and 3.3 Pa, respectively). Confocal images of set and stirred yogurt gels showed intact swollen starch granules embedded in the protein network with strands that became thicker and denser as the starch level increased.

Quantification of baking: Learning on methods and ingredient functionality

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Quantified baking structures in yeast-raised toast bread are derived and characterized from a DOE controlled multiple stage study covering the baking process methodologies and a representative ingredient range. The (causal) baking process from dough throughout mixing, baking and of the final bread is fully covered by multiple methodologies generating a comprehensive map of quantifiable specifics which are used to discriminate between added bread improving ingredients. Splitting information is partially available for methodologies with respect to \pm yeast, \pm freezing, \pm resting time/holding time and \pm shocking. The ingredient range is covered by emulsifiers, enzymes and hydrocolloids with known and well documented bread improver effects allowing an interpretation of specifics in baking terms. The quantified baking structures are mapped in different scales including the original measurement scale, a uniform size scale, a cost-in-use scale and sustainability penalized scale. Dough (bakers evaluation (stickiness, extensibility), farinograph, penetration, Kieffer, dough inflation system), heating/cooling simulates baking (rapid visco analyzer, mixolab, rheometer oscillation throughout heating/cooling), and final bread (specific volume (normal and stressed bread), bakers evaluation (firmness, energy, cracks, crumb structure), rapid visco analyzer, texture profile analysis, c-cell, sensory analysis, nuclear magnetic resonance, surface laser scanning, surfscan, and videometer) characterization was done on yeast-raised bread added: emulsifiers (DATEM (3), DIMODAN (2)), enzymes (amylase (1), fresh-keeping enzymes (4), lipase (3), xylanase (3), oxidase (2), protease (2)), hydrocolloids (guar, pectin), emulsifier and hydrocolloid (DIMODAN HR and guar – effect of encapsulation), additional water addition, or ascorbic acid. Dough is described using two variables (farinograph and Kieffer), baking simulation by one variable (rapid visco analyzer, mixolab, or rheometer oscillation throughout heating/cooling), and bread functionality five variables to be fully described (specific volume, two crumb structures, fresh-keeping/ moisture, and shock stability). Baking (heating/cooling) information is best at discriminating ingredient functionality. An overview of total ingredient functionality is given based on whether gluten is strengthened or how starch gelatinization is influenced. DIMODAN delay starch gelatinization opposite fresh-keeping enzymes. DATEM and lipase has similar performance. Cost-In-Use/ Sustainability-In-Use aspects are calculated.

Improved emulsification of phospholipase modified egg yolk is induced by lyso-phospholipids and improved protein properties as shown in mayonnaises

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Phospholipases catalyse the hydrolysis of phospholipids; phospholipase A2 (PLA2) specifically assists in the removal of the fatty acid on the sn-2 position of the phospholipid, leading to the formation of lyso-phospholipids. DSM Food Specialties produces several phospholipases for specific applications. This poster focuses on the changes in egg yolk after PLA2-catalysed hydrolysis, and how this improves emulsification behaviour, leading to firmer, more heat stable and creamier mayonnaises and dressings. It was always understood that this is due to the improved emulsification behaviour of lyso-phospholipids. Here, we present proof that also changes in protein properties play a major role.

Physicochemical, sensorial and textural characteristics of ice cream enriched with persimmon puree

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The main aim of the present study was to produce an ice cream enriched with persimmon puree and to determine the physicochemical, sensorial and textural characteristics of final product. In this respect, different concentrations of persimmon puree (8, 16, 24, 32 and 40 % w/w) were incorporated to the ice cream mix and the mixture was aged for 20 h at 4°C. After that, the mixture was processed to the ice cream and the final product was hardened. Addition of persimmon puree caused a significant change in the physicochemical, sensorial and textural characteristics of ice cream. Total dry matter, ash, fat and protein content of ice cream decreased significantly ($p < 0.05$) with the increase of persimmon puree concentration. Viscosity values of ice cream mix containing persimmon puree was found to be lower than that of the control sample ($p < 0.05$). Hardness values of ice cream samples enriched with persimmon puree was determined to be lower compared to control, but it increased with increasing persimmon puree concentration. Hardness value of control sample was recorded to be 16.5 kg, but the hardness value of ice cream containing 40 % persimmon was found to be 11.4 kg. Similarly, persimmon puree addition provided a significant increment in work of penetration, but the highest value was found in control sample. Sensory evaluation of samples revealed that the addition of persimmon puree increased the overall acceptability until certain concentration.

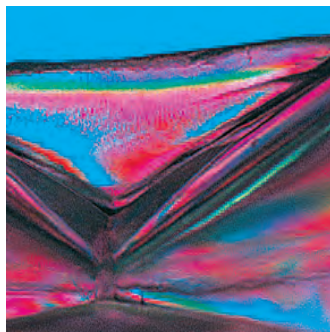
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Rheological Methods

The dynamic rheology of wheat – lupine flour dough's and their behavior changes during backing processes simulation

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Legume seeds are an abundant source of proteins and, among them; lupine is one of the richest. Lupine seed deserves great interest due to its chemical composition and augmented availability in many countries in recent years. The aim of this research was to study the effect of temperature on dynamic rheology of wheat lupine flour dough's. The lupine flour was used to replace 5, 10, 15 and 100 % of wheat flour. A rheometer (UDS 200, Physica, Anton Paar GmbH, Austria Europe) was used for measuring the rheological properties of dough samples. Temperature dependence of storage (G') and loss (G'') moduli as well as loss tangent ($\tan\delta$) were measured by heating the systems from 15 to 90°C. The temperature gradient was 1°C/min on heating scan, while the strain was fixed to $\gamma = 10^{-3}$ with a constant frequency of 1 Hz. This heating rate was chosen in order to secure gradient in the sample and to detect the occurring processes such as denaturizing of proteins, gelatinization of starch and the mobilization of the water. The oscillation measurements results of wheat flour dough showed that a quick and great changes of internal structure development in range 50 and 72.5°C, which was attributed to denaturizing of protein and gelatinization of starch. On the other hand, lupine flour dough results showed that no changes of internal structure development because lupine contained almost no or minute amounts of starch. For the composite flour dough at different addition, the majority of wheat flour proportion leads to similar behavior attitude compared with wheat flour dough. While, there were significant difference between composite flour dough themselves, in range below 50 and above 80°C the storage and loss modulus decreased with increase lupine flour concentration. The viscoelastic properties of dough could have been retained unchanged, because the wheat is dominant portion. Moreover, the nutritional values have been improved by the addition of lupine. In general, it is clear that the addition of lupine flour up to 15 % is not affected on the rheological properties of the dough.

Effect of partial replacement of wheat flour with Lupine flour on the dynamic rheological properties of batter cake

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Lupine seeds have a long tradition of use for human consumption in some European and South American countries. However, in recent years, sweet lupine seeds have been utilized in new and innovative ways due to their nutritional and food processing qualities. The aim of this study was to investigate the effect of partial replacement of wheat flour with lupine flour on the dynamic rheological properties of batter cake. The lupine flour was used to replace 5, 10, and 15 % of wheat flour. Rheology measurements of the cake batter were carried out just after its preparation, in a rotational viscometer (UDS 200, Physica, Anton Paar GmbH, Austria) coupled with a circulating cooling bath (Lauda RE312, Lauda-Königshofen, Germany). Operation, including temperature control and data handling, was conducted using PC-based software provided by the rheometer manufacturer (Rheological Instruments AB, Lund, Sweden). Quantities of 18 g of batter were subjected to a shear rate increasing from 0.1 to 10 s⁻¹ in 5 min at constant temperature 25°C. Viscosity was measured as a function of shear rate. Measurements of the elastic (storage) modulus (G') and loss modulus (G'') were continuously recorded. From these measurements, viscosity changes in the cake batter and rheological properties of the product were determined. The amplitude sweep measurement results showed that a linear viscoelastic behavior of module at the range of 10⁻⁴ – 10³. Oscillation measurements results cleared that the storage modulus is greater than the loss modulus and the measurement curves of module run nearly parallel in frequency sweep. Increase in the share of lupine flour additions caused the G' and G'' curves to shift towards higher values compared to the wheat flour dough. while the tan curve tended to shift towards lower values. So, wheat flour can carry up to 15 % (w/w) of lupine flour and produce cake has an acceptable dynamic rheological properties.

Physicochemical and rheological properties of xylanase supplemented whole wheat dough and bread

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Bread is the most popular cereal based food product throughout the world. Bread is manufactured from white wheat flour. At the time of milling, substantial amount of nutrients are lost as most of these nutrients are concentrated on the bran layer of wheat. Our aim was therefore to make more nutritious whole wheat bread. Deleterious effect of fibrous whole wheat dough is decreased loaf volume due to lower gas retention, objectionable gritty texture and unsuitable taste and mouth feel. These adverse effects can be counteracted by supplementation of hemicellulase enzymes. To overcome the problems associated with whole wheat bread texture, partially purified xylanase was added and its effect on the quality attributes of the bread during storage at room temperature ($25 \pm 2^\circ\text{C}$) and refrigerated temperature ($4 \pm 1^\circ\text{C}$) was studied. Effect of xylanase on rheological properties of dough was explored by testing dynamic rheological parameters and stress relaxation behavior was observed by creep measurement. Change of molecular structure and microstructure in xylanase supplemented dough and bread were investigated by Fourier transformation infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The linear visco-elastic region was observed from 0.1 to 1 % strain level. Reduction of elastic (G')- and viscous (G'')- modulus, was observed above 1 % strain, indicating the breakdown of structure of both untreated- and xylanase-supplemented wheat dough. The frequency sweep experiments performed under dynamic condition (in linear visco-elastic region) revealed that in both control and xylanase-supplemented dough formulation, the elastic modulus in the entire range of frequency was higher than viscous modulus. Our results represent the adequacy of fitting of dynamic moduli in Power law model and weak gel model. Creep compliance data revealed that control dough is stronger than xylanase-supplemented dough. Creep test data of control and xylanase-supplemented dough were fitted using Peleg, Kelvin, and Burger models. Considering the values of coefficients and R^2 values, Peleg model as well as six element Kelvin model described well the creep behavior of control and xylanase-supplemented dough samples. Xylanase supplementation tremendously improved the quality of bread.

A systematic analysis of protein covered air/water and oil/water interfaces

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Bovine serum albumin (BSA), β -lactoglobulin (bLac) and lysozyme are often used in the food industry to stabilize foams and emulsions [1, 2]. They adsorb at the interface and form a stable interfacial film. In order to apply them in a targeted manner with respect to their specific properties, it is necessary to analyze them under comparable conditions. Therefore, we study the protein specific adsorption kinetics and rheological properties of the resulting viscoelastic layer at two different interfaces. The mentioned proteins above were analyzed for various concentrations at an air/water (A/W) and oil/water (O/W) interface, where limonene was used as the oil phase. Adsorption kinetics and dilatational properties of the interfacial layer were determined using a drop profile analysis tensiometer (PAT). Shear rheological measurements were performed during adsorption with a rheometer equipped with a biconical disc. During dilatation a viscoelastic layer with mainly elastic properties is observed throughout. The layers show different discontinuities for both interfaces. Observe that the drop surface shows wrinkles after the dilatation at the O/W interface. At an A/W interface the surface pressure increases more slowly at the beginning of adsorption and the maximal surface pressure is reached earlier in contrast to an O/W interface [2]. For lower concentrations adsorption kinetics decrease and an induction time is measured for all proteins. At the O/W interface all layers formed show no viscoelastic region during the first hour of adsorption for low concentrations compare to the A/W interface. Notice that at the O/W interface over time a change from pre-gel like character to rubber-elastic character can be observed. After reaching a maximal surface pressure the layer behavior does not change anymore for both interfaces and the interfacial layer shows rubberelastic properties. The adsorption process of each protein and rheological properties of the formed interfacial layer depend on bulk concentration nature of the interface used, and molecular characteristics such as structure, size and stability of the proteins.

[1] P. Fischer, P. Erni: Curr Opin Colloid Interface Sci. 12 (2007) 196-205.

[2] P. Erni, E.J. Windhab, P. Fischer: Macromol. Mat. Eng. 296 (2011) 249-262.

How to measure and distinguish the contribution of protein structure elements with different length scales with regard to the rheological behavior of heterogeneous protein matrices

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Proteins play an important role in food for their nutritional value and texture functionality. Depending on their origin (e.g. plant, animal), their current applicability for texture functionality varies with the type of food product. In order to meet future requirements on a more efficient and flexible exchange of protein preparations/sources more knowledge is needed on the effects of protein type on texture. This then would allow an extension of the current applicability range of protein sources. Textures of food products are to a large extent determined by the properties of the (protein-based) micro-structural elements and their mutual interactions. These micro-structural properties stem from the aggregation behavior of the protein, which in turn is determined by the molecular characteristics and their ability to interact during processing. To enable exchangeability of protein ingredients, control of the properties of the micro-structural elements is crucial to modulate macroscopic behavior. Amongst others, the micro-structural elements will differ in morphology and sizes (length scales) affecting the rheological behavior of food products. Sizes of micro-structural elements may vary from nm size to mm size, e.g. protein molecules (nm) and protein molecules that are assembled into flexible fine stranded structure elements (0.1 - 5 μm), and coarse stranded or particle shaped structure elements (5 - 50 μm). Linking the characteristics of size-specific micro-structural elements to macroscopic behavior is reasonably well described within literature. However, if within one system micro-structural elements exist of different length scales, information about their individual contribution to texture lacks. From a rheological point of view it is challenging to distinguish between the contribution of these different micro-structural elements within such a mixed system. Within our study an inventory is made on the availability of rheological methods to characterize these mixed systems, containing micro-structural elements of different length scales.

Creep and large-amplitude oscillatory shear behavior of whey protein isolate/kappa-carrageenan gels

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Dynamic oscillatory and creep tests are two common rheological methods used to determine viscoelastic properties. In the food industry, these tests are commonly performed in the linear region of the material, although little correlation exists between small-strain data and sensory properties of foods. However, large-strain and fracture properties have been correlated with sensory properties. Several studies over the past decade have described a large amplitude oscillatory shear (LAOS) testing protocol to extract information on nonlinear viscoelastic behavior. In this study, whey protein isolate (WPI)/ κ -carrageenan gels with different structures were studied using LAOS and creep tests to determine the impact of structure on nonlinear oscillatory and creep behavior, and to examine correlations between nonlinear oscillatory and creep parameters. Three different structural types were evaluated: a homogeneous protein gel, a bicontinuous gel, in which both WPI and κ -carrageenan exhibited continuous network structures, and a carrageenan-continuous gel. Carrageenan-continuous gels showed the greatest degree of nonlinearity under LAOS (25 % strain), while homogeneous gels displayed the least. During both linear and nonlinear creep testing, bicontinuous gels showed the least instantaneous and retarded compliance and greatest retardation time and long-time viscous flow. Homogeneous gels displayed greater elastic and viscoelastic behavior and less long-time viscous behavior than bicontinuous or carrageenan continuous gels. Creep data were fit to 4-element Burgers models for further analysis, and the predicted compliance values were found to be in agreement with experimental data ($R^2 \geq 0.9$). Nonlinear oscillatory data was found to correlate ($R^2 > 0.7, p < 0.05$) with parameters used in the 4-element Burgers model; in particular, the duration a strain rate in the nonlinear viscoelastic region was applied to the sample, and the ratio of retardation time to were found to correlate to LAOS parameters indicative of nonlinear behavior. Hence, nonlinear viscoelastic behavior among materials may be evaluated by using creep data as well as LAOS data. Combining information on structural behavior derived from LAOS and creep data may yield insights on how various structural types deform and break down under application of stress or strain.

On the applicability of the foodtexture puff device for rheological analyses of food products

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The Foodtexture Puff Device (FPD) is a new apparatus for rheological measurements. This device yields a contact less, fast, easy and non-destructive measure of the rheological characteristics of food products [1]. It applies a controlled air pulse to the surface of the sample, whereby a laser distance sensor measures the deformation. This deformation can be related to rheological parameters or product parameters (e.g., storage and loss moduli, sugar concentration). Bamelis & De Baerdemaeker [2] demonstrated that the FPD can be used to determine rheological properties of dairy products. The measurements were related with the storage and loss moduli of milk during curd formation. Van Dyck et al. [3] studied O/W emulsions, where the FPD analyses were compared with the spreadability, determined with the spreadability rig (TA.XT-II+, StableMicroSystems). In the present research, three groups of food products with a broad range of rheological characteristics were studied with the FPD. Viscous systems, such as sugar solutions, glucose syrups and fruit purees showed a good correlation with the viscosity values determined with a shear rate ramp (Physica MCR 301, Anton Paar). The second group were products with a yield stress, such as oils, fats and chocolate. Oils and fats with different proportions of saturated and unsaturated fatty acids were measured, as well as several types of molten chocolate. The maximum deformation of the FPD signal correlated well with the viscosity values determined with a shear rate ramp. Thirdly, visco-elastic products, such as dough and mixtures of gluten and water, were analysed with oscillatory measurements from which the storage and loss modulus were calculated. These were compared with the maximum deformation of the FPD signal for doughs with different amounts of water and different flour types. There was a very good correlation between the storage modulus and this maximum deformation. Finally, the industrial applicability in quality control was evaluated and it was shown that for several applications, the Foodtexture Puff Device is a promising device for real time analyses of rheological characteristics of food products.

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[2] Bamelis, F., De Baerdemaeker, J. (2006). Use of the Foodtexture Puff Device to monitor milk coagulation, *Journal of Dairy Science*, 89, 29–36

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Hyperbolic Contraction Flow for characterization of extensional properties of foods

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Extensional rheological properties are important for diverse applications e.g. baking, mouthfeel of semi-solid foods, processing of viscoelastic fluids, cell mitosis, e.t.c. and are also a useful tool for testing the applicability of constitutive equations. Many foods are non-Newtonian elastic liquids, and any change in geometry during the process generates a flow with an extensional component. Despite the documented influence of extensional rheology, it is seldom measured due to experimental difficulties. There is equipment available for dilute solutions and for polymer melts such as the Meissner type methods for stretching a polymer ribbon. Neither of these methods is suitable for semi-solid materials such as foods, which have a viscosity significantly higher than dilute solutions, but are not solid enough for a melt rheometer. A new test method has been developed suitable for medium viscosity foods using extensional flow in a hyperbolic contraction flow geometry. In the method the transient stress is measured under constant extension rate through a specially designed contraction nozzle. The method has been used for measurements of the extensional rheology of e.g. dough and dairy products. The results from contraction flow are shown to be comparable to filament stretching results for model systems. The contraction flow method has been applied to characterize food and medical systems, distinguish between different food products having equal shear behavior, quantify ropy mouth feel and to predict foaming behavior of biopolymers. The flow behavior through a hyperbolic contraction and similar geometrical configurations has been numerically evaluated to find an optimal geometry for achieving a constant uniaxial extensional flow. The flow of elastic fluids with constant shear viscosity (i.e. Boger fluids) through several different axisymmetric contraction configurations was analyzed through the strain, extensional viscosity and N_1 profiles. The results showed that a constant strain rate was achieved in the hyperbolic configuration in contrast to e.g. a 45 degree contraction, where a peak around the start of the contraction was observed. Due to the gentle contraction of the hyperbolic configuration, lower forces are needed to push the fluid through the die. Higher forces can thus be used which will lead to a broader measuring range, enabling higher strains and strain rates.

Monitoring emulsions morphology under shear via simultaneous rheometry and in-situ FTIR spectroscopy

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Emulsions are of wide interest in food, pharmaceutical, health care and cosmetics industry. They typically consist of at least two liquid phases, surfactant agents and stabilizers. Emulsions exhibit complex rheology, making it difficult to understand flow phenomena on a microscopic level. A common technique utilized to analyze emulsions under shear deformation is light microscopy. However, emulsions showing droplet distributions with droplets on the submicron scale cannot be visualized using light microscopy because of given resolution constraints. Other limiting factors of light microscopy arise when the concentration of droplets with equal size is high or the refractive indices of the two phases are too similar. In such cases, the Thermo Scientific HAAKE MARS rheometer combined with the Rheonaut module, provides simultaneous rheometry and in-situ FTIR spectroscopy under shear deformation and offers a versatile tool for efficient and comprehensive emulsion research and stability testing. Simultaneous rheometry and FTIR spectroscopy proves to be useful tool in understanding the microscopic state of droplets in concentrated emulsions. In this contribution, we want to present some results on the dynamics of molecular organization of an emulsion under shear.

Mathematical model for viscous heating in a mini-Couette cell used in Rheo-XRD and Rheo-NMR research with a newtonian fluid

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A mathematical model is proposed to describe the temperature increase due to viscous heating in a mini-Couette cell used in Rheo-XRD and Rheo-NMR experiments on crystallization of triglycerides. The model was tested using a standard Newtonian rheological oil (Cannon N1000). The temperature of the cooling fluid inlet and outlet as well as the mechanical power delivered to the oil were measured. The measurements were conducted in a temperature range of 1 - 20°C under rotational speeds between 0.1 and 50 rps. The average shear rates corresponding to these rotation speeds were calculated taking into consideration the curvature of the cell and the temperature dependence of both the viscosity and the thermal conductivity of the oil. The model was cast into functions of the non-dimensional temperature and non-dimensional velocity of the oil, and their derivatives. Numerical solutions for the four variables were obtained for the boundary conditions of the mini-Couette cell. A first set of solutions were sought where the heating effect could be considered negligible and the fluid remained isothermal. The temperature and viscosity behavior of the oil was then analyzed under high shear rate, to study the viscous heating effect of the oil. At high shear rates, however, there is a high temperature gradient across the oil and consequently a considerable departure from the radial shear rate distribution compared to an isothermal oil. The effect of viscous heating was obvious at low temperatures, since the viscosity of the oil is high, but was considerably decreased as temperature increased. The model developed in this work is essential for the interpretation of viscometric experiments done under shear flow in small-enclosed cells with high curvature, for understanding the crystallization behavior of mixtures of triglycerides. They also help with the calibration of the rheometer under high shear rate conditions, as well as for the design of industrial shearing crystallizers.

Structural characteristics of chocolates

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Chocolate is a suspension of solid particles (sugar, cocoa solids and milk solids) in a continuous fat phase. Chocolate manufacturing is complex and requires a combination of several ingredients and technological operations to achieve the desired rheological, textural and melting qualities. Cocoa nibs contribute which around 30 % of the final chocolate fat content. Particle size distribution is a key determinant of the flow (rheological) properties in chocolates with a direct influence on sensory perception. Quality in finished chocolates is highly dependent on inherent size distribution of solid particles, composition of fat phase and emulsifiers. The rheological parameters (yield stress and plastic viscosity) of chocolates is usually quantified during processing, mainly through Casson model. The aim of this study was to evaluate structural chocolates characteristics made with different fat content and particles size by three steps test. The rheological behavior of the formulations was determined by a Haake MARS rheometer at 40°C with the test (CR) taken in three steps: (1) 0,00 1/s a 65,00 1/s, $t = 180$ s; (2) 65,00 1/s, $t = 60$ s; (3) 65,00 1/s a 0,00 1/s, $t = 180$ s. Fat content and quality as well particles size were determined. The chocolate samples were prepared using traditional process. The results of rheological studies showed the interference of fat content and particles size on the initial tension of the products. The sample with lower fat content and larger particle size showed higher yield stress and greater hysteresis area, with larger thixotropy, indicating the relationship of these parameters with the fat content and particle size. The viscosity has only a slight increase; this difference was not significant factor in practice, pointing out that both the applied variation of fat (36 - 42 %) as the particles size (18 - 22 μm) were not a great interference in this case.

Use of ultrasonic measurements of asian noodles to reflect their intrinsic gluten strength and texture

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Yellow alkaline noodles, 34 % water, 1 %w/w kansui reagent and 1%w/w NaCl, were prepared from Canada Western Red Spring (CWRS) and Canadian Prairie Red Spring (CPSR) wheat flours of significantly different protein content (14.4 versus 12.3 %) and gluten strength. The noodles' bio-mechanical characteristics were evaluated by longitudinal ultrasonic wave measurement (40 KHz) of varying layered thicknesses (2 - 7) of noodles to yield phase velocity and the attenuation coefficient. The noodles were also evaluated for bite by the traditional empirical maximum cutting stress (MCS) test as well as the fundamental stress relaxation and Peleg's k_1 and k_2 parameters. Harvest (CWRS) displayed a weaker gluten strength, based upon its Farinograph dough parameters; dough development time 7.25 min and stability 16.0 min as compared to the lower protein but stronger 5701PR's 19.75 min and 22.5 min, respectively. The bite test (MCS) surprisingly indicated that the significantly lower protein content CPSR variety 5701PR yielded very similar values to Harvest, 37.8 versus 38.5 g/mm², respectively. No significant differences in the nature of their starch properties, via RVA analysis, were observed to account for this. Stress relaxation at 20 second yielded similar values; 35.4 % for 5701PR and 33.5 % for Harvest indicating that while 5701PR had significantly lower protein content, it was capable of dissipating the compressional stress energy almost identically to that of the higher protein Harvest, due to a similar elastic mechanical behavior. This similar mechanical behavior is supported by Peleg's linear k_1 and k_2 parameters which were 7.06 and 2.47 for variety 5701PR while Harvest's values were 7.83 and 2.60, respectively. Examination of Harvest's ultrasonic wave velocity of 501 m/s was not significantly different from the 487 m/s observed for 5701PR. However, the ultrasonic wave attenuation was significantly different, with 561 1/m determined for Harvest and 466 1/m for 5701PR. The distinction in attenuation highlighted the difference in the noodle dough matrixes between these two varieties/classes. The lower attenuation 5701PR suggests a more developed gluten dough matrix, supported by a significantly greater dough density, which would account for its ability to display elastic mechanical behavior similar to the higher protein noodle flour.

Basic methods for the determination of honey origins: Applicability of rheological procedures on the classification of honeys

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Honey is a natural sweet and viscous product which is produced by honeybees from nectar of different plants. Honey is a highly concentrated solution of simple sugars namely monosaccharides such as glucose and fructose that are responsible for viscous characteristic of honey. The chemical composition of honey that does not contain only sugars but also organic acids, amino acids, macro and micro nutrients and biologically active substances, is variable, owing to the differences in plant species, soil, climate, and environmental conditions. According to the pollen frequency, honey is identified and entitled. Determination of botanical and geographical origin of honey has been necessitated by Commission of European Union and Codex. There are different techniques for the determination of honey origin. Traditionally, melissopalynology has been used to determine botanical origin of honey. Honeys are classified as monofloral, when the pollen frequency of one plant is higher than 45 %. However melissopalynology is extremely onerous, time-consuming and require experienced analysts. On the other hand, some constituents such as amino acids and proteins, flavonoids, carbohydrates, fermentation products, aroma compounds, minerals and trace elements, organic acids phenolic compounds, stable isotopes can be used as marker for botanical origin of honey. High performance liquid chromatography, gas chromatography, gas chromatography-mass spectrometry, emission spectroscopy and electronic nose are the other instrumental techniques using for this purpose. Rheology is the study of flow and deformation of semi-solid and liquids. Rheological properties of materials are directly correlated with physical properties. The physical properties of honey such as viscosity, electrical conductivity, crystallization etc. are related to nectar and pollen source of plant, color, flavor, moisture and contents of protein and sugars. In this respect, rheology may utilize for classification of botanical origin of honeys.

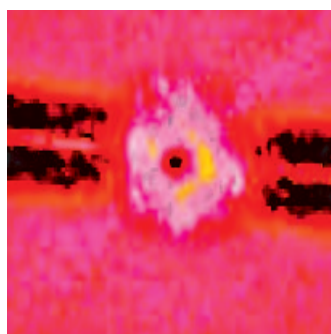
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Rheological Modeling and Numerical Methods

Fermented milk products as microgel suspensions - Analysis and modeling by means of rheology

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The texture properties of fermented milk products are mainly determined by two essential process steps, gel network formation during fermentation and its destruction due to subsequent mechanical load or targeted processing. In simple terms, the texture properties can be attributed to the interaction of the gel network, the microgel particles building up the network and the underlying casein micelles. The objective of this study was to analyze the structure relationship between the components and to identify their contribution to the product structure. The microgel suspension fresh cheese was studied by means of frequency sweeps in the region of LVE response. The effect of stress on particle-particle interactions was modified by different volume fractions. In addition the microgel particles were substituted stepwise with glass microspheres in the same dimension as the microgel particles to study the difference in particle's inner structure. The complex rheological properties of the fresh cheese and model suspensions were determined and fitted by means of coupled Cole-Cole-models differing in their parameters. Each model represents a frequency dependent process contributing to the entire texture properties. Using different sets of parameters provides a tool to identify the contributions of the casein micelles, microgel particles and other components based upon their differing relaxation times in frequency range. Influences of single processing steps, like fermentation, concentrating and shear treatment, could be assigned to changes in single components or to interactions of the components. According to rheological measurements a modeling toolbox was developed which allows to include the effect of each single component in a complex product matrix. Conclusions regarding the influences of simple components on the texture at all could be made using this toolbox. This study provides a deeper understanding of interactions of the microstructure of the product and enabled us to create a defined microstructure by means of altering the composition and processing.

Computational analysis of the spray processing of multiple emulsion structures

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Multiple emulsions are able to encapsulate both hydrophilic and lipophilic functional components (FCs) making them useful in industries such as the food and pharmaceutical industries. Emulsions are thermodynamically unstable and generally require surfactants and emulsifiers to prevent separation and loss of FC encapsulation. Spray processing of multiple emulsions to produce powders increases shelf life and stability of the multiple emulsions. However, multiple emulsion structures are sensitive to mechanical processes, which presents a challenge for process optimization. In this poster a computational approach is used to examine the spray processing of multiple emulsion structures in order to optimize spray nozzle parameters and spray conditions to minimize emulsive structure breakdown while producing spray droplets of a controlled size within a narrow size distribution. The OpenFOAM platform is to be used to simulate flow conditions within the nozzle and breakup conditions after the emulsion exits the nozzle. The simulation takes into account material flow properties from experimental data simplified as a single phase non-Newtonian fluid as well as known flow parameters and nozzle dimensions. The resulting drop size distribution from simulation is compared with experimental data in order to validate the model, which can later be used for design optimization.

A composite model to predict cheese sensory texture

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Many foods are bio-based materials that consist of structural elements spanning a wide range of length scales. The food sensation or sensory texture of these materials depends strongly on the structural arrangement, mechanical behavior, and interactions of the structural elements. In most food materials a full understanding of the relation between microstructure and even the basic mechanical aspects of sensory texture is still lacking. As a consequence, no systematic strategies towards influencing the sensory texture have been explored. In this study the relation between microstructure and mechanical properties (e.g. sensory texture) of semi-hard cheese will be established. At the micrometer scale, cheese is modeled as a composite material, in which fat particles are dispersed in a protein-water matrix. In order to clarify the separate contribution of fat and protein material properties and the composite morphology to the overall mechanical properties a hybrid experimental and computational procedure is adopted. A Representative Volume Element (RVE) is defined in which filler volume fraction, size distribution and spatial distribution is prescribed. For given values of these parameters a 2D filler composite morphology is created automatically and subsequently translated into an element mesh. Elastic properties of the fat particles are taken from literature and the constitutive behavior is described with a neo-Hookean law. A linear viscoelastic model for the water-protein matrix is created by fitting storage and loss modulus data from oscillatory shear experiments on a multimode Maxwell model. With the morphology of the composite and the material properties of its constituents defined, a virtual stress relaxation experiment is simulated with Finite Element Analysis. At different levels of fat concentration, this is compared with a stress relaxation experiments on real cheese samples. By systematically changing morphology of the virtual composite and comparing simulation results with experiments the contribution of structure to overall mechanical properties is elucidated. In future work, non-linearity will be introduced into the constitutive equations and large deformation experiments will be performed. Furthermore an extra length scale will be introduced in the structure-property model by describing the protein-water matrix as a network of sponge-like particles. Constitutive laws at this length scale will be derived using micro gel suspensions and Brownian dynamics simulations.

Velocity and attenuation analysis methods for noodle dough characterization

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We have shown that ultrasonic velocity and attenuation measurements at low frequencies (approx. 40 kHz) have the potential for discriminating technologically relevant properties of wheat flour dough products, including bread making doughs and Asian noodles made from flours milled from different wheat classes. A conventional method for analyzing pulse transmission measurements is to measure the travel time and amplitude of the first minimum and maximum of the transmitted wave pulses as a function of sample thickness. In experiments conducted on yellow alkaline noodle doughs (34 and 37 % absorption basis, with 1 %w/w kansui and 1 %w/w NaCl), small differences in velocity and attenuation values were found in the same samples from the measurements at these two different observation points in the acoustic pulse. To investigate the underlying mechanism for these differences, we propose another method in which the entire transmitted pulse is analyzed using Fourier transform techniques, enabling the transmitted phase and amplitude to be compared for samples of different thickness. This new method reveals a weak frequency dependence of both velocity and attenuation over the bandwidth of the input pulse (35 - 45 kHz). As a result, the pulse shape changes slightly as the different frequency components of the pulse travel at different speeds through the sample, explaining the slightly different results observed for the first minimum and maximum methods. The full frequency analysis technique gives more complete information on the velocity and attenuation in this interesting dispersive soft material, pointing to additional parameters for discriminating wheat classes of different quality.

Modeling of apparent viscosity of ice cream mix stabilized with xanthan gum using adaptive neuro fuzzy inference system

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In this study, the effect of xanthan gum concentration on the steady shear rheological properties of ice cream mixes (0 - 0.8 %) was determined. Oswald de Waale model was used to fit the steady shear data of the mixes. The mixes exhibited a pseudoplastic flow behavior. Adaptive neuro fuzzy inference system modeling technique based on fuzzy logic was used to estimate apparent viscosity of the final mix samples. Xanthan gum concentration and shear rate were selected as input while the apparent viscosity was selected as output. Two different membership function numbers (3 or 6), two different membership function types (triangular or Gaussian) and two different membership functions for output were tried to obtain the best results. In general, eight different predictive fuzzy models were constructed to estimate the apparent viscosity. The highest accuracy ($R^2 = 0.9934$) was obtained for the model constructed using six triangular membership functions and linear membership function types for output. Linear output membership functions were better than constant output membership functions.

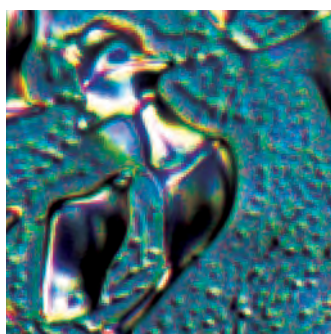
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Biopolymer Solutions and Gels

A non contact measurement for the characterization of the viscoelastic properties of biopolymers: Passive microrheology

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This work presents a new technique of the passive microrheology for the study of the microstructure properties of soft materials. Our technology uses Multi Speckle DWS (MS-DWS) set-up in back-scattering with a video camera. It allows to measure the mean displacement of the microstructure particles in a spatial range between 0.1 and 100 nm and a time scale between 0.1 and 10⁵ seconds. Different parameters can be measured or obtained directly from the Mean Square Displacement (MSD) curve like a fluidity index, an elasticity factor, a viscosity factor, a relaxation time, a MSD slope. Also when the particles move only thanks to the Brownian motion, the Generalised Stokes Einstein Relation given by Mason and Weitz can be applied to calculate the viscoelastic moduli G' and G'' over a large frequency range. This technique allows to monitor the evolution of the microstructure, the restructuration after shearing, the variation of the viscoelastic properties versus temperature, pH, the physical stability of emulsion or suspension. This work focuses on viscoelastic properties evolution and will show application examples and advantages of using a non intrusive method for:

- Gelation process of food products in order to measure the speed of the gelation and the final strength of the network (mesh size)
- Methylcellose viscoelastic properties evolution versus temperature
- Carrageenan networks formation and comparison of their viscoelastic properties versus concentration.

An investigation into the acid gelation of low-acyl gellan gum fluid gels

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One way of tackling the high levels of morbid obesity in today's society is to control the consumers' energy intake from foods. The problem with existing processed foods is that they have become softer, more easily digestible and therefore less satiating. Consumption of such foods leads to the individual feeling hungry more quickly and subsequently wanting to eat again. An approach that may well impact on satiety is the use of hydrocolloids that respond to the environmental (acidic) conditions experienced inside the human stomach by self-structuring. These hydrocolloids can be incorporated in liquid or soft-solid products without an adverse effect on consumer response. It has become evident that the use of hydrocolloids only as simple gelling and thickening agents does not offer the industry and consumer driven attributes of a healthy, convenient, but still high in quality formulated product. This has led to the development of fluid gels, which can be formulated to satisfy a wide range of structural requirements. Fluid gels are formed by applying an appropriate flow field to a biopolymer solution undergoing gelation; as a result a highly concentrated suspension of gelled particles is formed. We have investigated the acid gelation of low-acyl gellan gum "fluid gels" formed at different pH values, for a range of concentrations and processing conditions; by both direct addition and prolonged exposure (post-production) to an acidic environment of hydrochloric acid. The latter assessment of which, simulates the stomach conditions during and post-meal ingestion. The experimental data suggests that, with a low concentration of gellan gum the produced fluid gels' microstructure do not depend on the processing conditions. This was shown by obtaining material properties, rheological studies and calorimetric experiments. Texture analysis of the gel matrices suggests that manipulating the processing conditions during production has minimal impact on the gels' stiffness after exposure to an acid environment. The rheological flow properties of the fluid gel samples (no acid exposure) also displayed a similar trend in relation to the processing conditions, with each sample exhibiting shear-thinning behavior. Calorimetric analysis also revealed comparable melting and gelation temperatures between the samples despite their differing processing conditions.

Rheological studies on agarose hydrogels composed of different carbohydrates

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Carbohydrates are an essential part of the daily human nutrition. Due to their various functional properties, they are widely used as additives in food. The demand for characteristic properties that fulfill the requirements of manufacturers and consumers propel the research regarding the physicochemical properties of carbohydrates. Mixed carbohydrates are of special interest as they offer a versatile range of unique and novel properties. To be able to modify and use them aim-oriented, an intense research is required to understand the complex molecular processes occurring in such systems. In food, characteristic properties are based on the physicochemical functions of the biopolymers added. Thus, the impact of xanthan gum and two types of sugar on the viscoelastic properties, the sol-gel transition and the water holding capacity of 1 % agarose hydrogels was studied by using small deformation tests and moisture analysis. Agarose is due to its simple and neutral conformation a favored model gel for the gelation mechanisms of gelling polysaccharides. Agarose gels are very elastic, prone to syneresis and turbid. Additions of xanthan gum, being very stiff molecular rods of high negative charge, revealed less elastic gels with an unaffected water holding capacity. Progressive addition of two different types of up to 40 % of sugar yield an increase of the elasticity of agarose gels, whereby sugar concentrations of 60 % partially result in a structural breakdown and thus a significant lower network structure but better water holding capacity and clearer gels. However, in ternary systems, the effect of sugar concentration and sugar type used is diminished by xanthan gum presumably due to the different competition for the limited water amount present. The different impacts of different types and concentrations of co-solutes reveal that the gelation mechanism of agarose gels depends on the detailed interplay between chain charge fractions, chain flexibility and hydrate shells.

Determination of the gluten aggregation behavior from different wheat cultivars and correlation with the rheological and the baking properties

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Conventional mixing of wheat flour and water results in dough with properties suitable for the preparation of bread. This unique effect distinguishes wheat from other cereals and depends on the storage proteins, which are able to form a viscoelastic material called gluten upon addition of water. Although many studies about the rheological properties of gluten and their impact on the baking quality have been published, the process resulting in the aggregation of gluten during mixing is still unclear. In this study, a new torque-based technique for the determination of the gluten aggregation behavior is presented and the results obtained with different wheat cultivars are compared with the rheological and the baking properties. For the experiments a gluten peak tester (GPT) (Brabender, Duisburg, Germany) was used. In the instrument a flour/water suspension (8 g/9 mL, containing 16 g/L sodium chloride) was mixed under high shear (2750 rpm) at 36°C. The maximum torque (MT) and the peak maximum time (PMT) were determined. Nine wheat cultivars differing in baking quality were analyzed. The results were correlated with data obtained from micro-farinograph measurements (peak time), micro extension tests on dough and gluten using a Texture Analyzer with a Kieffer Dough and Gluten Extensibility Rig (resistance to extension (RE), extensibility (EX), work of extension (WE)), micro baking tests (loaf volume) and from protein determinations. The PMT was strongly correlated with the farinograph peak time ($r^2 = 0.86$). This could be expected because of similarities in both methods. For the MT the strongest correlation was obtained with the protein content ($r^2 = 0.98$). This parameter was also closely related to the rheological properties of dough, i.e. with WE ($r^2 = 0.95$), followed by RE ($r^2 = 0.86$) and EX ($r^2 = 0.81$). The correlations to the gluten properties (RE: $r^2 = 0.82$, EX: $r^2 = 0.28$, WE: $r^2 = 0.86$) were less significant compared to dough indicating that the aggregation behavior of gluten also depends on other flour constituents than protein. There was also a relationship between MT and the loaf volume ($r^2 = 0.88$). In conclusion, characterization of wheat flour with the GPT is a suitable method to predict the technological properties and to do fundamental studies on the aggregation behavior of gluten, which is also affected by non-gluten constituents of the flour.

Intrinsic viscosity of wild sage (*Salvia macrosiphon*) seed gum as affected by ionic strength

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In this paper, the effect of salt type (sodium and calcium chlorides) and salt concentration (0, 0.5, 20 and 50 mM) on the intrinsic viscosity of sage seed gum (SSG) solutions were investigated. SSG was evaluated for intrinsic viscosity by various models i.e. Huggins, Kraemer, Higo and Tanglertpaibul and Rao equations. The results showed that the plot of relative viscosity versus concentration, obtained from Tanglertpaibul and Rao model described the best phenomenon. The increase in ionic strength of the NaCl and CaCl₂ from 0 to 0.5 mM caused increase in intrinsic viscosity, but increasing the salts concentrations from 0.5 to 50 mM decreased the intrinsic viscosity. Divalent ions from CaCl₂ showed a more pronounced effect on the intrinsic viscosity compared with monovalent ions from NaCl.

Mechanistic insights into the formation of acid treated waxy potato starch gels

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Starch molecular structure modification is effective for improving texture functionality. In particular, acid treatment of waxy potato starches has been used to modify their gel forming properties [Buwalda et al. 2005]. Waxy potato starch (99 % amylopectin) was acid treated to obtain products with different degrees of hydrolysis. Relationships between molecular characteristics and gelling properties were investigated to provide mechanistic insight. Waxy potato starch hydrolyzed for longer time led to lower paste viscosity and reduced molecular weight, as confirmed by gel permeation chromatography. Branch chain-length distribution was analyzed by isoamylase treatment followed by high performance anion exchange chromatography, which showed little variation resulting from hydrolysis. Gelatinization temperature and enthalpy of melting (ΔH) measured using differential scanning calorimetry (DSC) showed no significant change with degree of acid hydrolysis, indicating similar crystalline structure following treatment. After gelatinization, starch solutions formed gels at refrigeration temperature (4°C). Gel formation was found to depend on starch concentration, time and degree of acid hydrolysis (molecular weight). A mathematical model was developed to describe their effects on storage modulus (G'). Compared at the same concentration, increasing degree of hydrolysis resulted in higher rate of G' increase at short time but achieved lower ultimate G' , suggesting that reducing molecular weight enables cross-links to form more quickly but their apparent number in the equilibrium gel network is fewer. Gels formed using starches with a higher degree of hydrolysis showed lower fracture stress and reduced fracture strain, suggesting a weaker gel network. Cross-links were found to be dissociated by heat, leading to gel structure melting corresponding to an endothermic DSC peak on heating. The melting peak shifted to lower temperature with increasing degree of hydrolysis. Results suggest that acid attacked amylopectin but did not significantly change crystal structure in starch granules, leading to a significant decrease in molecular weight but only minor changes in branch chain-length distribution and gelatinization enthalpy. The impact of these changes on the mechanical and thermal properties of waxy potato starch gels provided insight into the structural features controlling gel texture and melting behavior.

Dynamic rheology of oxidized starch filmogenic solutions with glycerol and zein addition during a heating-cooling cycle

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This work was done to determine the effect of glycerol (plasticizer) and zein (reticulant) addition, on the viscoelastic profile of film forming solutions of oxidized banana starch. Samples of starch solutions with 4 %w/w total solids, composed of modified (oxidation with 2.5 y 3.5 %w/w sodium hypochlorite) banana starch (2 %w/w solids), glycerol and zein either alone (2 %w/w solids), or combined (1.4glycerol:0.6 zein %w/w), making a total of eight treatments (oxidized starch 100, oxidized starch:glycerol 50:50, oxidized starch:zein 50:50 and oxidized starch:glycerol:zein, 50:35:15) were prepared. Amplitude (to define the linear viscoelastic region) and frequency sweeps were carried out using a stress controlled Rheometer TA Instruments (strain mode), model AR1000, using a cone and plate system, 60 mm of diameter and angle of 2°. Samples were heated up at 10°C/min starting at room temperature till 90°C, running tests at this temperature and then, filmogenic solutions were cooled down to 25°C at the same above mentioned rate, running isothermal tests also at this last temperature. All starch solutions at both 90 and 25°C, behaved as weak gel-like viscoelastic materials with the storage modulus larger than the viscous modulus over the amplitude and frequency ranges [1]. Starch filmogenic solutions oxidized with 2.5 % hypochlorite with both additives (glycerol, zein) produced gels with the highest moduli values when compared with those having any additive alone at any temperature, while those oxidized with 3.5 hypochlorite, presented overall the same event, but with zein addition. In all cases, both moduli presented higher values after the cooling down process. It was seen that the higher the level of oxidation, the higher the moduli values irrespective of the temperature. Results suggested that zein addition (reticulant) strengthened sample structures [2] of oxidized starch, in a higher extent than glycerol (plasticizer).

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Understanding perception of texture and flavour from thickened solutions in relation to their rheology for improved design rules for liquid and semi-liquid foods

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Liquid and semi-liquid processed foods often contain dissolved biopolymers to impart specific textural product characteristics. Added thickeners impact on flavor perception and the rule of thumb is that increased shear viscosity measured at 50 1/s leads to a decrease in taste intensity [1, 2]. It has also been suggested that extensional flow behavior impacts on taste perception [3]. Retronasal perception of volatile flavor compounds does not seem to be affected by product thickness and mouthfeel attributes may correlate better to viscosity data acquired at higher shear rates [4]. The aim of this research is to generate an advanced model for perception of texture and flavor from biopolymer solutions, including volatile and non-volatile flavor compounds. To develop this model, a series of thickened solutions of specific low and high shear viscosity behavior were designed ranging from nearly Newtonian to highly shear thinning. A set of five samples each was designed to either have a low shear viscosity of 0.35 Pas or a high shear viscosity of 0.01 Pas measured at 50 and 105 1/s, respectively. The rheological behavior of these samples was further characterized in small amplitude dynamic oscillatory shear and stretching flow. All samples contained 3 g/100 g sugar and 100 ppm of banana flavor (isoamyl acetate). A trained sensory panel generated and evaluated mouthfeel, aroma and taste attributes of these solutions. Thickness perception was found to be highly correlated to shear viscosity measured between 10 and 500 1/s as well as to complex viscosity measured at 100 rad/s. However, there was little correlation for viscosity measured at high shear rate (105 1/s). An example for a texture parameter that was better modeled by a combination of low shear and high shear viscosity was stickiness. Including extensional viscosity in the model further improved the correlation coefficient. Analysis of the flavor perception trials revealed that banana aroma perception was weaker for solutions that were also perceived as less sweet and as thicker than others. A shear viscosity model including both low shear and high shear viscosity fitted the results best. The sticky samples exclusively belonged to the set of samples that was viscosity matched at 50 1/s and varied in high shear viscosity. Sweetness and banana aroma were more intense with increasing high shear viscosity. Stickiness showed the same trend. The present results provide novel insights into the perception of texture and flavor from thickened biopolymer solutions and the models are currently being developed further.

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From wood to food: spruce galactoglucomannans in films, emulsions, and microcapsules

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The food industry has a substantial demand for novel, non-allergenic, plant-derived, and sustainable hydrocolloids to be used, e.g., as emulsifiers and/or stabilizers in a number of applications. Requirements for such a product include non-toxicity, constant quality, assured supply, and neutral taste and odor. Spruce (*Picea abies*) galactoglucomannans (GGM) are polysaccharides that can be recovered at high yield and low cost as forestry industry by-products: from thermomechanical pulping process water by purification and ultrafiltration following alcohol precipitation or spray-drying, from wastewater of fiberboard mills by ultrafiltration, or from wood chips by pressurized hot-water extraction. GGM are a new potential natural raw material, but they are not yet isolated or used in an industrial scale. We have recently studied the properties of GGM as water solutions and in different applications, i.e., biodegradable food packaging film formers, beverage emulsion stabilizers, and microcapsules. The viscosity of GGM solutions was shear rate-dependent at high concentrations, while Newtonian flow behavior was observed at low concentrations. In the presence of a suitable plasticizer, GGM formed films with promising oxygen barrier properties. The mechanical performance of GGM films could be improved by crosslinking with glyoxal or reinforcing with microfibrillated cellulose. GGM showed potential as a stabilizer of beverage emulsions, which are highly diluted systems with citrus oil emulsified in the aqueous phase. Due to the properties of GGM, e.g., water-solubility, emulsion stabilizing capacity, and low viscosity in concentrated solutions, GGM could also be an interesting choice for the microencapsulation of food compounds. Indeed, GGM microcapsules protected α -tocopherol from oxidation significantly better than capsules from gum arabic (*Acacia seyal*). GGM is a potential novel food hydrocolloid and a biopolymer with a wide range of possible applications.

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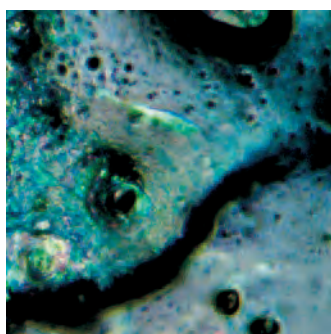
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Colloidal Systems

Stabilization and rheological properties of Iranian fermented milk drink (doogh)

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Doogh is a traditional fermented milk drink produced in Iran. It is manufactured by adding water (50 - 60 % wt) and salt (maximum 1 % wt) into yoghurt (40 - 50 % wt). Optimum consistency and no serum separation are desired characteristics for acidified dairy drinks (pH 3.5 - 3.9) [1]. It was shown that interactions between polysaccharides and proteins may affect the properties of food products such as yoghurt and acidified dairy based beverages [2]. Gum tragacanth is a complex mixture of polysaccharides containing D-galactonic acid, D-galactose, L-fucose, D-xylose and L-arabinose. Tragacanth is one of the most acid-resistant gums; therefore it is chosen for this characteristic to use under acidic conditions and is stable over the pH range of 3 - 10 [2]. It was shown that different species of gum tragacanth have different functional properties [3, 4]. In this study gum tragacanth exudates from three different species of *Astragalus* (*A. gossypinus*, *A. rahensis* and *A. flucosus*) were used to prevent sedimentation during storage and to improve rheological properties of doogh. Different concentrations (0, 0.1, 0.2, and 0.3 % wt) of the mentioned species were added to doogh. Characteristics such as serum separation (in 50mL graduated cylinders), particle size measurements (Mastersizer 2000S, Malvern, UK), and rheological properties (steady and unsteady experiments) (MCR301 Rheometer, Anton paar, Austria) were determined in a constant temperature ($3 \pm 1^\circ\text{C}$). It was shown that use of *A. gossypinus* at 0.3 % wt led to the best stability as a result of protein-polysaccharide interactions and formation of soluble complexes in this concentration which could be explained by increased electrostatic interactions due to the higher uronic acid content of the mentioned species.

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Phase behavior of microemulsion and liquid crystalline formulated with triacylglycerols: A rheological approach

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Microemulsions and liquid crystalline have attracted considerable attention because of their ability to solubilize large amount of lipophilic or hydrophilic additives and to enhance reactions efficiency. These systems are formed by an oil and water phases with surfactant moieties in their interface. Besides, a cosurfactant is generally used in order to obtain higher microemulsion formation. In addition, the ingredients and the system composition are extremely important to the kind of structure formed and its functionality. Food-grade microemulsions are difficult to be formulated because of the complexity of the ingredients and the relative high concentration of surfactant needed. The possibility of using vegetable oils as the oil phase has been scarcely investigated despite of their importance for food industry and to be in accordance with the principles of the green chemistry. Thus, the aim of this work was to construct phase diagrams using commercial sunflower oil or high oleic sunflower oil, water, Tween 80 and ethanol, evaluating the effect of the prevailing oil unsaturation and the cosurfactant. The systems were previously classified using crossed light microscopy, conductivity and droplets size distribution measurements. Nevertheless, the systems were rheologically characterized to evaluate the efficiency of this technique in identifying the phase diagrams limits. Pseudo-ternary phase diagrams showed small areas of one single translucent phase with production of water-in-oil and oil-in-water microemulsions, which showed mean droplets size in a range from 4 to 30 nm. The higher cosurfactant concentration increased microemulsion area and allowed to solubilize more water using less surfactant. Moreover, systems with different characteristics were formed depending on the water content, which influenced the surfactant self-assembly. Rheological measurements were effective to evaluate these differences varying from microemulsions to liquid crystalline. Microemulsions showed Newtonian behaviour and liquid crystalline showed shear-thinning, elastic character and some thixotropy. Systems with lower cosurfactant concentration showed gel-like structure and the less unsaturated prevailing oil led to a slightly stronger gel with small frequency dependence. The variety of systems with distinct textures depended on oil, water, ethanol and surfactant concentrations, which can show a wide applicability in food products.

Spatial variability in fundamental cheese material parameters and their use for Finite Element Modeling

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In recent years, the use of numerical simulations to replace complex and time consuming physical experiments is becoming increasingly popular in the food industry. More specifically, finite element modeling (FEM) is a powerful framework to model complex mechanical and rheological behavior of food and other biomaterials. An accurate description of the behavior of the food product by material characteristics is crucial to obtain a realistic model. This contribution focuses on measuring the most important material parameters describing the complex rheological behavior of cheese. These parameters are needed in FE models to simulate the cutting process of cheese [1], to model heat transfer in cheese [2], to model the salt diffusion in cheese during the brining process [3]. In the past researchers have always modeled cheese as a homogeneous material, but due to the production process and the ripening of cheese there are significant chemical and physical gradients in one cheese block. To obtain a valid FE model, it is necessary to take this heterogeneity into account. The fundamental material parameters were measured at specific places in rectangular blocks of Gouda cheese from 4 till 19 weeks old, ranging from measurements close to the cheese rind to measurements at the center of the block. Young's moduli and fracture stresses were measured with uniaxial compression tests using a Texture Analyser TA.XT2 (Stable Microsystems). Prony series were deduced from the results of stress-relaxation compression tests. 5-term Prony series were fitted to the relaxation functions of the cheese using 0.1, 1, 10, 100 and 10000 s as relaxation times. Young's Moduli turned out to be significantly higher near the crust compared to the center of the cheese blocks for every ripening stage. For a 4 weeks old Gouda E is approximately three times higher at the corners of the block compared to the center. During ripening this difference even increases. The residual Prony series coefficients representing the elastic behavior of cheese at an infinite relaxation time are significantly higher at the corners compared to the center. The same trend is seen for cheese of different ages. For the fracture stress there is no statistical difference throughout the cheese block at any ripeness stage.

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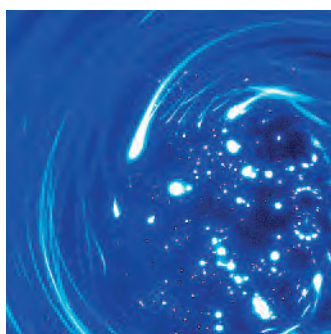
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Dispersions

Influence of surface active components on fat immobilization and flow behavior in ground lipophilic suspensions

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A lipophilic suspension is a disperse system containing a lipophilic, continuous phase and solids being dispersed in it. Food examples are chocolate masses or peanut butter. In milk chocolate, solid phase consists of sugar particles, cocoa particles and milk powder. Due to the high volume fraction of solids typically found in chocolate mass the suspension properties, e.g. flow behavior, are mainly determined by interaction of solids among each other and with the lipid phase. Normally, grinding of the solids being dispersed in the suspension is required for sensory reasons (smoothness). Such a mechanical treatment leads to an activation of particle surfaces affecting suspension properties, too. Food-grade surface active components (SAC) like lecithin are added to control the interactions of solids with the continuous lipophilic phase to obtain a low equilibrium viscosity and yield value. However, the effects of SAC on fat immobilization of the different solids are not known in detail yet. So far, type and amount of SAC added have been selected empirically. Therefore, investigations were carried out to obtain more information on these effects. A special interest was directed to the presence of SAC in the dispersion during the grinding step where new surfaces are generated. For this purpose, suspensions of sucrose and milk powder in cocoa butter were ground by a roller refiner and treated mechanically according to the conching process of chocolate mass. Phospholipids (lecithin), PGPR and citric acid esters were added as SAC before and after refining step. Fat immobilization and flow behavior of the suspensions were determined. Presence of SAC during grinding led to lower fat immobilization at solid surfaces compared to a later addition. This effect was more pronounced for milk powder than for sugar. Degree of fat immobilization correlated significantly with suspension viscosity independent of SAC added. For the yield value of the suspension, correlation to fat immobilization depended on type of SAC. For all SAC except PGPR an optimum degree of fat immobilization could be detected resulting in the lowest yield value of the lipophilic suspension.

Effects of nanofibers containing starch on rheological properties of olive oil

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The structures and rheological properties of electrorheological fluids (ER) are dramatically altered by external electric fields. Olive oil containing starch is a known ER fluid which use for drug delivery. ER fluids may be used for carrying and delivering functional ingredients in foods in the future. The size and shape of particles in ER fluids affect rheological properties. From this point of view, nanofibers may be used as particles in ER fluids more effectively than their counterparts, due to fact that they have favorable in terms of carrying functional ingredients. Electrospinning is an efficient technique for the fabrication of nanofibers. In this study, the effects of electrospun nanofibers containing polyvinylalcohol (PVA) and starch on rheological properties of olive oil were investigated. The electrospinning equipment (Inovenso NE100, Turkey) was used for obtaining nanofibers on a collector plate. The PVA solution was prepared at 6 % (w/w) and used for increasing electrospinnability of the feed. The starch dispersion at 3 % (w/w) was heated at 80°C for 30 min. The PVA solution and gelatinized starch were mixed at 60:40(v/v). The feeding rate of the mixed solutions to the electrospinning equipment was 3 ml/hr and the collector plate distance was 8.5 cm. The applied voltage was 28 kV. The electrospun nanofibers were mixed with olive oil at 7.5 and 10 % (w/w). The rheological measurements were conducted using a rheometer (Haake RheoStress1, Germany) with a 3.5 dia parallel-plate sensor (gap = 0.5 mm) at 24°C. The viscosity measurements of olive oil, dispersion of starch particles-olive oil at 7.5 % (w/w) and mixtures of nanofiber-olive oil were done in the shear rate range of 0.1 - 50 1/s. The viscosity of olive oil was 0.096 ± 0.001 Pas, whereas the viscosity of starch-olive oil dispersion at 7.5 % was 0.117 Pas. The viscosity curves of nanofiber-olive oil samples were modeled using power-law equation. The K values of nanofiber-olive oil mixtures at 7.5 % and 10 % were 1845 and 3801 Pasⁿ, respectively. The n values of both nanofiber-olive oil mixtures were 0.7 ($r = 0.77 - 0.80$). The addition of nanofibers increased the viscosity of olive oil substantially. The effect of nanofiber concentration on viscosity of olive oil will be determined.

Influence of skim milk components on the swelling behavior of starch and rheological properties of starch suspensions

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Starch is often used as a thickening agent in food systems in combination with other ingredients such as milk. The viscosity of starch dispersion in milk is higher than that in water. This effect can not be completely explained by the contribution of milk proteins to the viscosity, and thus interactions between milk components and starch granules are suspected [1]. This study focused on the influence of skim milk components on the swelling behavior of modified (resistant to thermal and mechanical treatments) waxy maize starch during gelatinization and the rheological properties of the resulting starch suspensions. Three suspension media were selected for stepwise building up of milk complexity: NaCl (0.1 Mol), reconstituted skim milk permeate (ions and lactose) and reconstituted skim milk (ions, lactose and milk proteins). Suspensions of starch (3 %w/w) were heated under shear (up to 85°C, 10°C/min). Sampling of the suspensions at different stages of the thermomechanical treatment (between 50 to 85°C) was done. The rheological study was performed by means of flow curve measurements at 60 and 20°C using a MCR 301 (Anton Paar, Austria) fitted with coaxial cylinders. Viscosity at the Newtonian plateau was recorded, and the relative viscosity calculated. The swelling level was assessed by means of particle size analysis using a Malvern Master sizer (2000, Malvern Instruments, UK). For each medium, the evolution curves of relative viscosities at 60 and 20°C in relation to the starch volume fraction of the samplings were built. The curves obtained for the three media, presenting the relative viscosity at 60°C, were overlaid. The size of the starch granules versus the relative viscosity (60°C) were not modified by the presence of milk constituents. Nevertheless, at 20°C, the relative viscosity of starch/skim milk mixtures increased as a function of starch volume fraction quicker than the two other media. It is thus suspected that interactions between starch and milk proteins during gelatinization could impact the characteristics of starch granules, the effects of those modifications would be increased with their retrogradation. Several ageing durations of the suspensions were tested and DSC experiment set (DSC7, Perkin-Elmer, USA).

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Food composite structures with low water or fat

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Production of highly concentrated suspensions and pastes is a common process in industry, and is for example used in dough production, confectionary masses, crèmes and gels, as well as in ceramic pastes and filled polymer systems. When mixing the disperse powder phase with a low amount of liquid it is difficult to achieve a homogenous mixture due to formation of lumps and clogging. In order to reduce the lumps, additional fluid is usually added to the mixture. Commonly more liquid than what is desired in the final product is required to reduce the lumps, and in turn an additional drying step is needed to remove the extra liquid, which is both time and energy consuming. By using a new mixing process where the liquid continuous phase is transformed into solid state by spray chilling, concentrated suspensions and pastes can be produced by mixing these liquid powders (LPOWs) with the disperse solid powder phase. Thanks to that powders are more easily mixed with powders than with liquid, no or very little extra liquid needs to be added, and hence there is no need for an additional drying step. Initially silica will be used as solid model powder, since it is less complex than most food materials and for which surface properties and particle size distribution can relatively easy be adapted. To produce the LPOWS, both fat and water based materials will be used. To start with, tests will be made on palm stearin fat since it is solid in ambient temperature, and secondly, on different sugar/starch-water solutions, for which the glass transition temperature, T_g , can be varied with different recipes. Of importance will be to characterize the solid and liquid powders for particle size distribution, morphology, and powder and interfacial properties, as well as looking at storage stability and mixing quality. The desired result is to produce stable LPOWs that can easily be mixed with a disperse powder phase, in order to produce a homogenous highly concentrated suspension or paste, which in turn can be further processed into new and innovative, high quality products.

Accelerated stability evaluation of dairy and vegan milk products

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Milk has a high inherent dispersion stability, however, this may be lost when processed and/or amended with additives to obtain various variants with different textural properties and flavor or to enrich it with nutritional supplements. The development of related vegan products faces the same problems. Oil/fat rich constituents tend to cream, others tend to sediment. Flocculation purposely induced for structure building may stabilize against separation but may also lead to enhanced separation. Obviously, separation stability of these products is very complex and almost impossible to predict. For screening of the dispersion stability during the formulation process a multisample approach is proposed using analytical centrifugation, which allows for an accelerated and rapid characterization of the separation behavior (creaming, sedimentation, coalescence, phase separation) of dispersions without dilution. By means of a STEP-technology, space and time resolved extinction profiles quantify the alteration of particle concentration due to the demixing phenomena over the entire sample height during centrifugation. The kinetics can be traced simultaneously for up-to 12 different samples in a temperature-controlled way between 4 and 60°C. The paper illustrates this approach for different dairy and vegan milk products.

Investigation of the porous structure of chocolate surfaces using confocal raman microscopy

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Chocolate is a complex multicomponent food system consisting of solid particles (hydrophobic, hydrophilic, crystalline and amorphous) in a lipid continuous matrix. Fat migration in chocolate products, often leading to fat bloom development, is a major issue for the confectionery industry, since it negatively affects both the visual and textural quality of the products [1, 2]. The mechanism of fat migration in chocolate is until today not fully understood, and hence the detailed development of fat bloom remains unclear. However, some recent studies have suggested that topological features at the chocolate surfaces, featuring imperfections in form of pores and protrusions, could be related to fat migration and fat bloom development [3 - 5]. Thus, the objective of this study is to evaluate if the observed pores and protrusions at chocolate surfaces could be part of a network of pore structures beneath and at the chocolate surface. This is done by analyzing the surfaces of white chocolate pralines enclosing a hazel nut filling, using confocal Raman microscopy. With this technique we can obtain further information about the distribution of the different components in the chocolate, the topological features and how these continue further into the chocolate matrix. These results can lead to deeper understanding of the chocolate structure and thus, also extend the understanding of fat migration and thereby fat bloom development.

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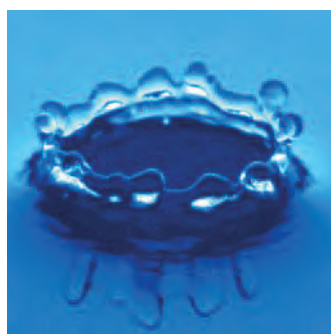
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Influence of Processing on Structure and Rheology

Towards sustainable fish feed production using novel protein sources

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Evaluation of feed ingredient functionality plays a vital role in modern fish feed manufacturing practice. The aim of this study was to examine the extrusion behavior of blends containing alternative protein sources from plant origin to fish meal (FM), such as wheat gluten (WG) and soy protein concentrate (SPC), and the consequences for the physical attributes of the resulting feed extrudates. A mixture design was applied, varying the levels of protein sources included in the formulation from 50 to 450 g/ kg. Each diet was produced with added feed moisture content of 20, 26 and 32 g/100 g (wet basis). The partial least squares regression models were fitted and their performance was evaluated on the basis of R² and the root mean squared error of cross-validation over the complete data set. A higher inclusion level of FM in the diet decreased the values of the extruder system parameters, such as torque, pressure at the die and melt temperature. In contrast, inclusion of SPC significantly increased the values of these extruder- related parameters. The viscoelastic properties of WG gave higher radial expansion; FM showed the opposite effect. The results show that the feed moisture was the dominant factor for extrudate density and oil absorption capacity. Products with higher breaking strength were observed with increasing levels of WG and SPC. Combining the product requirements for both extrudate density and hardness showed that the largest optimal compositional range is available at low feed moisture content. However, maximum FM replacement is possible at high feed moisture content.

Viscosity and small amplitude oscillatory measurements of liquid and solid pekmez (grape molasses) samples

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Liquid pekmez is produced from boiled grape juice without adding sugar or other food additives. It is dark-colored and consumed alone or mixed with tahin (sesame paste) in Turkey. There are different types of solid pekmez, however the Zile pekmez, from Tokat in the northeastern region of Turkey, is one of the most known. It is made of liquid pekmez, egg white and aged solid pekmez. It is white-colored and spreadable due to a homogenous three-dimensional structure, consisting of sugar, protein and air. There is very little known about how the Zile pekmez produced and there is no investigation on how three-dimensional structure occurs from liquid pekmez in the literature. The knowledge of flow behavior of concentrated juices requires in quality control, calculating energy requirements, process control and selection of proper process equipment. The objective of this study was to investigate material behavior of liquid and solid pekmez samples using viscosity and small amplitude oscillatory (SAOS) measurement techniques. The rheological measurements were conducted using a rheometer (Haake, Germany) equipped with a parallel plate sensor (3.5 cm diameter) at 24°C. The viscosity of liquid pekmez, egg white and their blends at 50:50 (v/v) were measured in the shear rate range of 0.1-300 1/s. The addition of egg white decreased the viscosity of pekmez. The linear viscoelastic region was determined for the SAOS measurements. The strain sweep was applied in the range of 0.01-10 at 1 Hz. Then, the frequency sweep measurements were conducted for liquid and solid pekmez samples at 0.1 strain and 0.1-1 Hz. The blends were prepared with the additions of solid pekmez to liquid pekmez at 3, 5 and 30 % (w/w) and measured as well. The G' of solid pekmez dominated G'' whereas it was opposite for the liquid pekmez. The frequency sweep of the blend at 30 % showed liquid-like character. The G' increased almost one decade for the 5 % blend comparing to the 3 % blend. The effects of temperature and concentration on the behavior of solid pekmez will be investigated. The outcomes of this study may help to understand in situ solidification characteristics of pekmez.

Formation of hierarchically structured plant protein materials using simple shear flow

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Unsustainable production process and increasing consumption leads to an urgent need to deliver high quality meat substitutes. A number of such products was already successively introduced to the market. However, consumer studies showed the poor diversity between those foods and the preference for products with meat-like texture. There food structuring science could deliver new options for meat analogues development. Designing new structuring techniques has the potential to form innovative textures but also to improve sustainability of existing methods through reduction of water and energy usage. For example, formation of anisotropic, even fibrous material from mixture of soy protein and gluten in extrusion process is already well-known. But the extrudate is often dried and used as an ingredient in products like vegetarian burger, where it is rehydrated. We studied structure formation of soy protein and gluten under mild, well-defined shear flow at temperature of 95°C. Such conditions were applied using a prototype device designed in WUR. The construction principles were based on cone-plate rheometer. Simple shear flow resulted in an aligned structure from soy and gluten proteins with 70 % moisture where the domains rich in one of the biopolymers were hierarchically organized. Thermal gelation of soy proteins accounted for fixation of the anisotropic structure without the necessity to cool the aligned product. Understanding of structure formation of soy proteins in a shear field was studied by using different commercial soy protein extracts. The composition and rheology of the starting material was linked with the final properties of the formed structure. Our ambition is to substitute soy by another plant proteins in this application in the future. A hierarchically organized anisotropy which resembles meat – like textures and is made of plant protein could be designed into new generation meat analogues.

Structuring of plant proteins by de-structuring plant cells

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The use of plant proteins instead of proteins from animal origin is a promising route to make food production more sustainable. That explains the necessity for food research to be more and more focused on the application of plant proteins in modern food products. The aim of this project is to investigate the isolation of plant (legume) proteins and subsequently study their potential to make plant-based alternatives for animal products. A key factor is the availability of high-quality based ingredients, such as proteins. An important functional property of meat proteins is its water holding capacity. Meat consists generally of 30 % of protein and about 70 % of moisture, which suggests that the water holding capacity of these proteins is at least 2. Apart from this, the texture is usually rather fibrous. Besides, the rheological properties are important, because previous research [1] indicated that those can be used to predict the structuring potential of the ingredients. Wet fractionation methods of legumes like soy and lupine focus on protein yield, purity and protein composition. The protein fraction has to be isolated from a rather complex matrix. A plant cell naturally consists of water, proteins, carbohydrates, fats and some minerals. Carbohydrates are mainly found inside the cell wall, of which cellulose, hemicellulose and pectin are the main constituents. Legume proteins are often present as seed storage proteins, of which about 70 % is present in the form of so-called protein bodies. The protein bodies surround the oil bodies inside the plant cell. Industrial isolation processes separate the individual components on the basis of differences in solubility. Soybeans are dehulled and subsequently milled to powder. Fat is removed by defatting the soybean meal with a solvent and the insoluble fibres are separated from the sugars and soluble proteins by an alkaline wash. Proteins are then isolated by acid or salt precipitation. This isolation process greatly influences the functional properties of soy protein isolates. Physicochemical, physical, chemical and enzymic treatments will cause aggregation, denaturation or dissociation of the protein. Altering the natural properties of the proteins might be detrimental for the creation of new, fibrous or anisotropic structures. A different path to obtain 'more natural' protein isolates is preferable, in which (enzymic) hydrolysis of the carbohydrate fraction seems promising. The protein and oil bodies will not be affected. Additionally, instead of drying the isolate and rehydrating it upon structuring, the wet fraction will be extensively characterized, including the rheological properties. In case the protein fraction is not susceptible to shear flow, it is possible to further modify the properties through a kind of pre-processing step in which proteins are aggregated to a certain extent.

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Controlled fracture behavior of field peas via the state diagram

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Field peas (*Pisum sativum*) and other pulses exhibit highly organized cotyledon tissue structures consisting of starch granules embedded in a protein matrix [1]. Fine milling of peas results in a powder mixture of which small particles ($< 20\mu m$) have a higher protein content. Air classification is applied to produce protein-enriched concentrates up to 56 w/w % protein from the fine fraction. Although this dry fractionation route provides a functional protein concentrate, its purity is relatively low. During milling, cotyledon material is stressed by the action of mechanical moving parts (e.g. pins). When the local strain energy exceeds a critical level, fracture occurs along lines of weakness, and stored energy is released. Ideally, fracture of the material leads to complete disentanglement of starch granules and protein without damaging the starch granules. The mechanical properties of the cell constituents determine to a large extent fracture behavior and vary with moisture content and temperature [2]. This can be understood from the different structural states (i.e. glass or rubber) of starch and protein domains under varying conditions as represented in a state diagram. This work presents a state diagram for the cotyledon tissue of field peas based on Differential Scanning Calorimetry (DSC) and Thermal Mechanical Compression Test (TMCT) analyses of pea starch and pea protein. The state diagram of pea starch is compared to the state diagrams of other starches and their respective prediction based on Flory-Huggins free volume theory [3]. Controlled breakage experiments of single peas, in which the structural state of protein and starch is varied, will be done using a texture analyser. These experiments are meant to identify resulting fracture lines through the cotyledon tissue and could provide an indication to what extent protein matrix and starch granules are disentangled. The latter will be confirmed by Scanning Electron Microscopy (SEM) pictures. Finally, the ambition is to translate the single pea breakage results to pilot-scale milling. The novelty of this work is that mechanical properties of the individual cell constituents are studied and controlled to provide optimal breakage behavior resulting in complete disentanglement of starch and protein.

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Micro-structured powder of emulsion through prilling process

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Emulsions are widely used in food, flavor, cosmetic, pharmaceutical, and coating industries. Double emulsion (DE) offers better encapsulation capacity compared to simple emulsion (SE), even though it is thermodynamically less stable in long-term storage. Production of powder from double emulsion through prilling process would be an alternative to produce well-defined microstructured powder that can be used for encapsulation of various components (both hydrophilic and lipophilic). Although, powder formation from such double emulsion systems by prilling (spray chilling) is a technological challenge due to the mechanical sensitivity structure of the emulsions. Previous studies [1, 2] show the impact of mechanical treatment on the microstructure of the simple and double emulsions during spraying process using twin-fluid atomizer. The goal of the present investigation is to study (i) the influences of rheology of emulsions on the size and the size distribution of powder particles produced by prilling (prills), (ii) the impact of spray process parameters on the microstructure of prills from SE and DE. The microstructure of prills was investigated by cryo-SEM and compared with pre-emulsion. Higher viscous emulsion gives larger particles with less destruction of microstructures. The particle size was also adjusted by changing the gas to liquid mass ratio (GLR) in the twin-fluid atomizer. We have defined the critical gas Weber number, $We(g)$ for keeping the microstructure of prills unchanged. Above the critical $We(g)$ the microstructure of the prills starts to breakdown compared to the pre-emulsion.

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Structure formation in cheese analogues and evaluation its substitution potential

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The objective of the present paper is to elucidate the effect of changes in the concentration of rennet casein (15 - 21 %), melting salts (1.2 - 3 %), fat content (45 - 60 %), some kinds of starch (wheat, corn, potatoes) and process parameters (such as cooking time, rotational speed) on the structuring process of imitation cheese. The structure formation was detected by using oscillation measurements by Rheometer MCR301. For temperature sweeps: $\gamma = 10^{-3}$, $f = 1$ Hz, cool-running 85 - 10°C with a cooling rate of 1 K/min. The amplitude sweep was performed at the measurement temperatures of 80, 60 and 40°C. The linear viscoelastic range was at deformation < 0.01 . The frequency sweep was performed at deformation of 0.001 % and frequency of 0.01 - 50 Hz and at the measurement temperatures of 80, 60 and 40°C. The cheese analogue (about 1 Liter) was manufactured at laboratory scale by a Thermomix TM 31. A processing sequence for the manufacturing of cheese analogue was performed. Throughout the processing and for all the measurements, the storage modulus (G') was larger than the loss modulus (G''), which indicates the solid state properties with dominant elastic properties. The produced cheese analogue has a dispersion character based on a food suspension type (water immobilization) and an emulsion type (fat immobilization), which was confirmed by frequency sweep measurements at different temperatures. From the investigations, it can be concluded that the rennet casein was the most important component for the structure formation in cheese analogues. The best concentration of rennet casein was 17 or 19 %. With 17 % rennet casein 2.1 % melting salt was needed. An increase of the fat content (coconut fat) led to firmer cheese. The addition of extra starch up to a maximum of 2.0 and 2.5 % improved the structure of the mozzarella-like cheese which was produced. With the use of maize starch, no significant differences to wheat starch were found. Replacing the wheat starch with potato starch, leads to products which are less firm. By partially replacing the rennet casein with wheat starch up to 40 % an imitation cheese with acceptable consistency was produced. Cooking for various lengths of time had no significant effect. An increase of the rotational speed led to a firmer Structure. A good cheese-dough resulted at 200 - 250 rpm in the Thermomix TM 31.

Effect of ripening time on microstructure and textural properties of Lighvan cheese – a traditional raw sheep cheese

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Lighvan cheese is made from raw ewe milk in the mountainous areas of Lighvan in Iran. It is considered to be a semi-hard, starter-free and traditional cheese and ripening has a significant effect on its special texture. After Lighvan cheese production, their cubes were stored in 10 - 12 % brine, were ripened over 90 days at $10 \pm 2^{\circ}\text{C}$, and physiochemical, microstructural and textural changes were investigated. Cheese microstructure was studied by Scanning Electron Microscopy (SEM) and 2-D images converted to 3-D images using image analysis software. A texture analyzer was used to determine hardness and brittleness of cheese. The moisture content, salt-in-moisture ratio, pH, acidity and water soluble nitrogen to total nitrogen ratio changed significantly during ripening. Since Lighvan cheese was made from raw milk, the most important biochemical change during its aging was extent of proteolysis which had a significant effect on cheese hardness. During the first month of ripening WSN/TN increased significantly and hardness of cheese decreased during this time due to proteolysis. After one month, due to inhibitory effect of salt on proteolysis, hardness didn't change until the end of ripening. The microstructural study showed that the size and number of pores increased significantly and the dense microstructure with large protein aggregates converted to a casein network with more homogenous aggregates. Diffusion of NaCl into the protein matrix during cheese ripening in brine produces chloride anions in the casein network. Chloride anions play a kosmotropic ion role in the cheese matrix and promote hydrophobic interactions that increase casein linkages and produce a more homogenous texture. Changes in cheese porosity affect the salt transport profile because the brine uptake of the porous cheese matrix will be notably different compared to the non-porous matrix, where brine uptake occurs by diffusion mechanisms. Brittleness of Lighvan cheese during ripening was affected by porosity of cheese which was a result of microbial fermentation.

Effect of addition of milk protein concentrate, casein and k-carrageenan on microstructural, compositional and textural properties during ripening of Lighvan cheese produced from cow's milk

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Lighvan cheese one of the most popular Iranian traditional cheeses, is a semi-hard, starter-free cheese from the Azerbaijan region, in the northwest of Iran, and manufactured from raw ewe's milk. Unique texture of traditional Lighvan cheese produced from raw sheep's milk was adversely affected by substitution of sheep's milk with cow's milk. In this study, effect of addition of milk protein concentrate (MPC), bovine casein and k-carrageenan on microstructural, compositional and textural properties of Lighvan cheese produced from cow's milk were investigated over a 90-day ripening period. Microstructure and texture of cheese samples were studied by scanning electron microscopy (SEM) and uniaxial force-compression method, respectively. The 2D SEM images were converted to 3D images using image analysis software. The moisture content, salt content, ratio of nitrogen to dry matter and the ratio of water-soluble nitrogen to total nitrogen were affected by addition of proteins and polysaccharide. In casein containing cheese, diffusion of salt to cheese matrix was lesser than other samples which could be related to increase in compaction of texture in the presence of it. Addition of MPC and casein increase hardness and brittleness of Lighvan cheese produced from cow's milk. Increase in hardness of cheese in the presence of proteins could be related to increase in dry matter. Increase in cheese brittleness indicated that cheese became less brittle in the presence of proteins which could be related to increase cheese flexibility by their addition. Due to water holding capacity of κ -carrageenan, its hardness and brittleness was different from other samples. Compaction of texture and porosity of Lighvan cheese were affected by polysaccharide and proteins addition.

The effect of hydration on calcium mediated structural formation in a model cheese system

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The hydration of protein is essential in determining cheese structure as it affects the ability of protein to emulsify the fat in the system. This is especially of importance in the manufacture of imitation cheese products that utilize protein powders. It is therefore essential to understand how hydration of protein affects the structure of cheese. The objective of this work was to determine the effect that calcium had on the disruption of structural formation in a model cheese. The impact of hydration time on structural formation in a model cheese system was also investigated. In order to investigate the effect that calcium has on disrupting protein hydration a model cheese system was manufactured. The time period allowed for the protein to hydrate prior to the addition of calcium was varied and the structure of the resulting cheeses was analyzed utilizing a range of techniques each looking at a different level of the structure. The various techniques used to assess the structure and functional properties include dielectric spectroscopy, small amplitude oscillation rheology and nuclear magnetic resonance. The variation in the hydration time prior to calcium addition was found to have a significant impact on the structure and functional properties of the model cheese. These differences in the bulk structure were observed by confocal microscopy and were related to the changes observed in the functional properties of the cheeses. These structural changes were linked to a number of key differences in the binding of compounds measured by the various techniques. The addition of calcium to the model system was found to impact with the hydration of the protein powder. However, by increasing the time for the protein to hydrate prior to the addition of the calcium; significant changes in the structure of the resulting cheeses were observed.

Impact of low temperature extrusion processing on rheology of crystallized high melting water in oil emulsions

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Crystallized water in oil emulsions are most relevant as confectionery coatings, fillings and margarine products with reduced calorie density. One important property, especially developed for puffed pastry products, is plasticity. Aim of this work is to control plastic properties of such w/o emulsions with respect to microstructure and its modification by crystallization. From the status quo of a brittle high melting margarine with a solid like behavior, we developed a processing concept to achieve perfect plastic properties for such w/o emulsions. The structure is build of the partially crystallized continuous phase (fat blend) and the dispersed phase (water). The most important requirement for plastic behavior is a weekly interconnected crystal network, which can be achieved by crystallization under shear treatment balancing between breaking crystal bonds and re-melting crystals. Knowledge about the interrelationships between structure, properties and processing parameters of plastically deformable o/w emulsion-based confectionery and margarine systems are transferred for Low-Temperature-Extrusion processing of low energy density plastic confectionery-, truffle- or high melting margarine masses. In a conventional processing of high melting, high viscous, fat based masses there is high energy dissipation due to high shear and high viscosity. This limits the reachable solid fat content resulting in less crystallization under shear and therefore less fine distribution of fat crystals in the product. With Low Temperature Extrusion it is possible to produce such high viscous, fat based masses with a significant enhanced plastic behavior due to less energy dissipation during further crystallizing under shear resulting in fine distributed, less interconnected fat crystals.

Effect of blanching parameters on structure of microwave-vacuum dried chanterelle

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Chanterelles (*Cantharellus cibarius*) belong to Basidiomycetes, which typically grow in coniferous forests. Basidium, which bears basidiospores, is developed on fruiting bodies (gills) located on the bottom part of the mushroom cap. A layer of basidium that covers the surface of gills is called hymenium. Literature review allowed conclude that pre-treatment of mushrooms may improve quality of dried product. Among other methods blanching is widely used as a pre-treatment method to inactivate enzymes and soften mushroom tissue. The aim of study was to evaluate the effect of blanching parameters on structure and color of microwave-vacuum dried chanterelles. Chanterelles were picked in the second part of August 2011 in Latvia. Blanching time of 3 minutes at temperature 70, 80, 90 and 100°C was selected for the study. A control sample was dried without pre-treatment. A microwave-vacuum drier Musson-1 was used for drying of experimental samples. The following drying program was applied: number of magnetrons 4-3-2 (the program was developed gradually decreasing number of magnetrons), pressure 7.47 - 9.33 kPa, sample chamber rotation speed 6 1/min, product load per cycle 0.7 - 1.0 kg (depending on type of pre-treatment), drying time 12 - 18 min. For analysis of mushroom microstructure the samples were cut both from caps and stems in thickness of 5 μm . The fields observed under the microscope Axioskop 40 were fixed using a digital camera at 16 X 20 (VAREL contrast) or 16 X 40. At least ten measurements of the thickness of hyphae were performed using Axiovision Le Rel 4.5. Color was detected using Color Tec-PCM device. Color of sample was measured at least in fifteen various points. Weight loss was observed in blanching process due to diffusion of water and some soluble compounds from mushrooms to blanching water. The significant difference was observed in color parameter L* of samples dried without blanching or after pre-treatment at 100°C and other studied samples. The preliminary results show the tendency of smaller changes in microstructure, weight loss and color was observed for samples blanched at 70 - 80°C temperature comparing to unblanched or blanched at higher temperatures.

Separation of isoflavones from okara

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The increasing production of soy milk is associated with the production of its by-product okara. It is produced in large amounts, which leads to increasing disposal problems. Moreover, its economic potential and use as food or feed is not fully exploited. Being mainly the insoluble part after water extraction of the soybeans, okara contains predominantly insoluble fibers. Besides these fibers, proteins and lipids, okara contains a considerable amount of isoflavones. Isoflavones are minor components belonging to a group of polyphenols having potentially a high economic value. An increasing amount of research about isoflavones suggests that those components largely contribute to the health benefits of soy. Therefore, isolation and purification of isoflavones is of high interest and can increase the economic potential of this by-product. In this research we aim to understand the role of the matrix structure and the mechanical properties on the extraction and separation process of the isoflavones. Okara has a dense and concentrated texture, despite the high moisture content of about 80 %. Its residue after extraction remains an interesting product for food structuring purposes or further utilization. The aim of keeping all components intact and reducing waste results in large challenges for the development of the separation process. Different solvent compositions for extraction of isoflavones from okara were tested, including water as the most environmental friendly solvent. In this contribution sustainable approaches for this separation process and their drawbacks will be discussed. It is concluded that the matrix-solvent interactions have the largest influence on process development, due to their influence on the swelling behavior of the matrix and resulting rheological properties of suspension.

Dynamic rheological characteristics of low-calorie pistachio butter: Effect of fat replacer and sweetener

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The dynamic rheology of different low-calorie pistachio butter formulas was studied in temperature range of 5 - 65°C. Samples were prepared using the Iranian endemic hydrocolloids (Reihan seed gum and Balangu seed gum) and xanthan gum as fat replacers and sucrose and isomalt as sweeteners. All samples showed a strong viscoelastic behavior with consistently higher values of storage modulus (G') as compared to loss modulus (G'') in the entire frequency (?) range studied (0.1 - 100 Hz). A modified Cox-Merz rule could superimpose the steady and complex viscosity data. The elastic structure of all samples changed to viscous behavior with increasing temperature (5 - 65°C), regardless to the type of fat replacers and sweeteners' level. The temperature sweep showed that the heating and cooling cycle resulted in higher elastic properties and more solid-like behavior.

Intensified protein structuring for more sustainable food: development of a continuous process

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Meat production is extremely inefficient with respect to use of land, water and raw materials. As consumers, we obtain only 10 % of the necessary proteins that are initially present in the cereals fed to animals. Furthermore, to produce one kilogram of meat, two orders of magnitude more water is needed, compared to cereals. A proven solution to that problem is the introduction of meat replacers to our daily diet. Meat replacers are products that contain high amounts of plant proteins like soya. Today's processes are not providing high quality meat replacers and that is due to no dedicated equipment for their production. The experimental set-up to be investigated has been developed by the Food Structuring group at Wageningen University. This set-up is called the Variable Shear Cell. The shear-based structuring technique was only recently discovered. The operating principle of such a device is the application of well-defined shear flow. This version will be the starting point towards the development of a continuous process. The device is called experimental Couette-type device and is based on a two coaxial cylinder configuration with the inner cylinder rotating. Rotation induces a simple shear flow in the protein dispersion. Major focus is drawn on the evaluation of the product quality as a function of the product thickness. Successful product in this project is an anisotropic structured fibrous product. The overall goal of this project is to develop and deliver a continuous process for the production of finely fibrous/structured, plant-based protein food products, meant as sustainable replacers for meat. Achieving this goal is not trivial, as the quality of the shear flow field is essential to the generation of the right textural properties. Imposing flow for transportation of the material through the equipment may well inhibit the formation of a good structure.

Steady-shear rheological properties of low-calorie pistachio butter: Time and Temperature dependency

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Stressing the importance of diet in the prevention of certain diseases, nutritional scientists have emphasized on the reduction of calorie in consumers' diet. Low-calorie pistachio butter is a novel food product which is an alternative to the worldwide consumers' demands for consuming healthy foods. The behavior of samples as a function of time and temperature was evaluated for different formulas. To quantify the thixotropic recovery rate of samples, a 3 - step thixotropy test was conducted. The temperature dependency was studied for temperature range of 5 - 65°C. The Arrhenius model fitted all samples reasonably and the formula containing xanthan gum showed the highest temperature dependency according to the activation energy. The structural recovery evaluation revealed a weak thixotropic behavior for all samples.

Characterization of growth and shrinkage mechanisms of expanding extrusion-cooked corn grits

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Today, many ready-to-eat cereals and snacks are manufactured by extrusion cooking. The brittle and porous product structure is highly important for the sensorial properties such as taste, crispness and 'mouth feel' of the shaped products. Extrusion cooking is a high temperature ($> 100^{\circ}\text{C}$) and shear process leading to flash evaporation of the water available in the starch based dough due to the pressure drop at the die exit. This evaporation leads to bubble nucleation and growth inside the melt and is responsible for significant expansion of the product. After maximum expansion the bubbles can collapse and shrink until the surrounding matrix reaches glass transition temperature and solidifies. Especially, viscous and elastic properties of the plasticized starch matrix affect the bubble nucleation, growth and collapse. The rheological properties of starch melts during the high speed extrusion processing (i.e. screw speeds up to 1800 rpm) are significantly different to those found at conventional extrusion processing conditions (i.e. screw speeds up to 400 rpm). This, in turn, leads to different expansion behavior. In order to model the expansion behavior at high screw speed conditions, mechanisms of nucleation, bubble growth and shrinkage have to be well understood. In this work, expansion behavior of extruded corn grits at varying high speed extrusion conditions was experimentally determined by digital image processing. Influence of the process parameters such as screw speed, moisture content and product temperature on the resulting expansion rate and shrinkage was investigated. For characterization of this behavior, the rheological properties of the melt, as well as cooling and dehydration rates of the expanding product at the die exit were measured. These results were further used to investigate the specific mechanisms of growth and shrinkage during an extrusion cooking process.

Changes of nutritional value and structural properties of new type jellies

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In previous experiments [1] it was determined that the syrup of Jerusalem artichoke (*Helianthus tuberosus* L.) can be used to replace 40 % of sugar in jellies. To improve nutritional value of jellies rolled, groat or flour of hulless barley grains was added. The purpose of research was to evaluate the changes of structural properties and nutritional value of jellies with added hulless barley products. The sample with 40 % Jerusalem artichoke syrup was used as a control. 15 % of barley products were added to jellies. The rolled barley and groats were steeped in water prior adding to jellies. Mixture was hot filled in polystyrene containers. Measurements were performed at room temperature samples on the next day after jelly samples were prepared. The dry matter of jellies was evaluated by drying 5 g of sample in dryer at 130°C for 40 minutes. The pH values were determined by pH-meter Jenway 3510. The jelly strength (hardness) was measured by cutting force using the Texture Analyser TA.XT Plus. The jelly samples were compressed by cylindrical probe (P/25). The cross-head movement as set at a constant speed of 2 mm 1/s, a trigger point of 0.049 N, distance 10 mm. The samples of experimental gels were cut in a 5 µm layer by device Microm HM315 for microstructure analysis under the triocular microscope Axioskop 40 (epifluorescence, excitation 450 - 490 nm, emission > 500 nm). Pictures were taken by digital compact camera via 16 × 10 or 16 × 40 magnification. Obtained results showed that the dry matter in experimental jellies were higher, pH value increased (from 3.56 till 4.11), jellies gel strength decreased (from 35.46 ± 0.18 till 28.92 ± 0.65 N) compared with the control sample. Microstructure analyses show differences between control and experimental samples. Added barley increased content of vitamin B2 in all samples. Different products of hulless barley grains added to jellies improve the nutritional value, but the jellies structure changes and it becomes more porous and crisper.

[1] Kronberga M., Karklina D., Murniece I., Kruma Z.: Changes of agar-agar gel properties after replacing sucrose by inulin syrup. Foodbalt-2011 - 6th Baltic conference on food science and technology, Jelgava, May 5-6, 2011. Conference proceedings published by Latvia University of Agriculture/Faculty of Food Technology, Jelgava (2011) 137-142

Extrusion of snacks containing oat bran concentrate

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Oats are rich in dietary fibre particularly in β -glucan, which is water soluble fibre beneficial to human health. Despite the health effects, oats are not commonly used in extruded snacks because of the high lipid content, which decreases the expansion of extrudates making them dense and hard. The aim of this study was to prepare healthy snacks by extrusion using defatted oat bran concentrate (10 % of solids) with defatted oat endosperm flour (BO-trial) and with conventional corn flour (BC-trial). Moreover, defatted oat bran concentrate (20 % of solids) was used with defatted oat endosperm flour and waxy corn starch (BOS-trial). Extrudates were prepared using a co-rotating twin screw extruder. Water content, expansion and hardness of the extrudates were determined. Extrudates with the highest expansion and the smallest hardness were obtained in all trials when water content of the mass was 16 %, screw speed 500 rpm and temperature of the last section and die 130°C. The highest expansion (161 %) and the smallest hardness (35 N/mm) was obtained in BC-trial while expansion and hardness of extrudates in BO-trial was 72 % and 145 N/mm, respectively. Addition of waxy corn starch (BOS-trial) increased expansion and decreased hardness. Calculated β -glucan content of the extrudates was the lowest (2.9 % of solids) in BC-trial and the highest (6.3 % of solids) in BOS-trial. This study showed that it was possible to prepare extruded snacks containing defatted oat bran concentrate. However, expansion of extrudates containing defatted oat bran concentrate and defatted oat endosperm flour had smaller expansion and higher hardness than extrudates containing defatted oat bran concentrate and corn flour. Use of waxy corn starch improved the quality of extrudates, thus, higher amount of defatted oat bran concentrate could be added in order to obtain well expanded snacks.

The effect of bran and modified bran on physical state of frozen dough

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The main problems in frozen doughs are the death of yeast cells and the weakening of gluten network during freezing and frozen storage. The addition of fibers to dough disturbs the gluten network and makes it weaker. That is why it is challenging to produce fiber-rich frozen dough with high-quality end products. The aim of the present study was to investigate the effects of bran and modified bran to the physical state of frozen dough. Three different brans (steamed wheat bran, unsteamed wheat bran and steamed oat bran) were added to the wheat dough. Steamed brans were modified using enzymes, malts and a food-grade microbe aiming at changing the fiber structure of brans to be more suitable to the frozen doughs. Chemical analysis of the bioprocessed brans was carried out. Melting enthalpies of ice in doughs stored at 35 and 20°C were measured by differential scanning calorimetry (DSC). Relaxations of doughs with maximum amount of ice were determined by dynamic mechanical analysis (DMA) and dielectric analysis (DEA). Also the rheological properties of the doughs stored at 20°C were studied using rheometer. The melting enthalpies of ice of doughs stored at 20°C were lower than those of doughs stored at 35°C showing that the amount of unfrozen water was higher at 20°C. However, the comparison of the doughs was difficult because water contents varied in the doughs the consistency of which was adjusted to be constant. The results of the relaxation determinations carried out by DMA and DEA suggested only small differences in the temperatures of the relaxations. Also, the differences in G' and G'' values obtained using rheometer were small in most cases. Addition of bran and modified bran affected the physical state of frozen dough but the effects were difficult to separate from the effect of various water contents of doughs with constant consistency. Thus, clear trends of the effect of bran addition and bran modification could not be observed probably due to the changes in content of water and water solubles of frozen doughs.

Effect of heat treatment and enzymatic cross-linking of whey proteins on the mechanical and structural properties of whey proteins films

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Whey proteins as a by-product of cheese industry have many novel appliance possibilities in humans' food, because whey proteins are easy to process and have a wide functionality. The aim of the present study was to investigate the effect of physical and enzymatic treatments of whey proteins (WP) solutions on the rheological properties of gels and morphology of the edible films made therefrom. Films were made from WP (7 %) solutions, pre-treated at different temperatures (40, 70, 90°C) followed by incubation with or without transglutaminase (1, 2 and 4 U/g WP) at $40 \pm 1^\circ\text{C}$. Covalent cross-linking of whey proteins in the presence of transglutaminase produced a polymerized network of whey proteins and resulted in the formation of whey protein-based films with specific mechanical and structural properties. It was found that 7 % whey proteins dispersion's viscosity increased from 0.009 to 0.022 Pas during its heat induced gelation at 90°C for 15 min at pH 6.8. Additional heat pre-treatment at 40°C for 30, 60 or 300 min led to sensible modifications in proteins conformation and more positively affected WP gels viscosity, however WP pre-denaturation at 70°C for 1 s does not make significant impact to WP gels rheological properties as pre-denaturation at 90°C for 1 s. Moreover, heat pre-treatment increased the susceptibility of the whey proteins to the reaction with microbial transglutaminase. This microbial enzyme catalyzes cross-linking reactions between specific whey proteins amines and creates thicker molecular network structure, which is visible in the whey proteins films mechanical properties and morphology. Effect of transglutaminase was observed using texture analysis of WP films which showed the variable impact in strength and elasticity of films, made from WP solutions pre-treated with TG. Scanning electron microscopy images demonstrated the tighter films matrix with narrower gaps in TG treated whey proteins films comparing with heat treated specimens.

Influence of ultrasonication parameters and NaCl on the stability of olive oil model emulsions containing xanthan.

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The use of ultrasonics in the food industry is gaining more interest. It represents a less energy consuming technology, hence is considered a trend of green chemistry. This research involves the ultrasonic emulsification of primary-coarse 20 wt% o/w emulsions (pH ~ 4) containing a food-grade emulsifier (whey powder isolate, WPI, 2.7 wt%) and xanthan gum (XG, 0.25 wt%). Two major parameters of the ultrasonic treatment (20 kHz), time and amplitude, were investigated to evaluate their impact on emulsion droplet size characteristics (by diffusion NMR and microscopy), viscosity and stability (by multiple light scattering profiles) during cold storage (10 days at 5°C). Ultrasonic duration was ranged from 1 to 4 min at constant amplitude of 70 %, whereas amplitude was ranged from 40 to 100 % with a changing step of 20 % for a constant time of 1 min. The same conditions of time and amplitude were used to treat solutions of XG 1wt% and evaluate their influence on viscosity of simple solutions. Increase in sonication to significant decrease in oil droplet size resulting in finer emulsions. Although the viscosity of the emulsions and XG solutions was decreased with sonication duration, a decrease in serum layer was observed, thus more stable emulsions were produced. A similar trend was observed when the amplitude was increased. In general, the effect of sonication duration was more effective towards emulsion stability in comparison to the sonication time applied. Finally, a combination of time and amplitude (70 %/3 min and 90 %/1 min) was used in order to further reduce the droplet size in the submicron range in the presence of salt changing the ionic strength from 100 to 400 mM. Salt presence up to a limit of 400 mM was beneficial towards stability (SI = 28.9 %) when comparing to the reference samples (0 mM NaCl) (SI = 29.1%). The moderate use of sonication time and amplitude as well as low NaCl concentration increased the stability of emulsions containing relatively low amounts of stabilizer.

Effect of ultrasound processing on flow rheological properties of carboxymethyl cellulose

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Hydrocolloids are large polysaccharide or protein macromolecules used in different systems (foods, pharmaceuticals, etc.) to enhance their functional properties. Hydrocolloids can make large changes even at low concentrations. Hydrocolloids are essential ingredients of many food and non-food formulations which may undergo different processes along with other ingredients. In recent years, ultrasound processing is gaining more and more attention from the researchers and the processors and is considered as an emerging and promising technology. Ultrasound is a technique in which sound energy is applied aiming at material processing to introduce new physicochemical functionalities in different food and non-food systems. Along with positive impacts of ultrasound energy, it may have negative effects, e.g. depolymerization of biopolymers and decreasing their functional properties. In this research the effects of ultrasound waves (20 kHz) at a range of powers and amplitudes at different temperatures (5 - 50°C) on viscosity, flow behavior index and intrinsic viscosity (i.e. molecular weight) of carboxymethyl cellulose solutions are investigated using response surface methodology. The relative importance of the three variables is compared and experimental models are presented to enable to estimate the resulting parameters. Overall, ultrasound treatment of carboxymethyl cellulose solutions can cause depolymerization of CMC macromolecules and its extent depends on temperature, time, power and amplitude differently.

Creep and recovery properties of ice cream mixes as influenced by different temperature levels

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Creep and recovery measurements were performed to observe the effect of temperature (5, 15, 25 and 35°C) on the viscoelastic properties of ice cream mix. In the creep and recovery phases, the system deformation per unit stress, called compliance (J), versus time data were obtained. The viscoelastic behavior of all systems was characterized by Burger model, based on the obtained data. The Burger model was well fitted to the ice cream mixes at different temperatures with determination coefficient close to unity. Instantaneous modulus of the Maxwell unit (G_0), the elastic modulus of Kelvin-Voight unit (G_1), the residual viscosity (η_0) and the internal viscosity (η_1) values decreased with increase in temperature, which were determined to range between 8.77 - 15.87 Pa, 0.48 - 2.75 Pa, 82.1 - 178.36 Pas and 28.37 - 59.73, respectively. G_0 values of the samples were greater than G_1 values, indicating that the ice cream mixes were deformed initially before a flow was established. The Burger model was also performed to recovery data of all systems. The recovery value due to the Kelvin-Voight element (JKV) increased with temperature and changed between 0.11 - 0.25 (1/Pa). Other recovery parameters B and C that define the recovery speed of the system, were in the range of 0.07 - 0.10 s^{-c} and 0.79 - 0.82, respectively. The percent recovery value decreased with increase in temperature and changed between 9.01 - 16.23 %. There was a direct proportion between permanent deformation as a result of applied force and temperature. The results of present study revealed that temperature caused a change in the creep-recovery parameters of the ice cream mix samples, which means the resistance of the samples to the applied stress based on the temperature. From rheological point of view, it can be suggested that the processing temperature of the ice cream mixes be important for rheological structure of the samples.

Simplex lattice mixture design approach for the production of wheat chips enriched with different legume flours: Optimization of textural characteristics

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In the present study, different legume flours (soy, pea and chickpea flours) were incorporated into the wheat chip formulation to increase the nutritional value of the snack. Experimental design was set up according to the simplex lattice mixture design. Textural characteristics of final products were measured using a texture analyzer equipped with 5 blade Kramer shear attachment. A predictive regression model was constructed for the hardness value with relatively high coefficient of determination ($R^2 = 0.80$). Increasing legume flour ratio in the formulation of chips provided a significant increment ($p < 0.01$) in the hardness values of final product. And also, interaction effect of legume flours was found to be significant ($p < 0.05$). The hardness values of samples ranged from 15.9-26.4 kg according to the chips formulation.

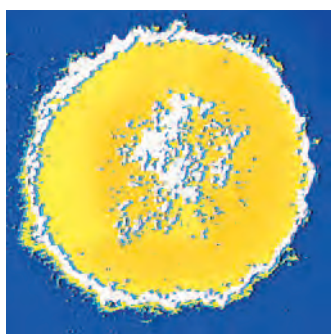
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Encapsulation

Microencapsulation of phenolic compounds extracted from sour cherry pomace

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Sour cherries (*Prunus cerasus* L.) contain phenolic compounds which decrease the proclivity to several chronic diseases such as types of cancer and cardiovascular diseases. The objective of this research was to study the effects of different coating materials, ultrasonication duration and core-to-coating ratio (CCR) on microencapsulation of phenolic compounds extracted from sour cherry pomace, which is a byproduct of juice production. For this study, maltodextrin (MD) and gum Arabic (GA) were chosen as coating materials. Different MD:GA ratios of 10:0, 8:2, 6:4 (total solid content 10 %) and two different core-to-coating ratios (CCR) of 1:10 and 1:20 were used. Emulsions were prepared by homogenization by ultrasonication at 160 W power and 20 KHz frequency for different time periods (5-30 min). Then the emulsions were freeze dried for 48 hours to obtain the microcapsules. The microcapsules were analyzed for surface and total phenolic contents, antioxidant capacities, surface morphology and moisture contents. Encapsulation efficiency was also calculated. The microcapsules having a CCR of 1:20, were found to have higher efficiencies (78-92 %) than those with a CCR of 1:10 (70-85 %). Encapsulation efficiency increased up to 15 min. of ultrasonication time and then remained constant. Because of its emulsifying effect, increasing GA ratio in the coating material decreased particle size of emulsions. Mean diameter of microcapsules prepared using MD:GA ratio of 10:0 was 3.229 μm while mean diameters were 1.735 and 1.613 μm for samples prepared with MD:GA ratio of 8:2 and 6:4, respectively (CCR of 1:20). There was no significant difference in antioxidant activities of microcapsules in terms of different types of coating materials. According to the results of surface morphology analyses determined by Scanning Electron Microscopy, microcapsules with CCR of 1:20 had smaller particle sizes than those with CCR of 1:10. When the size of samples sonicated for 15 and 20 min. were compared, it was seen that sonication for 20 minutes decreased particle size of microcapsule. There was no significant difference between moisture contents of capsules which were in the range of 3.27-5.63 %. Capsules prepared by sonication for 20 minutes and with CCR of 1:20 can be recommended to be incorporated into functional foods because they have the lowest particle size and high efficiency.

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Macromolecular Assemblies

Structural and physical properties of protein fibrils at fluid-fluid interfaces

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Long, semiflexible protein fibrils are obtained by combined acid and heat denaturation of globular food proteins, for example β -lactoglobulin. They bear many similarities with amyloid fibers, but can be tuned in their shape, flexibility, length and charge density by using various production protocols. These amyloid-like protein fibrils merge some of the unique properties of protein and rod-like particles and are therefore interesting candidates for the study of adsorption to and stabilization of air-liquid and/or liquid-liquid interfaces. The understanding of processes taking place at interfaces is of great importance not only in medicine but also in material and food science (emulsion or foam stabilization). A huge number of different systems has been investigated but information on protein fibrils at interfaces is still very limited [1, 2]. Here, we use a novel design of an optical microrheology device to probe the interfacial rheological properties of protein fibrils, which also allows for the exchange of the lower phase. In optical microrheology, the idea is to study the motion of colloidal tracer particles embedded at the interfacial layer. The thermal motion of particles can be analyzed to obtain information about the viscoelastic properties of the fluid interface, via the mean square displacement. In contrast to macroscopic interfacial rheology techniques, particle tracking can probe changes at the interface after shorter adsorption times as well as on smaller length scales. Correlating the results from particle tracking with shear rheological measurements helps us to understand the behavior of fibrils at liquid interfaces. This will for example help to understand the effects of protein fibril conformations on their 2D organization, i.e. their random or 2D liquid crystalline arrangement.

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Rheological properties of common wheat doubled haploids with the high grain protein content gene gpc-b1

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Rheological Properties of Common Wheat Doubled Grain protein concentration (GPC) is an important quality trait in bread wheat, which determines processing properties, quality of the end products, nutritional and market value of the grain. The application of the Gpc-B1 gene from *T. turgidum* ssp. *dicoccoides* into wheat breeding programs has the potential of improving GPC in a wide range of germplasm due to the absence of the functional allele in most of the commercial hexaploid cultivars. In this study, the effect of the Gpc-B1 allele on the rheological properties and bread-making quality of flour dough was evaluated in 68 double haploid (DH) lines. Genotypes were grown in three localizations in Poland during the autumn - summer 2010/2011. The samples were compared in respect of HMW- and LMW-GS composition, seed protein content, flour yield, rheological properties of dough and loaf properties. The quantitative and qualitative composition of particular HMW- and LMW-GS was determined by capillary zone electrophoresis (CZE). The rheological analyses were performed in micro-scale using 10g-mixograph, 10g-farinograph and the SMS-Texture analyser with Kieffer gluten extensibility rig. In the study material the Gpc-B1 introgression resulted in significant increase in GPC across genotypes and environments. Farinograph characteristics of the wheat flour also showed a significant increase in water absorption in most genotypes, which is a trait known to be highly correlated with GPC (4.5 - 8.4 % increase relative to control material). On the other hand, in some genotypes the Gpc-B1 introgression was associated with reduction of grain weight and flour yield. The increase in GPC associated with the Gpc-B1 introgression was also paralleled with improved gluten quality. The mixograph parameters showed that flour dough had better rheological properties than that of the recurrent parent. A positive effect on several bread-baking quality parameters was also observed in DH lines with the Gpc-B1 gene.

Effect of hydrophobic tail length on the self-assembly of food grade surfactants

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Surfactant self-assembled particles are formed from a low energy process due to their amphiphilic nature and are potential carriers for bioactive delivery. Despite surfactant particles have being widely explored in drug delivery systems, their application in food products is still scarce. Thus, the aim of this work was to study the effect of hydrophobic tail length of polysorbates (Tween-20 and Tween-80) on their aggregation process. The concentration of each polysorbate was varied from 10 to 90 % (v/v) and their rheological properties were determined by flow curves. Changes in particle size and conductivity were evaluated by Dynamic Light Scattering (DLS) and sample structures were observed by cross light microscopy. The results indicated particles with mean diameter of 4nm at 10 % (v/v) of Tween-20 probably related to micelles presence. Similar particle size was observed for Tween-80 at the same concentration. The conductivity of both samples showed a sharp increase with the enhancing of surfactant concentration up to 30 %, indicating the formation of more complex structures such as oblate micelles or hexagonal phases. The more viscous samples were observed at 60 % (v/v) of surfactant for both polysorbates, but viscosity was higher for Tween-80. Because of this, it was not possible to measure the conductivity and particle size of samples with higher than 60 % (v/v) of Tween-20 and from 40 up to 70 % (v/v) of Tween-80. Between 70 - 80 % (v/v) of Tween-80, the conductivity of samples became measurable probably due to the degradation of bilayers which tended to form reverse structures with reduced viscosity. All Tween-20 systems showed Newtonian behavior and liquid appearance, however the samples composed by 50 - 70 % (v/v) of Tween-80 were gel-like and showed shear thinning behavior. Such characteristics were probably related to the formation of a liquid crystalline network. In fact, the cross light microscopy confirmed the formation of a birefringent network, usually associated to this type of structure. Thus, the results suggest that as Tween-80 has a longer hydrophobic tail and bulkier side chains it can form a wider range of structures than Tween-20.

How to obtain a galactomannan standard for solution rheology?

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Water-soluble polysaccharides extracted from legume seed endosperms such as galactomannans are well known as viscosifiers due to their high molecular weight. The molecular structure has been found to consist of a backbone of mannose with galactose sidegroups at variable degree of substitution according to the specific plant source [1-2]. Galactomannan polysaccharides extracted from seed endosperms of various species have been characterized by size-exclusion chromatography, dilute-solution viscometry, and oscillatory-shear rheology to determine their potential as aqueous thickeners. The molecular composition and chain-length distribution were found to be nearly identical, and thus galactomannans within the same genus are presumed to share a common molecular structure. Obtained scaling properties suggest that in contrast to common belief, water can be considered as a good solvent and therefore the Colby-Rubenstein model can be utilized to describe galactomannan solution rheology [3]. Solutions above the crossover concentration exhibited shear-thinning behavior, and strong dependence of viscosity on concentration. Seed characteristics such as shape, mass, and endosperm content were also assessed, and based on this investigation, some legume endosperms are advantageous for industrial processing and could be adapted for e.g. guar gum replacement [4-6].

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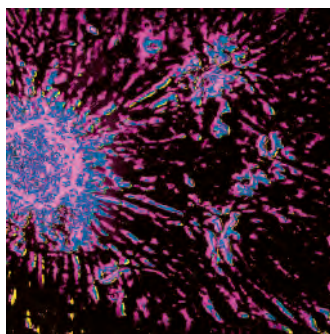
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Structure, Nutrition and Health

On the use of cross-linked tapioca starches to reduce oil absorption in deep-fried chickens

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This work was performed to investigate the effectiveness and feasibility of using cross-linked tapioca starches in batter formulations to reduce the oil absorption in deep fried chicken. Different degrees of cross-linked tapioca starches, i.e. low, moderate and high, were used. Water retention capacity (WRC), viscosity, pick-up as well as cooked yield of the prepared batters were examined. Differential scanning calorimetry was used to determine the oil uptake of fried chicken crust samples. Such a technique is faster and easier to perform, and also requires less samples compared to the conventional solvent extraction method. The viscosity results showed that all studied batters were pseudoplastic fluids, i.e. having shear thinning characteristics. The substitution of 20 % cross-linked tapioca starches for wheat flour in the batter formulations significantly reduced the viscosity and WRC of the batters. However, batter pick-up and cooked yield values were insignificantly affected. In comparison to control (wheat) formulation, crusts of fried chicken samples obtained from the formulations containing 20 % cross-linked tapioca starches showed significantly lower oil content by at least 16.9 % relative oil reduction. The highest relative oil reduction was found in the batter containing the highly cross-linked tapioca starch. It was hypothesised that the chemical cross-linking, which makes starch granules rigid and persistent to high temperature as well as limits their hydration property, could be the predominant factor in the reduction of oil content for the cross-linked starch-substituted batters.

Influence of food viscosity on aroma release after swallowing: A combined biomechanical modelling and in vivo study

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After eating a liquid or a semi-liquid food product, a thin film responsible for the dynamic profile of aroma release coats the pharyngeal mucosa. A biomechanical model of swallowing was developed to understand the role of saliva and food bolus viscosity on the coating of pharyngeal mucosa. An analysis of the biomechanics of swallowing showed that the pharyngeal mucosa coating are due to a thin film flow, stationary, in a soft elastohydrodynamic contact whose the kinematics is equivalent to a forward roll coating process lubricated by saliva. Based on this analysis, a biomechanical model of swallowing was developed for Newtonian liquids. Two sets of conditions were distinguished. The first one was obtained when the saliva film is thin, in which case food bolus viscosity has a strong impact on mucosa coating and on flavor release. The second was obtained when the saliva film is thick and the food bolus coating the mucosa is very diluted by saliva during the swallowing process. Moreover, at the light of the present numerical simulations, we show that shear thinning, viscoelasticity and yield stress of saliva and/or food product should affect mucosa coating phenomena quantitatively. In parallel, in vivo aroma release from glucose syrup solutions varying widely in viscosity (from 0.7 to 405 mPas) was assessed by five panelists using Proton Transfer Reaction Mass Spectrometry. Aroma release kinetics presented maximal differences of 30 % between the most extreme products. Results obtained by the biomechanical model were integrated in a mechanistic model describing aroma release while eating a liquid food. Under the second set of condition described herein, the aroma release model gives a satisfactory prediction of the in vivo data. We conclude that the viscosity of the initial product has a limited effect on aroma release for products with Newtonian properties because viscous solutions are highly diluted by saliva during the swallowing step and the relevant properties are those of relatively similar product saliva mixtures [1, 2].

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[2] Doyennette et al., Food Chemistry, 2011

The effect of minerals concentration on the stability of high caloric oral nutritional supplements

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The stability of high caloric oral nutritional supplements (ONS) has been investigated as a function of minerals concentration, in terms of viscous flow behavior, particle size distribution, conductivity and thermal resistivity. Commercial-based (Fresubin®) protein energy drink, Fresenius Kabi Deutschland GmbH) oil-in-water emulsions, 6.3 % weight vegetable oil, were prepared by using milk protein (MP, 9.4 % weight), as emulsifier, dispersed in water (74 % weight) at several minerals concentrations, with and without added cocoa powder. In the present study, the following minerals have been considered: sodium, potassium, calcium, magnesium, phosphorous and chloride. Heat coagulation tests have been performed in order to investigate the tendency to gelation. After heat treatment, the consistency index decreased for solutions having low minerals concentration, while the opposite effect, that is an increase in consistency index and a decrease in flow index, was noticed when increasing the amount of minerals. In addition, heat treatment only yielded a dramatic increase in emulsion consistency for samples with very high minerals concentration. Concerning particle size distributions, a bimodal dispersion was observed when adding cocoa powder. As cocoa concentration increases, particle size tends to increase, no matter minerals concentration is.

Influence of sugar replacers on the mechanical and rheological properties of doughs for sweet baked goods

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Sweeteners are important ingredients in bakery products. Besides providing a sweet taste, they also affect fermentability, appearance, flavor, dimensions, color, and texture of the finished products. There are many available choices of sweeteners, and the type chosen depends on the functions to be performed and on the desired level of sweetness, appearance and texture of the baked product. As regards human diet, sugars play a double role: they are a fundamental energetic source, but their excessive intake has been often connected to short- and long-term pathologies. Therefore, food industries express a growing interest in sucrose substitutes to be used in low-sugar products. However, when the sugar content is changed, food rheology and texture may be negatively affected. Even if often considered secondary, and thus little investigated, these aspects have a fundamental importance, since a novel food adequately formulated from a nutritional and healthy point of view should also be pleasant and satisfactory at consumption. Thus, the aim of this study was the evaluation of the effects of the presence of different sweeteners on the mechanical and rheological properties of doughs for sweet baked goods. Different croissant-type formulations without sweeteners or containing, as alternatives, sucrose, fructose, or sucralose were investigated. The structure of the dough was evaluated by means of different techniques able to describe the intrinsic properties of the material and to predict its behavior in a real process. Both empirical (farinographic test, dynamometric measurements) and fundamental rheological tests (dynamic oscillatory measurements) were performed. Generally, the doughs containing sucralose showed mechanical and rheological properties very similar to those of the doughs produced with no added sugars: they resulted to be firmer, more resistant to tensile forces and less viscous than the dough containing sucrose or fructose. It came out how the use of alternative sweeteners, and of a high-intensity sweetener in particular, have to face with the technological challenge of a complete substitution of sucrose properties. This is the reason why frequently other ingredients (e.g. bulking agents) should have to be added to low-sugar baked goods to compensate sucrose functionalities complementary to sweetening.

Structure–mechanical properties of pastry dough and products with Jerusalem artichoke (*Helianthus tuberosus* L.) powder

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Structure–mechanical properties for pastry products are important parameter in the research of quality control in products' usage. Texture is one of a general quality traits related to pastry products' typical quality. Jerusalem artichoke powder (JAP) made from Jerusalem artichoke roots is a valuable product and its addition to pastry products increases their nutritional values. The addition of JAP in pastry products modifies structure–mechanical properties and their typical structural characteristics. The aim of this study was to evaluate how the addition of JAP in different concentration levels influences structure–mechanical parameters of such pastry products as cakes, butter biscuits and honey biscuits and their dough. Structure–mechanical properties have been evaluated by a texture analyzer "TA.XT.plus". For experiments were used pastry dough and products, which prepared by the classic recipe and technology and part of wheat flour in them was substituted with JAP in concentrations of 10, 20, 30, 40 and 50 %. As control sample were used pastry products without JAP. Experimental results showed that addition of JAP has a significant impact on all the studied pastry dough's structure ($p > 0.05$). Increasing of JAP concentration in dough's structure was getting softer. So addition of JAP in pastry products increases the quantity of pentosans. Physical characteristics of pentosans are determined that they are strong swelling, linking a lot of water and forming a gel, in resulting them dough is getting a softer and stickier. This explains why the greatest influence on the structure of the dough of honey biscuits: it becomes much softer and stickier, because exactly it is the highest proportion of flour ($\sim 50\%$) in comparing with the cakes and butter biscuits. Structure changed significantly in comparing with control, when adding JAP in pastry products. However, the concentration of JAP has not significant impact on the cakes and butter biscuit hardness, but existing small changes was showed the observed trend. Honey biscuits there are significant structural changes affecting of honey and the observed trend is drastic. As a whole, the addition of JAP modifies the pastry products typical structural characteristics: cakes become denser, butter biscuits - more fragile, honey biscuits - stickier.

Effect of immobilization and salt concentration on the growth dynamics of *Salmonella Typhimurium*

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A gelified environment does not only immobilize microbial cells but also gives rise to differences in growth dynamics (e.g., growth rate) as compared to growth in liquid systems. Literature suggests that (i) the growth region of bacteria is limited by this structure-induced stress, and that (ii) a gelified environment can enhance survival and growth. *Salmonella Typhimurium* grown at 23.5°C in (a) liquid Brain Heart Infusion (BHI) and (b) BHI gelified with (i) gelatin, (ii) xanthan gum and (iii) a binary mixture of gelatin and xanthan gum, results in quasi coincident growth curves. Contrary to what is mentioned in literature, the gelling agent (and resulting immobilization) does not seem to have an effect on growth dynamics at moderate conditions. To further elucidate this phenomenon, growth was studied at stressing conditions, i.e., at five salt concentrations ranging from 1 to 5 % (w/v) added salt. Static experiments were performed in BHI, structured with (i) gelatin (100 g/L), (ii) xanthan (15 g/L) and (iii) a binary mixture of gelatin and xanthan, in parallel with experiments in liquid BHI. Hereto, 1mL of liquid or structured medium is brought in spectrophotometer tubes, which are simultaneously placed in a temperature controlled water bath. At regular times, a tube was removed and cell density was determined via plate counting. Temperature was set at 23.5°C, i.e., a practically feasible temperature at which the gelified media are rheologically stable. Preliminary results show a decreasing growth rate and an increasing lag phase with increasing added salt. Coincident growth curves are observed for all media with 1 % added salt. However, when 2 and 3 % salt is added, a stronger decrease in growth rate and a longer lag phase is observed in the liquid system compared to the gelified systems. Results for 4 and 5 % added salt show a constant cell count within the first 8 hours of the experiment except for gelatin in 4 added salt, for which a small increase in cell count is observed after 6 hours. These preliminary results suggest that adding a gelling agent, i.e., inducing microbial immobilization, enhances survival and growth in salty environments.

Effect of immobilization on the inactivation dynamics of food pathogens

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In predictive microbiology, mathematical models are developed that enable description of the microbial behavior in food products, from production to consumption. Often applied processing procedures are sterilization and pasteurization, and, more recently, mild heat treatment, a technique that kills undesired microorganisms while maintaining textural and nutritional quality. Up till now, models describing inactivation have been developed based on data obtained from liquid laboratory media or liquid food systems. However, many foods have a solid or gelled character. Compared to liquid systems in which microorganisms grow planktonically, such a solid(like) environment leads to the formation of colonies. In this work, the effect of growth morphology, i.e., planktonically grown cells vs. cells grown as colonies, on the heat inactivation of *Escherichia coli* and *Salmonella* Typhimurium is investigated. *E. coli* is studied in a system that consists of Brain Heart Infusion broth (BHI) gelified with xanthan gum. For *Salmonella*, xanthan gum is combined with Tryptic Soy Broth (TSB). On the one hand, both microorganisms were grown in the xanthan gum system at 37°C for 24 h to reach stationary phase colonies. On the other hand, liquid cultures were obtained after growth for 24 h at 37°C in BHI or TSB without xanthan gum. Subsequently, glass tubes containing small volumes of the liquid culture or the colony culture were placed in a water bath at 54°C. Regularly, tubes were removed and cell density was determined via plate counting. For *E. coli*, the inactivation curves reveal a higher heat resistance when grown as colonies. Inactivation rates for the liquid culture are around 0.13 1/min, while inactivation rates of the colony cultures are around 0.08 1/min. For *Salmonella*, inactivation curves evolve approximately in parallel up to 100 min, i.e., inactivation rates are similar. Hereafter, a tailing phase is observed for the colony culture, i.e., a resistant subpopulation seems to exist. Generally, cells within a colony inactivate at a slower rate than planktonically grown cells. This implies that the currently available predictive models, which have been developed for liquid cultures, are no longer fail-safe. Further research is needed to extend models towards all types of growth morphologies. This will lead to more accurate models with practical relevance to food industry.

Generation of sub-micron iron for food fortification

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Iron deficiency is the most common and widespread nutritional health problem in the world. The effect of the prevalence of nutritional iron deficiency can be reduced by food fortification, which is a challenge. Water-soluble, highly bioavailable iron compounds often cause adverse sensory changes in food during storage and preparation. On the other hand, poorly soluble iron compounds, although more stable in food, tend to have lower bioavailability. Particle size reduction is one approach to increase the bioavailability of poorly soluble iron compounds. It could be shown that nanoparticles of ferric phosphate made by flame spray pyrolysis have a solubility and bioavailability similar to ferrous sulfate, the reference Fe compound for nutrition due to its high absorption [1 - 3]. Our goal in this project is to produce sub-micron iron in a top-down process by micro-media milling, and to investigate process parameters such as bead size or rotor speed and the role of surfactants. Results show that solubility of micro-media milled iron performed as well as flame-spray-pyrolysis generated iron (bottom-up process) and iron sulphate. Moreover micro-media milled iron succeeded in a rice extruded application, too.

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Multi-scale characterization of the evolution of the food structure during digestion

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Digestion of nutrients is an essential function for human to allow normal growth and development. During digestion, the structure of the food is strongly modified and detailed characterization of the digestion products is essential for a better understanding on how food is disintegrated in the gastro-intestinal tract. It appears therefore crucial to have methods for characterizing the evolution of the matrix at different scales, from the macroscopic to the molecular level. Different methods such as biochemical techniques (SDS-PAGE, mass spectrometry) but also molecular immunology (ELISA) have been developed and allowed the monitoring of the proteolysis throughout the gastro intestinal tract at a molecular level. Evolution of the food at the microscopic level was evaluated by confocal microscopy and light scattering. In order to characterize the rheological properties and the clotting ability of a liquid dairy sample in the stomach, an infant formula was placed in an AR 1000 rheometer (TA Instrument) and glucono-delta lactone (GDL) was added in order to mimic the decrease in pH that will occur in the stomach of the neonate after meal ingestion. Formation of the acid gels was monitored using low-amplitude dynamic oscillation by measuring the elastic modulus (G'), the loss modulus (G'') and the loss tangent of the sample under acidification at 37°C. The applied deformation was 0.1%, frequency was 1Hz, and gel formation was followed for at least 4h. The onset of gelation was defined as the time when $G' > 1\text{Pa}$. Combination of this set of techniques allowed demonstrating that an increase in the intensity of the heat-treatment applied to the milk concentrate during infant formula manufacture increased significantly the firmness of the bolus in gastric conditions and modified the kinetics of proteolysis of the milk proteins. We also demonstrated that the presence of insoluble particles in a rehydrated infant formulas lead to the absence of gel formation in the stomach.

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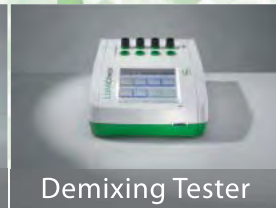
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