International Symposium of Food Rheology and Structure

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Book of Abstracts

PL Plenary lectures

PL 1. Neutrons and Food: From emulsion and foams to analogues

Peter Fischer
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Neutron and x-ray scattering techniques are used to study the structure and thus the performance of multiphase food systems. Surface active materials such as surfactants, proteins, and particles are used to stabilize foams and emulsions with a protective adsorption layer. With small angle neutron scattering and neutron reflectivity measurements we were able to show the adsorption of small molecular weight surfactant in raft-like aggregates that allow limited coalescence followed by a long-time foam stabilization for example used in ice crème manufacturing [1-3]. Neutron and x-ray reflectivity measurements were used to understand the adsorption of proteins at liquid interfaces and thus link interfacial and bulk rheological properties to the structure of the protein layer [4, 5]. The same techniques were used to design stimuli responsive Pickering emulsions made from cellulose nanocrystals and protein composite layers. It could be shown that the structural and mechanical properties of the emulsion systems can be controlled by surface modification of the cellulose nanocrystals and thus are able to react to physiological triggers during human fat digestion [6-8]. Scattering methods were used to elucidate the morphology of protein-based meat analogues. Here in situ extrusion experiments in the neutron beamline could show the structure development in real time and along with contrast variation also the role of the main ingredients for the final product properties. With high-resolution x-ray scanning scattering also the orientation and composition of meat analogues could be linked to the extrusion flow field and sample rheology.

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[2] Lutz-Bueno V, Zhao J, Mezzenga R, Pfohl T, Fischer P, Liebi M: Scanning-SAXS of microfluidic flows: Nanostructural mapping of soft matter, Lab on Chip 16 (2016) 4028
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PL 2. Quantifying food structure formation under flow by MRI

Camilla Terenzi
Wageningen University, the Netherlands

Heterogeneous structured food materials, in either liquid-state dispersed or semi-solid form, undergo complex physico-chemical phenomena during food manufacturing. These depend on formulation composition and process conditions, and ultimately determine the macroscopic and sensorial properties of food systems. Key examples include, among others, shear-induced flow enhancement in structured microgel networks or fat crystal dispersions (FCDs), and manufacturing of meat alternatives involving anisotropic structure formation via extrusion of plant-based biopolymer mixtures. Commonly adopted techniques for mechanical/rheological analysis and microscopic/spectroscopic characterization are often destructive, of limited or no use for optically-opaque materials, and unable to provide both spatially- and chemically-resolved quantitative information. Nuclear Magnetic Resonance (NMR) and Magnetic Resonance Imaging (MRI) techniques enable overcoming all these issues, and can be adapted to acquire in situ measurements during flow.

In this talk, the performance of MRI measurements, and related modelling approaches, will be shown, in dynamic or static conditions, for two case studies: (i) in situ MRI measurement and modelling of cooperative microfluidic flow behaviour of yoghurt[1] and FCDs[2,3]; (ii) ex situ imaging and quantification of multiscale protein anisotropic structure formation via deadstop operation of high-moisture extrusion[4,5].

Ongoing and future applications in in situ assessment of structure formation by food 3D printing and extrusion, aimed at enabling comprehensive food soft matter characterization under industrially-relevant conditions, will be presented.

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PL 3. Foods inside out: unveiling the hidden architecture of nanostructured soft materials by super-resolution microscopy

Ilja Voets
Eindhoven University, the Netherlands

Understanding how the structure of complex materials composed of different ingredients develops and relates to their functional properties is actively investigated in many fields ranging from materials science to supramolecular chemistry, from soft matter to biophysics, and from biomedical engineering to food science. Advances in both scattering and microscopy tools offer more and more insight in the evolution of the hierarchical structure of such materials across orders of magnitude in length- and time scales [1-3]. In this talk I will focus on recent work from my group, in which we use super-resolution microscopy for in-situ, three-dimensional, sub-diffraction imaging of complex soft materials with chemical specificity and minimal perturbation [4]. I will showcase how we used nanoscopy to study the behavior of single proteins at solid-liquid interfaces [5], the position of individual particles at liquid-liquid interfaces [6,7] and to quantify the distribution of proteins in protein/polysaccharide gels [8].

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[5] Tas, R.P.; Hendrix, M.M.R.M.; Voets, I.K.; Nanoscopy of single antifreeze proteins reveals that reversible ice binding is sufficient for ice recrystallization inhibition but not thermal hysteresis, Proceedings of the National Academy of Sciences of the United States of America 2023, 120 (2), e2212456120
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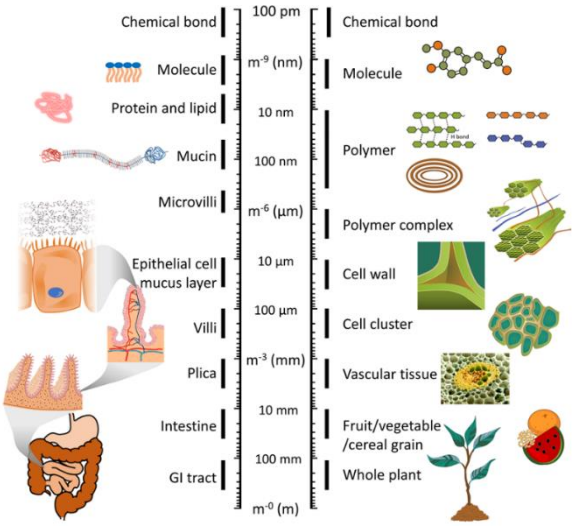
[7] Giakoumatos, E.; Aloi, A.; Voets, I.K. Illuminating the Impact of Nanoparticle Size and Surface Chemistry on Interfacial Position, Nanoletters 2020, 20, 7, 4837–4841
[8] Foroutanparsa, S.; Brüls, M.; Maljaars, C.E.P.; Tas, R.P.; Voets, I.K.; Spatial distribution of αs1-caseins and β-caseins in milk gels acidified with glucono-δ-lactone Food Hydrocolloids 2023, 139, 108506

PL 4. Dietary Fibre in the Gastrointestinal Tract: Emergence of Health Functionality from Rheology, Physical Interactions and Biochemical Transformations

Gleb Yakubov
University of Nottingham, UK

Dietary fibre functionality is greatly impacted by rheology. Rheological properties play a critical role in various physiological processes such as controlling gastric emptying, digestion of macronutrients, colonic fermentation, and supporting healthy microbiome. This presentation focuses on the journey of dietary fibre through the gastrointestinal tract, from oral processing to the colon, with particular emphasis on how different rheological properties affect physical interactions and biochemical transformations of fibre materials. Examples are provided to illustrate how complex rheological properties can elicit specific physiological responses that lead to positive health outcomes and long-term wellbeing benefits.

One example presented in this talk is the effect of the gelling properties of complex arabinoxylans from Plantago ovata (psyllium) on in vitro colonic fermentation. The presentation shows how polysaccharides with similar composition but different rheological properties display distinctly different fermentation behaviours due to the ability of bacteria to colonize the gel particles and for the glycosidase enzymes to attack the polysaccharide chains. Additionally, the talk explores the hydrodynamic interactions between soluble cereal arabinoxylans with mucins and demonstrates how different structures of dietary fibre can affect mucus properties and its barrier function.



Furthermore, the presentation covers new approaches for exploring the structure and rheological behaviour of complex fibre polysaccharides, which allow monitoring of the dietary fibre response to digestive environment both in vitro and in vivo.

[1] Harris H.C. et al. (2023) The impact of psyllium gelation behaviour on in vitro colonic fermentation properties, Food Hydrocolloids, 139, 108543
[2] Yu L. et al. (2021) Viscoelastic behaviour of rapid and slow self-healing hydrogels formed by densely branched arabinoxylans from Plantago ovata seed mucilage, Carbohydrate Polymers, 269, 118318
[3] Gidley M.J. & Yakubov G.E. (2019) Functional categorisation of dietary fibre in foods: Beyond 'soluble' vs 'insoluble', Trends in Food Science & Technology, 86, 563-568
[4] Meldrum O.W. et al. (2017) Mucoadhesive functionality of cell wall structures from fruits and grains: Electrostatic and polymer network interactions mediated by soluble dietary polysaccharides, Nature Scientific Reports, 7(1), 1-15

PL 5. Designing the stability of aerated food products by bulk and interfacial rheology

Valeria Garbin
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Foams made from edible oils are a potential substitute for solid fat, combining solid-like behavior with low-fat content and appealing organoleptic properties. Structuring vegetable oils enables to replace solid saturated fats with poly-unsaturated fats, which are liquid at room temperature, all while achieving the desired mouthfeel and ensuring consumer acceptance. An example is oleofoams, which are dispersions of gas bubbles in a continuous oil phase, stabilized by crystals of fatty acids or surfactants adsorbing at the oil-air interface. Because excess crystals in the continuous phase form an oleogel, the bulk rheology of the continuous phase also affects their formation and stability. The stability and mechanics imparted by both the interfacial layer of crystals and the bulk oleogel underpin product shelf life, as well as optimal processing conditions and performance in applications. We use temperature-controlled microscopy and rheological measurements to provide a fundamental understanding of the micromechanics of the interfacial layer, and to decouple the roles of bulk and interfacial rheology on oleofoam stability. We discovered an unexpected mechanism of destabilization that is not captured by classical criteria for bubble stability. We also show that the fate of the interfacial crystal layer upon deformation is highly dependent on the deformation rate, with implications for shelf life and processing flow conditions.

PL 6. Challenges and opportunities in plant protein based delivery systems

Stephan Drusch
Technical University of Berlin

Various microencapsulation techniques are used to protect functional food ingredients such as nutritional oils, vitamins, colorings or microorganisms against external influences such as light, oxygen and temperature and the associated changes. Furthermore, delivery systems are used to release functional ingredients upon a specific trigger during food processing, storage or consumption and digestion. Encapsulation techniques for food ingredients on industrial scale include, but are not limited to, spray drying, extrusion and complex coacervation. Most frequently matrix materials included animal-derived proteins like gelatin or whey protein and in general, scientific understanding and product development have reached maturity. In the last decade, interest in plant protein-base delivery systems has exponentially increased. However, this is not necessarily attributed to their superior functionality, it is rather driven by the increasing demand for vegetarian and vegan food products.

A first obvious issue is the poor, pH-dependent solubility of plant proteins. In emulsion- and gel-based delivery systems solubility is a pre-requisite during early stages of formulation. Process conditions during protein recovery may address this issue, while there is also a range of techniques to modify protein functionality ex post. Furthermore, commercially available plant proteins represent the sum of a wide structural variety of individual proteins. Fundamental investigations of interfacial behavior and stress response of different plant proteins clearly show that there are both, synergistic and antagonistic effects between protein fractions. This holds true on one hand for their emulsifying properties, on the other hand for protein aggregate formation in gelation. Both are highly relevant for tailoring delivery systems. Non-soluble fractions of plant proteins may be favorable in delivery systems, in which reactions between the encapsulate and the protein matrix must be expected.

It is therefore obvious that plant proteins represent challenging matrix constituents, if one thinks of a simple replacement of animal derived proteins in delivery systems. However, one may look at it as an opportunity for innovation, which is not fully understood in its potential to overcome current limitations and give a push towards a new generation of delivery systems.

PL 7. Large and small deformation rheology in multi-phase food products

Theo Blijdenstein
Unilever Innovation Center, the Netherlands

Many Food products consist of different phases of immiscible liquids, solids and sometimes gas. Nevertheless, the products should live up to consumer's expectations, despite of a usually complex interplay between the components of which it consists. Moreover, the intermediate products have to live up to the processing constraints in a supply chain environment which is typically rigid in nature. During the production, handling and storage, products experience various forces like gravitational, shear, elongational/compressional and interfacial forces.

At the same time, the food industry is committed to move to more plant-based options, reducing waste and improving the nutritional aspects of its products. This poses and additional challenge to the product formulation and process control.

In this lecture, some case studies will be presented in which various rheological approaches towards product functionality will be shown and how these can be used to rationalize product behaviours, in process as well in end-use as in its production.

PL 8. Some microfluidic tools for studying structure and stability in food dispersions

Deniz Gunes
KU Leuven, Belgium

Microfluidic tools have been growing in their use in food and bioengineering sciences since their surge about two decades ago. Specific to food science and engineering, they proved useful to help unlock the potential of many systems for food structure design, in particular for food emulsions and foams. That holds specifically with regard to questions relating to structuring under flow at interfaces, as well as in bulk e.g. in suspensions of attractive particles. Microfluidics further helped investigating the phenomena destabilizing emulsions and foams, and questioning the influence of interfacial vs bulk parameters in the stabilization of foams.

In this talk, we will give some examples illustrating each of the above cited cases, covering in particular:

- design of food-based encapsulation systems based on interfacial complexation [1]
- study of protein aggregation and formation of protein-based fibrous structures using microfluidics [2,3]
- mass transport in emulsions and foams (e.g. Ostwald ripening) [4-7]
- coalescence in emulsions and foams [9,10]

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[8] Decoupling of mass transport mechanisms in the stagewise swelling of multiple emulsions. Langmuir 31 (19), 5265 (2015).
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[10] A study of extensional flow induced coalescence in microfluidic geometries with lateral channels. Soft Matter 9 (31), 7526 (2013).

A Colloidal suspensions

Monday, 12th of June 2023, Bosrandzaal

A1. Thermogelation nanoemulsions stabilized by pea protein

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Massachusetts Institute of Technology, USA

Nanoemulsions are kinetically stabilized droplets with diameters on the order of 100 nm. They are used in food science for fortification due to their versatility and stability against environmental changes. A food product can be enriched with hydrophobic nutrients such as vitamin A or essential oils by dissolving them in the nanoemulsion droplets. Nanoemulsions are also used in the production of plant-based dairy products. With a droplet size on the order of the wavelength of visible light, they have a similar appearance to dairy milk.

We form a peanut oil-in-water nanoemulsion with ultrasonication at room temperature and use pea protein isolate as surfactants to stabilize the nanoemulsion. A successive increase in temperature induces denaturation of the adsorbed proteins which leads to gelation of the nanoemulsion.

The application of shear flow during the gelation process introduces orientation to the forming structure. Our rheological investigation provides a detailed description of the gelation process as well as the final mechanical properties of the nanoemulsion gel. In addition, we accompany the measurements with microscopy tools to visualize the formed structure.

The structure of the nanoemulsion serves as a template for the final texture of the food product. Thus, we demonstrate with thermogelation the potential of nanoemulsions as applications beyond milk analogues and fortification, but also as precursor for denser structures.

A2. Plant protein functionality-driven refinement: linking plant protein refinement with multiphase functionality using interfacial rheology

Jack Yang, Claire Berton-Carabin, Constantinos Nikiforidis, Erik van der Linden, Leonard Sagis

WUR, the Netherlands

Plant proteins play a crucial role as functional ingredients in our foods. One such functionality is stabilising multiphase systems, such as emulsions and foams. Currently, the major focus is to perform extensive refinement, yielding high-purity ingredients. But a problem here is the impact of the refinement process on the protein's functional properties. In this presentation, we will show a series of works, where the plant protein refinement process is altered, which will change protein molecular and functional properties.

The molecular properties were linked to functional properties (emulsion and foam stabilisation) by extensively characterising the interfacial properties. Here, techniques such as surface dilatational and shear rheology are performed, especially at large deformations, to enter the nonlinear viscoelastic regime. The interfacial microstructure was also visualised using atomic force microscopy on Langmuir Blodgett films. This combination of techniques allowed us to establish strong links between the various length scales. Plant proteins were refined using the conventional alkaline-acid precipitation processes; and compared to less resource-intensive and more mild refinement processes. We found that the conventional method gave significant protein aggregation, lowering protein solubility. Also, the conventional process removes a group of proteins called albumins, which can form stiff viscoelastic interfacial layers, thereby giving extremely stable foams. Using mild refinement, we were able to create extracts with higher solubility; and also extract the albumins. This was found to improve overall functionality. However, several challenges remain in the mild extraction, such as retaining non-protein components. We will also show how the impact of phenols and lipids can be controlled in mildly extracted protein systems.

In our approach, the plant protein's functional properties can be directly linked to the protein refinement processes. This allows us to control the protein functional properties by using the refinement processes. This functionality-driven refinement could have a high potential as a tool to control protein functionality, thereby assisting the industry in structuring plant-based food products.

A3. Development of an innovative plant-based mayonnaise stabilized by vegetable proteins

Rui C. Pereira, Arlete M. Marques, Marta V. Vieira, Ana I. Bourbon, Kamila Calderón, Pablo Fuciños, Diogo Castelo-Branco, Ana Tasso, Diogo Figueira, Lorenzo M. Pastrana and Miguel A. Cerqueira

International Iberian Nanotechnology Laboratory, Portugal

Over the past years, plant-derived proteins have been emerging and growing in interest, due to their unique functionalities and trend to replace animal-derived proteins, such as egg protein [1]. Their use in the development of emulsions has been explored but several changes arise, such as emulsion instability, and low emulsification capacity. In this work, we develop oil-in-water nanoemulsions using ultrasounds (US) and plant-based emulsifiers, aiming the production of mayonnaise without the presence of the common additives, and maintaining their traditional sensorial characteristics. Oil-in-water nanoemulsions were produced through high-speed homogenization, followed by US, using lupin protein as emulsifier. A 23 central composite rotatable experimental design was used to evaluate the influence of three independent variables: water:oil ratio (65-75% of water), protein content (1-4%) and US time (1-7 min) on the average size and polydispersity index (PDI) of the nanoemulsions. A total of 17 experiments were performed with 14 three-level experimental points, and 3 replicates at the central point. Once the best conditions to develop the nanoemulsion were selected, salt, sugar, hydrocolloids, and vinegar were added under agitation in a homogenizer, at 10000 rpm, for 5 min to freshly prepared nanoemulsions. The mayonnaises were placed in cylindrical jars and refrigerated (4 °C) for 24 h before rheological measurements.

Results showed that the use of lupin protein led to stable nanoemulsions for 50 days stored at 4 °C. The smallest mean droplet size for lupin protein was 505.5 nm and PDI value 0.434 [23.6:73 (w/w) oil/water ratio, 3.4% (w/w) of protein and 6 min of US]. The mechanical spectra showed a similar slope and pattern for both plant-based mayonnaise formulations (with fibers and starch, respectively) and commercial one. The oscillatory rheological tests indicated a predominantly elastic behaviour of the samples (G'>G"). The textures of the plant-based formulations were very close to commercial mayonnaise, with similar resistance to deformation. The consistency (k) and flow index (η) calculated from the Herschel-Bulkley model are similar between samples, and indicate that the samples are viscous due to the considerable value of k. US seems to be an effective strategy to develop plant-based nanoemulsions foreseeing their use in the production of vegan mayonnaises with promising rheologic and texture characteristics.

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[2] Comparison of Different Protein Emulsifiers on Physicochemical Properties of β-Carotene-Loaded Nanoemulsion: Effect on Formation, Stability and In Vitro Digestion, Liu et al., Nanomaterials, 11, 167, (2021).

A4. Effects of different anti-freezing agents on ice crystal size and physical properties of ice cream

Qi Wang, Guido Sala, Elke Scholten

WUR, the Netherlands

Ice cream is a popular dairy product, and usually contains a lot of sugar. Due to the negative health effects of sugar, the development of low-sugar ice creams is desired. However, the removal of sugar in ice cream is not straightforward, as it plays a pivotal role in determining texture and sensory properties. Besides providing sweetness, sugar is mainly responsible for controlling ice crystal growth and final ice fraction. In addition, the high concentration of sugar in the unfrozen matrix provides a high viscosity, which imparts "body" to the ice cream. Therefore, suitable replacers for sugar need to compensate multiple functionalities. The aim of this research was to gain more understanding of the role of different antifreeze agents, such as sucrose, glucose, xylitol, ethanol and salt, on the properties of ice cream. We first determined by differential scanning calorimetry the hydration numbers of these ingredients to calculate the ice content at different temperatures of the ice creams made with them, and thus to establish their ice curves. Below 0 ℃, the hydration number was negatively correlated with concentration but positively correlated with molecular weight. The ice curves were therefore dependent on the type of anti-freeze agent used.

Next to the ice content, we also investigated the effect of these anti-freeze agents on ice crystal formation. Performing microscopy characterization at -18 ℃, we found that the ice crystal size was affected by the type of anti-freeze agent, and relations between ice crystal size, ice content and molecular weight were found. Ice content and ice crystal size had a large effect on ice cream hardness and melting properties due to differences in ice connectivity. Ice cream with smaller ice crystals was harder and showed a slower melting profile. Based on the evolution of the ice content, we also determined changes in viscosity of the serum phase as a function of temperature. Serum phase viscosity had a large effect on the lag time before the ice cream started to melt but did not affect the melting rate itself. These results show that different anti-freezing agents control both structural and physical features of ice cream, and are of relevance for the formulation of low-sugar products.

A5. The effect of cellulose nanocrystals on the stability of Pickering emulsion prepared with the microalgal protein

Wonsik Shin, Joung Sook Hong, and Kyung Hyun Ahn

Seoul National University, Korea

Pickering emulsion is a suspension of droplets of one immiscible fluid in another, of which droplets are stabilized with particles. In recent years, there is an increased interest in the use of microalgal protein as natural surface-active molecules due to their high nutritional value, which is based on their high protein content. However, the stability of the protein-stabilized Pickering emulsions was relatively poor, which was attributed to the relatively large oil droplets. Therefore, polysaccharide-based stabilizers have been incorporated into these emulsions to improve emulsion stability. In this study, we used cellulose nanocrystals(CNC), the eco-friendly polysaccharide, in enhancing the creaming stability of Pickering emulsion stabilized by microalgal protein particles.

Spirulina species were used as microalgal proteins and Pickering emulsions were prepared with Spirulina protein particles(SPI, 0.1wt%) and CNC(0~0.5wt%). MCT(medium-chain triglyceride, volume fraction=0.1) oil was used as the dispersed phase. In the images of confocal laser scanning microscopy, SPI which are amphiphilic particles were observed at the oil-water interface, and CNC with hydrophilic side groups was observed in the bulk phase.

As the CNC concentration increased, the stability of the emulsion prepared with SPI was enhanced gradually against creaming. The rheological measurements of the emulsions and the suspensions were carried out and the Pickering emulsions with CNC exhibited yielding behaviors which means that the sample spanning network of CNC was formed. The network of CNC and the localization of particles were observed through cryo-FESEM.

A6. Plant protein integration for food emulsion

Yoann Lefeuvre, Serife Korkmaz, Roland Ramsch, Guillaume Lemahieu, Giovanni Brambilla, Gérard Meunier

Formulaction, France

Plant-based protein use has been expanding over the past years and offers numerous advantages: sustainable origins, vegan alternative, cost effective and health benefits. Proteins obtained from sustainable sources do not have the same performances especially to be used as emulsifier and with the increasing offer, selecting the right protein can represent a bottleneck in new product development.

Removing or partially replacing traditional emulsifiers with plant-based proteins can be very complex: protein selection (including its origin), possible functionalization, optimum concentration... Three parameters must be considered: protein solubility, protein emulsifying and stabilizing properties. Several methods are available to characterize [1] proteins functional properties, but they usually request several instruments, multiple and tedious experiments and sample denaturation.

To help formulator in the transition towards greener formulation, the characterization of the dispersion via Static Multiple Light Scattering (SMLS) have demonstrated to be a valuable ally. SMLS technique detect variation of the backscattering (BS) and transmission (T) in function of the sample height and in function of time which are directly linked to change in particle size variation (aggregation, coalescence, or size reduction process) or a migration phenomenon (sedimentation and creaming). The measurement can be done at rest to study dispersion shelf life and stability or online to determine particle dispersibility, solubilization and emulsification properties. The measurements are done without any dilution and on a wide range of concentration (from 0.0001 to 95% v/v) and particle size from 10nm to 100µm.

Formulaction recently launch the TURBISCAN DNS, combining SMLS technology, measurement under mixing condition for solubilization and emulsification studies and measurement at rest for shelf life studies. In this talk, we will present data and results comparing pea and soybean protein:

- Solubilization kinetic, optimum solubilization time, final solubility and solubility optimization by varying the ionic strength. All the data are acquired while mixing and at high frequency for precise monitoring of the solubility speed and quality.

- Emulsification property via online particle size monitoring and determine the protein efficiency to create a fine emulsion.

- Stability and shelf life measurement : stability study and comparison of the created emulsion to understand the best plant additives to stabilize an emulsion.

[1] McClements et al, Proposed methods for testing and comparing the emulsifying properties of proteins from animal, plant and alternative sources

A7. Interfacial and foaming properties of jackfruit seed protein extract

Xiaoning Zhang, Leonard Sagis

WUR, the Netherlands

Jackfruit is a tropical fruit that originates from the southern part of Asia. The meat of Jackfruit is very popular and found to be a good source material for meat replacers. Recently, due to the demand for greener protein sources, the proteins extracted from jackfruit seeds (a waste product from jackfruit meat processing) have also been explored for use in techno-functional applications.

Studies have shown that unlike other seed proteins, which have both globulins and albumins present in their protein extracts, jackfruit seed protein (JFSP) consists primarily of albumins. In functionality tests JFSP showed good emulsifying/foaming properties. However, the mechanisms behind its stabilizing ability are not well understood.

The aim of this study was to find the reasons behind the good performance of JFSP as a foam stabilizer. A very high dilatational modulus was observed for the air/water interface stabilized by JFSP, with values higher than those observed for whey protein isolate (at the same concentration and strain), which is quite rare for plant protein extracts. Compositional analysis showed that in addition to albumins, saponins are present in the JFSP extract. The interaction between saponins and proteins on the interface were explored using a combination of atomic force microscopy on Langmuir-Blodgett films, and large amplitude oscillatory dilatational rheology. The latter measurements were analyzed using a novel technique, the general stress decomposition, recently developed in our group. The results show that JFSP has excellent potential as a replacer for dairy and meat proteins with respect to foam formation and stabilization.

A8. Structure-function relationships of protein-based foams

Judith Krom, Thomas A. Vilgis

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Protein-based foams are ubiquitous systems with a hierarchical structure. Macroscopically the time-dependent decay of the foam height is a relevant parameter describing foam stability. Meanwhile, on a mesoscopic scale bubble size and lamella thickness are characteristic properties of a foam. On a microscopic scale however, the arrangement of the proteins at the air-water interface is relevant for the foamability of a system. This arrangement of the proteins is caused by the structure of the protein, the arrangement of amino acids and their properties at the protein surface. How stable a foam is macroscopically crucially depends on the ability of the proteins to arrange at the air-water interface and by this to stabilize the air bubble in the liquid. These microscopic mechanisms define how fast bubbles grow and escape, and consequently how fast the foam decays. Furthermore, a higher viscosity of the continuous phase enhances foam stability. Nonetheless, especially for food foams, mostly the macroscopic foam properties are focus of research. Therefore, in the current study we use a model system comprising the structurally well-characterized protein bovine serum albumin (BSA) in water and investigate effects of changes in pH. This way, we relate the known structural properties of BSA to measurements of foam height and image analysis of light microscopy of bubbles and lamellae to determine structure-function relationships. Changes in pH cause a variation of the protein surface charge. We develop a model on how these changes influence the arrangement of the proteins at the interface which effects the bubble size as well as macroscopic foam stability. These fundamental findings can be applied to food systems usually consisting of proteins, fatty acids and polysaccharides, which interact with each other. Thus, such a consideration of the physical properties and interactions on different lengths scales facilitates a deeper understanding of food foams. Applications of these findings include for example the question why cow’s milk naturally shows better foaming properties than common plant-based milk alternatives.

A9. Effect of moderate hyperbaric treatments on lipid crystallization

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The possibility to control lipid crystallization is of great importance to conferring peculiar technological and sensory properties of many foods. Tempering, shear and mixing, seeding and additive application, and, more recently ultrasonication, are traditionally applied to drive and control fat crystallization. Besides these processing interventions, reduced attention has been paid to the possibility of steering lipid crystallization by hyperbaric treatments.

In this work, lipid crystallization under moderate pressure treatments was studied in binary systems containing sunflower oil (SO) and increasing concentrations (70, 80, 90, 100 %, w/w) of palm stearin (PS). To this aim, lipid blends were inserted in plastic pouches, melted at 70 °C for 30 min, and positioned in the chamber of a hyperbaric plant to carry out the crystallization. Samples were allowed to cool and crystallize in the chamber maintained for 24 h at 20 °C and 200 MPa. After the treatment, samples were removed and analyzed after a 24-h period to allow network relaxation. Samples were characterized for mechanical (compressive dynamometry), rheological (oscillatory rheology), thermal (differential scanning calorimetry), microstructural (optical microscopy), and morphological properties (synchrotron X-ray diffractometry). Samples crystallized at atmospheric pressure and room temperature (0.1 MPa) were used as references.

Crystallization under pressure significantly increased the firmness of SO/PS systems. Accordingly, hyperbarically-crystallized samples showed elastic behavior and critical stress significantly higher than that of control samples, indicating an increased capacity to tolerate external mechanical stress. This was attributed to the formation of smaller and homogeneously distributed crystallites with different crystal morphology.
This work clearly shows that the application of moderate pressure treatments could be effective in steering lipid crystallization behavior. The proposed technology could be particularly interesting in the industrial framework, being an energy-efficient strategy.

A10. The Effect of the HLB Value of Sucrose Ester on Physiochemical Properties of Bigel Systems

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Biphasic systems exhibit a wide range of physiochemical and sensory characteristics depending on their composition. The texture of these systems may be altered by structuring the oil or water phases, forming gel filled emulsion, bulk gel emulsion. Recent studies explored the ability to structure both phases, leading to a combined hydrogel-oleogels systems, termed bigels. The current research explored the effect of different sucrose esters (SEs), with different hydrophilic–lipophilic balance (HLB) values, on bigel structure and properties. Bigels consisting of a water phase with glycerol and gelatin and an oil phase with glycerol mono-stearate, lecithin, and SEs with different HLB values were prepared. Rheological and thermal analyses revealed similar gelation-melting transitions governed by glycerol-monostearate crystallization (at ≈55 °C) for all bigel samples. The bigel matrix of the H1 and H2 samples (bigels consisting of SEs with HLBs of 1 and 2, respectively) demonstrated physical gel rheological characteristics of higher elastic and solid-like behavior compared with the H6 sample (bigel consisting SE with HLB 6). A similar trend was observed in the mechanical analysis with respect to hardness, firmness, and spreadability values, which were in the order of H1 > H2 > H6. This behavior was attributed to droplet size observed in the microscopy analysis, revealing significantly smaller droplets in the H1 and H2 samples compared with the H6 sample. These differences in droplet size were attributed to the diffusion kinetics of the low-molecular-weight surfactants. More specifically, the ability of mono-esterified SEs to diffuse faster than fully esterified SEs due to lower molar mass leads to a higher SE content at the oil-in-water (O/W) interface as opposed to the bulk oil phase. The results demonstrate the importance of the interface content in O/W bigel systems, providing an effective way to alter and control the bigel bulk properties.

A11. Rheological, mechanical, and microstructural characterization of oleogels of oil glycerolysis products: the suitability as fat replacers in plant-based meat analogs

Yasamin Soleimanian, Saeed M. Ghazani, Alejandro G. Marangoni

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Glycerolysis products (GPs) are systems formed by the conversion of native triacylglycerols in liquid oils into monoacylglycerols (MAGs) and diacylglycerols (DAGs), mainly studied in the last few years for their unique structural ability. Here we report on the use of specific GPs as the oil phase of ethylcellulose (EC) oleogels. EC oleogels were prepared using a variety of oils including palm olein, tigernut, rice bran, peanut, and cottonseed oil, and EC with different molecular weights (20 cP, and 45 cP) at 5% w/w level. The rheological (small deformation oscillatory rheology) and mechanical properties (texture profile analysis) of the EC oleogels were compared to those of whole pork, beef, and lamb adipose tissue to map and compare thermal softening behavior and absolute mechanical characteristics. Dramatic changes in the crystallization behavior, solid fat content (SFC), and microstructure were found after glycerolysis (GL). GPs presented broader melting profiles with significantly higher melting points, demonstrating the presence of MAG and DAG crystals. The peak melting temperatures of the EC systems were nearly identical to that of GPs, indicating that the interaction between EC and GPs does not promote, nor delay the thermal behavior. SFC increased followed by GL, especially at elevated temperatures, making GPs an ideal candidate for the substitution of plastic fats. The micrographs of samples showed the formation of different crystal networks (platelet-like, needle-like, or spherulite/feathery structures), affected by the different chemical compositions of oil systems and thus different fat crystals which was further confirmed by X-ray diffraction. Strain and frequency sweep measurements showed that GPs and their oleogels behaved as solid-like materials, as elastic moduli (G') were greater than viscous moduli (G'') throughout the linear viscoelastic region (LVR). The addition of EC produced a more deformable and cohesive product (greater LVR and crossover point). In general, EC 45 cP produced stronger oleogels than 20 cP, when comparing their G' values. However, SFC was a dominant contributor to viscoelastic properties. Oil systems containing higher levels of saturated fatty acids such as palm olein-GP oleogel exhibited a more rigid, ordered, brittle structure, whereas tigernut, peanut, and rice bran oil GP oleogels were significantly softer and more deformable. Finally, temperature sweep experiments were conducted to visualize the general softening behavior during cooking. Comparison of the rheological and mechanical properties of the EC oleogels with those of whole pork, beef, and lamb adipose tissue showed the potential for EC oleogels of oil glycerolysis products to be used as adipose tissue mimetics in the new generation plant-based meat analogs.

B Rheology Methods

Monday, 12th of June 2023, Lijsterbeszaal

B1. Structural evolution of pea-derived albumins using SAXS during pH changes and heat treatment

Ruifen Li, Milena Corredig, Jacob Judas Kain Kirkensgaard

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Albumins are small serum proteins soluble in water which are usually found in large amounts in the side stream extracts during plant protein isolation, which have been reported to have better functional properties than their globulin counterparts. In this study, we have investigated the internal structural dynamical behavior of albumins derived from pea protein by the use of small-angle X-ray scattering (SAXS) experiments, combining with size-exclusion chromatography coupled with a multi-angle laser light scattering (MALLS) detector and light scattering. Albumins extracted from pea protein were characterized at different pH (3, 4, 4.5, 7, 7.5, and 8) under heating of 85 °C for 1, 3, and 5 minutes with non-heating conditions as controls. In order to interpret the SAXS scattering of albumins, a model combining a local ellipsoidal shape to describe the individual albumin contour and the power law model to describe larger aggregates of each sample. The results showed that albumins at pH 3 distinguished themselves by showing only the internal ellipsoidal shape and no structural changes during heat treatment. Albumins at pH 7.5 and 8 displayed only the internal ellipsoidal shape before heating, while those at pH 4, 4.5, and 7 already aggregated by fitting both models, and then all these samples formed larger aggregates with increasing power numbers against longer heating time. Furthermore, both the polar and equatorial radius of the internal ellipsoidal shape decreased with increasing heating time, whereas the opposite for those at pH 3 and 4. When combing with data from SEC-MALLS, where 4 peaks were detected, 2 peaks can fit ellipsoid shape with the molecular weight of around 12 kDa to be monomer of native pea albumin 1 (PA1) and that around 24 kDa to be dimers of pea albumin 2 (PA2). The other 2 peaks represent the formation of aggregates, which can be up to 300 kDa. Knowledge of albumins structure during processing is still missing in this area and will be critical in the future both at a fundamental and applied industrial perspective, as we seek to develop more sustainable and functional foods. It will be possible to contribute to more circular, sustainable food chains during protein ingredient manufacturing.

B2. Neutron scattering and neutron spectroscopy for insights into food emulsion interfaces

Theresia Heiden-Hecht, Henrich Frielinghaus and Olaf Holderer

Forschungszentrum Julich, Germany

The stability of food emulsions depends -beside other effects- on a complex interplay between interfacial active components like proteins or phospholipids, oil and water. Preparing milk-based and sustainable plant-based emulsions requires good knowledge in interfacial and emulsion stabilization mechanisms, affected by the emulsion composition. To understand these mechanisms in detail different length scales from interatomic to macroscopic distances need to be investigated.

Neutron scattering techniques provide insight into such emulsions on these length scales depending on the technique used. Combining structural information on molecular length scales from small angle x-ray and neutron scattering (SAXS and SANS) with time dependent neutron spin echo spectroscopy (NSE) allows to expand our understanding towards intermolecular interactions within the interface. These interactions are linked to the emulsion stability – the elastic properties of the protein or protein/phospholipid stabilized oil/water interface on molecular length scales. NSE provides in this combination the time dependent correlation function in reciprocal space, S(q,t), on molecular length scales and time scales in the nanosecond range relevant for thermally driven motion of mesoscopic systems such as the emulsion interfaces.

This presentation introduces the neutron and x-ray scattering techniques which broadens the classical characterization of food emulsions. Results from emulsions stabilized with beta-lactoglobulin as a representative milk protein, and different plant-based proteins, are presented and discussed. Contrast variation by deuteration of some components of the emulsions is applied to focus on the interfacial region, relying on the uniqueness of neutrons.

Connecting these emerging results with classical characterizations such as interfacial tension or viscoelasticity helps understanding the complex mechanisms of interfacial stability and may contribute to a knowledge driven development of sustainable food emulsions.

B3. Rheological fingerprinting of pizza cheese using large deformation oscillatory rheology

Julie Frost Dahl, Sandra Beyer Gregersen, Ulf Andersen, Milena Corredig

Aarhus University, Denmark

Structural heterogeneities and anisotropy in food matrices is critical to the functionality of many food products. In recent years, there has been an increasing interest in characterizing these structures using large amplitude oscillatory shear (LAOS) rheology, to learn how to control their formation through modulating formulations, processing, or storage conditions. This approach provides additional information on the viscoelastic properties occurring to the material subjected to large deformations, in regimes of increased relevance to aspects such as sensory attributes or processing parameters. In this work, pizza cheese, known for the characteristic macroscopic anisotropy of its protein fibers, result of the curd stretching process, was studied with a combination of LAOS, small amplitude oscillatory shear (SAOS), texture profile analysis (TPA), and confocal laser scanning microscopy (CLSM), thereby enabling a close evaluation of the rheological properties at different length scales.

Pizza cheese was tested at four different times of production: during stretching, immediately after stretching and cooling, and after 1 and 2 weeks of cold storage. Samples were tested either transversally or parallel to the anisotropic fiber direction, probing the properties in various directions. LAOS was performed also during melting by heating up to 80°C, measuring the stress response to strain values ranging 1-1000%. A new software - VEOS - (Madsen et al., 2022) based on data visualization theory, was applied to highlight differences in the non-linear rheological dynamics between treatments, and to unlock the potential of LAOS data.

The combination of LAOS, SAOS, TPA and confocal imaging provided complementary information on the process- and storage-induced structural differences between samples at various length scales and while probed in different directions. It was possible to distinguish the properties of the pizza cheese not only as a function of processing stages, but also storage time. The LAOS data was analyzed using the newly developed VEOS software, which allowed for a detailed view of the viscoelastic changes during deformation, and of the responses to the strain as a function of temperature during melting. The unique features of VEOS made it possible to create network maps, grouping Lissajous curves into clusters of similar shapes and thus comparing material properties between samples and during deformation. Using the similarity network map it was possible to capture the compelling differences in the elastic response of the material by comparing the overall rheological LAOS properties at different conditions. Comparing the mechanical responses derived from TPA, SAOS and LAOS, clearly demonstrated the importance of probing these complex anisotropic samples at different length scales and direction of shear, to better understand their rheological properties at various deformation conditions.

B4. Quantifying Extensional Texture in Viscoelastic Cheese Melts using Composite Harmonic Exponential Waveforms (CHEW)

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Textural properties of viscoelastic liquid foods have been described in the past using conventional rheological techniques such as large-amplitude oscillatory shear (LAOS). For example, Faber et al. (2017) used LAOS on Gouda cheeses to quantify textural attributes such as firmness, rubberiness, springiness, and fluidization, as functions of rheological material parameters. Building upon this idea of quantification of these (consumer-relevant) higher-order mechanical properties of liquid and soft-solid foodstuffs, we investigate extensional attributes of these materials, embodied in terms such as 'stringiness' and 'extensibility'. We observe that there are non-trivial correlations between perceptive extensile properties ('stringiness') and rheomechanical properties of these materials induced by nonlinear strains. For example, inspired by an early textural state space map of dairy products (Davis, 1933) we show that in a crossplot of the median loss modulus vs. storage modulus measured during an LAOS amplitude sweep (similar in spirit to the "Scott-Blair" square in Wagner et al., 2016), there appears to be robust clustering of specific cheese-types commonly considered exceptionally stringy/extensible.

Motivated by this observed connection between shear and extensional properties, we have developed a new rheometric protocol based on "Composite Harmonic Exponential Waveforms" (CHEW). Fundamentally, this new "CHEW" technique allows us to measure transient extensional properties of semi-solid foods with a conventional shear rheometer. CHEW is based on the idea that imposing complex strain histories on a material microstructure can result in a specific sequence of stress states that accurately emulates entirely different modalities of loading. Specifically, it was suggested by Doshi & Dealy (1987), and later demonstrated by Kwan et al. (2001), that an exponential shear strain will move the microstructure of a polymeric liquid through a similar sequence of stress states as a planar extensional flow. Fundamentally, this is enabled by the mechanical coupling between the normal and shear stresses that develop in complex fluids particularly at large strains (Lodge-Meissner Relation). Here we develop a periodic version of this concept, to probe dynamic extensional properties of complex fluids, using a shear rheometer. The strain history is given by: $\gamma(t) = A \sinh(\alpha \sin(\omega t))$, where A controls the maximum amplitude of the imposed strain and is normalized such that $A = \gamma_{max} / \sinh(\alpha)$, ω is the frequency of the cyclic deformation and the flow type parameter α effectively tunes the signal between a weak sinusoidal ($\alpha << 1$) deformation (e.g. SAOS with $\gamma_{max} << 1$, or LAOS for $\gamma_{max} > 1$) and a strong ($\alpha >> 1$) deformation with exponential character. We demonstrate this method on a viscous fluid with no elasticity, on a viscoelastic fluid (3% wt PIB solution) and on several different cheese melts. Implementing CHEW on a torsional rheometer, we compute an appropriate periodic extensional viscosity function ($\eta_{ext} = \Delta\sigma / (\alpha\omega)$) from the time-evolving principal normal stress difference, and compare it to a traditional transient extensional viscosity (Padmanabhan,1995). Finally, to study long-term material evolution (arising for example from microstructural degradation), we have constructed a nonlinear theoretical model to interpret the complex time-dependent material response arising from CHEW, and discuss how the extracted model parameters correlate with perceptive properties (e.g. 'stringiness', and sample degradation/evolution).

B5. In-line rheometry and spectroscopy for controlled tailoring of textural and nutritional characteristics of HMEC-processed plant protein-based meat alternatives

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High moisture extrusion cooking (HMEC) is a technology to create fibrous structures from plant proteins resembling meat texture. Derived meat analogues provide similar textural mouthfeel to meat but are an alternative with only a fraction of the massive environmental impact of meat. During high moisture extrusion cooking plant protein containing materials are typically hydrated, sheared, heated (up to 170°C) and subsequently cooled and shaped in a long cooling die to avoid flash evaporation at the die exit. During such thermal and mechanical processing, proteins go through structural changes and reassemble in a shear and elongational flow-induced structure, stabilized by crosslink formation. To adjust textural and nutritional characteristics in a customized consumer-relevant range, in-line measuring methodologies for (a) the viscoelastic rheological protein melt characterisation and (b) the product structure and related texture development, were designed and successfully adapted in extruder and cooling die sections of the HMEC process. The latter was run in traditional and novel micro-foaming modes in order to generate meat analogue products of controlled structure porosity and correlated texture and sensorially perceived tenderness.

For the first time in-line measured viscoelasticity characteristics like (a) the First and Second Normal Stress Differences (N1, N2) and (b) the shear viscosity h as a function of shear rate, temperature and water content were made in-line accessible and correlated with in-line measured spectroscopic (RAMAN, NIR) data as well as with mechanical characteristics (Young's Modulus, tensile- and cutting strength) of the resulting product structure.

Key technological aspects that unlock the next level of production autonomy were addressed by coupling a multilayer advanced control structure with the before-mentioned in-line techniques. The interplay of various parameters was considered by applying a model-based predictive approach that anticipates process future changes and derives optimal set-points. Extrusion results proved potential advantages and practical implementation of the approach.

B6. 3D printing of food for dysphagia - shaping and functionality

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The utilization of 3D printing within the food industry has gained attention in the recent years. In our research we use the 3D printing process together with 3D scanning to shape texturized food for dysphagia patients. Dysphagia patients have difficulties in swallowing and are therefore in need of pureed and cohesive foods. Due to the difficulty of eating and loss of appetite in this patient group frailty is a wide problem which comes at a high cost for both the patient and society. To increase the visual likeness of the pureed food to the original items we have coupled 3D printing with 3D scanning to get digital copies of the foods which we then have 3D printed. Starch is often used as a texturizing agent in this type of food in this work we have partially replaced it with a dietery fiber, citrus fiber, allowing for an up-concentration of the concentration with 5% whilst retaining a similar rheological profile. This comes with the added value of increased dietary fiber intake in a group which due to their intake of texturized food do not naturally have a diet where it exists. The aim is that an increased visual recognition of the food which is eaten together with a higher intake of fiber can in turn lead to an increased appetite and reduced frailty. This work is a proof of concept showing the use of 3D-printing and 3D scanning to realize structures of a chicken leg, using rheology to gain knowledge of the formulation in relation to the 3D printing process.

Ahlinder et al 2023, Towards attractive texture modified foods with increased fiber content for dysphagia via 3D printing and 3D scanning, Frontiers in Food Science and Technology DOI=10.3389/frfst.2022.1058641

B7. Evaluation of the ability of microscopic image analyses and low-frequency time-domain NMR to predict and assess stirred commercial yogurts quality.

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The rate of new product development for stirred yogurt in Canada is fast. Therefore, yogurt producers are seeking solutions to accelerate development. One of their challenges is to assess stirred yogurt quality quickly and efficiently.

The present study aims to evaluate the potential use of microscopic image analyses and low frequency nuclear magnetic resonance on proton (1H-LF-NMR), as new quality assessment tools for stirred yogurt.

Stirred yogurt producers in the province of Quebec provided three different production batches of eight different commercial stirred yogurts that varied in fat, protein contents, and the presence of stabilizers. The usual methods used for quality assessment in industry [viscometry, texturometry (firmness), and centrifugation (syneresis)] were measured. Yogurts were also characterized using pH, serum viscosity, microstructural features (laser diffraction, digital microscopy), and water mobility (low-frequency nuclear magnetic resonance on proton, 1H-LF-NMR).

Digital microscopy and the granulometric method of reference (laser diffraction) correlated well together ($0.60 < R < 0.95$). Moreover, those methods presented moderate to strong correlations ($0.50 < R < 0.70$) with stirred yogurt physical properties, especially when only yogurts with stabilizers were used to calculate the correlations ($R > 0.70$). The influence of water entrapment into yogurts' physical properties was highlighted by significant correlations between water mobility and nearly every stirred yogurt physical property. Predictive models of the stirred yogurt physical properties were built from the different yogurt characteristics and succeeded to explain induced syneresis and firmness at 70 to 90 % depending on whether the models were built from all yogurts data or data from yogurt categorized on the presence or not of stabilizers. Based on this categorization, microstructural features and pH alone could explain at least 70% stirred yogurt physical properties variability. However, without yogurt categorization, pH, serum viscosity, and water mobility were also needed to reach a minimum of 70% of variability explained. In conclusion, stirred yogurt microstructure and water mobility explain and potentially predict commercial stirred yogurt physical properties. While 1H-LF-NMR is complex, the digital microscope image analyses can help producers during routine quality analyses or product development. If only pH and image analyses are available, models or criterions need to be adapted by stirred yogurt categories, for instance with or without stabilizers. In this work, stirred yogurt properties were used as quality indicators, but similar work could be done including sensorial attributes (eg. creaminess).

B8. Particle Size Analysis of Milk – A case of mistaken identity?

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Milk is a suspension of fat globules and casein micelles in a solution of whey protein and lactose. Characterisation of milk in industry is most commonly via particle size distribution methods such as laser scattering techniques or optical light microscopy. Results from such methods show that milk exhibits an overall bimodal distribution, whereby it is generally accepted that the larger population is fat globules (0.2-2 um diameter) and the smaller population is casein micelles (0.1-0.2 um diameter).

Our recent work has shown that Differential Dynamic Microscopy (DDM) is very good at sizing bimodal suspensions using model systems. Therefore, we have used DDM to analyse milk as a more complex system and compared our results with conventional laser scattering techniques in the literature. Our DDM results agree with conventional laser scattering techniques but, both techniques do not provide the full picture and information on individual particles is masked.

By use of high pressure freezing in combination with cryo FIB SEM, we have revealed and imaged the microstructure of milk in situ (i.e. with no ice crystals formed from freezing). Our results suggest that the fat globules, which are darker in contrast compare to casein, are in fact, in both the large and small populations. This is further confirmed by separation of the fat droplets and casein micelles by ultra-centrifugation. Therefore, we conclude that use of correlative techniques is pivotal to achieve clarity and full characterisation of complex materials.

B9. 3D structure evolution during bread baking in a combined microwave-convective oven revealed by in-situ synchrotron-based X-ray microtomography

Niklas Lorén, Florian Schott, Sven Isaksson, Emanuel Larsson, Federica Marone, Camilla Öhgren, Magnus Röding, Stephen Hall, Rajmund Mokso, Birgitta Wäppling Raaholt

RISE, Sweden

The food industry has an interest in better understanding the impact of baking processes on the final bread quality for better use of resources such as time, energy, and raw materials. The energy consumption and the time needed to bake a bread is dependent on the baking technology. Bread-baking is a complex process, due to the interaction between heat induced chemical processes and structural mechanisms, such as formation of crumb and crust, expansion of pores, inactivation of yeast, and flavor development. The effect of baking technology and the formulation on the time-dependent 3D structure evolution is not well known because in-situ time-resolved bread structure analysis is challenging since baking occurs inside an oven and in the bread bulk.

In this talk, the development of a new concept for characterizing in real-time the evolution of the three-dimensional pore and lamella structure of bread during baking using synchrotron X-ray microtomography (SRμCT) will be presented. A commercial, combined microwave-convective oven has been modified and installed at the TOMCAT synchrotron tomography beamline at the Swiss Light Source, to capture the 3D dough-to-bread structural development in-situ at the micrometer scale over time. This concept allows quantitative comparison between for instance the influence of baking technologies, different dough types and positions inside the bread. Here, baking has been performed with convective heating, microwave heating, or a combination of convective and microwave heating. The effect of three different dough types and three different positions was also evaluated, but the emphasis in the work was put on the effect of baking technologies. Time series of tomographic images were recorded with an acquisition time of 0.4 s. Automatic image processing and image analysis have been performed to extract porosity, pore volume, elongation, local wall thickness and pore coordination number as a function of time.

Results on the effect of the baking technology on the 3D bread structure evolution will be presented. As expected, a large impact of the baking technology on the 3D bread structure evolution and the final bread was found. The results showed that in general the porosity, mean pore volume and mean coordination number increase with time, while the mean local cell wall thickness decreases. The influence of heat propagation into the bread due to the different baking technologies was linked to the microstructure development. During baking, strong correlations between the mean pore volume and porosity, and between the mean local wall thickness and the mean coordination number, with no strong influence of the baking technique, nor the flour type, neither the position inside the bread, were established. This work shows one important example of the usage of syn8chrotron x-ray imaging techniques in food science. The technique opens new opportunities to improve our understanding of the mechanisms governing baking and of the process parameters that control the final bread quality.

B10. Confocal Raman Microscopy of Low Moisture Food Products: Breakthroughs and Challenges

Mark Auty, Taranvir Bedi, James Spinks, Gleb Yakubov, Michael George

Mondelez, UK

Confocal imaging, in particular confocal scanning laser microscopy (CSLM), is a well-established technique for characterizing the microstructure of food systems which is key to understanding their rheological and textural properties. A major disadvantage of CSLM is that fluorescence labels are needed, and these dyes are generally confined to fat and protein localisation so lack specificity. In addition, the labelling procedure can disrupt the microstructure, particularly for low moisture foods such as confectionary or baked products. Consequently, there is a need to map specific food components at the micron scale. The development of confocal Raman microscopy (CRM) is the ideal analytical tool and enables direct chemical mapping of complex food matrices in 2D or 3D at high spatial resolution, close to the diffraction limit (~300nm). Raman spectroscopy is highly specific but is a relatively weak inelastic scattering phenomenon and can often be overwhelmed by intrinsic sample fluorescence. Raman microscopy has successfully been used for dairy products but low moisture snack foods such as confectionary or baked snacks present a major challenge. Many of these products contain fluorescent ingredients such as cocoa solids or Maillard browning compounds that significantly reduce the Raman signal-to-noise. In this presentation we will highlight the major challenges and mitigation strategies for successfully mapping food ingredients with CRM using biscuits and chocolate as examples. The application of CRM for mapping amorphous sugars in baked snacks will also be presented.

B11. How potato starch structural transitions impact microstructure development during deep-frying

Isabella M. Riley, Ujjwal Verma, Nand Ooms, Mieke A. Nivelle, Pieter Verboven, Bart Nicolai, Jan A. Delcour

KU Leuven, Belgium

Deep-fat frying is a complex process of heat and mass transfer that leads to the distinctive palatability of a wide variety of deep-fried food products. Starch, an important constituent in several deep-fried foods, may undergo various physical transformations (e.g. starch gelatinization, glass transition) during deep-frying when sufficiently hydrated. While the functional roles of these transformations have been well-researched in several food processing unit operations, it is not yet well understood how the changing physical state of starch contributes to the microstructural changes throughout the deep-frying process.

In this work, time domain proton nuclear magnetic resonance (TD 1H NMR) techniques [offline and novel online (at 180°C) methods] were used to assess the phase transitions and structural changes of fried (0 -180 s) potato starch-water model systems (30-60% moisture content). TD 1H NMR is a powerful tool to examine the physical state of starch based on the evaluation of proton distributions and spin-spin relaxation times (T2), which reflect the different microenvironments present in a sample and molecular mobility therein. The use of both online and offline NMR techniques allowed the structural changes of starch occurring during frying and post-frying (i.e. after cooling), respectively, to be decoupled. These starch transformations were linked to the microstructural changes, assessed using X-ray micro-computed tomography (μCT) analysis.

Online TD 1H NMR showed the swelling and gelatinization of starch to occur within the first moments (10 s) of its immersion in oil, during which starch was present in the rubbery state. When in this state, the flexibility of starch chains to resist expansion limited permanent changes to the microstructure. As the frying time lengthened, both online and offline TD 1H NMR techniques showed the portion of rigid protons to progressively increase and the T2 relaxation time to decrease, reflecting the progressive transition of starch from a rubbery to a glassy state. This transition made the matrix more rigid and thus, prone to undergo permanent changes to its structure. Due to the rapid evaporation of water and consequent disruption of the solid matrix, X-ray μCT analysis showed pores to become more abundant and increase in size. Fried samples with a high starting water content exhibited a high total porosity (ca. 80%) with a high quantity of air-filled pores, thin cell walls, and oil concentrated in the outer layers of the structure. In contrast, fried samples of low starting water content displayed a lower total porosity (ca. 55%) with more oil-filled pores and higher oil absorption, and with oil penetrating to both the outer and inner regions of the structure.

The findings of this work provide a basis for understanding how the physical states of starch contribute to microstructure deformation and transport phenomena of oil during deep-frying.

C1. White Asparagus bud proteins, from waste to interface stabilizer in food foams.

Anteun de Groot, Jack Yang, Leonard M.C. Sagis

WUR, the Netherlands

White asparagus buds are a large waste stream that, despite having a high nutritional quality, is currently underutilized. In this study, we explored the potential of mildly processed white asparagus bud protein extracts for food application with respect to interface and foam stabilization. Waste stream utilization is often based on extensive processing, which can hamper protein functionality, and uses copious amounts of resources. In this study, screw pressing followed by mild extraction was used to obtain protein extracts. Asparagus protein stabilized air-water interfaces were studied by profile analysis tensiometry, which revealed formation of a stiff interfacial network. Formation of the interfacial network was affected by the presence of insoluble particles (>1 μm) and small molecular impurities. The non-linear responses to oscillatory dilatational deformations were studied with Lissajous plots and detailed analysis of the higher harmonics. Analysis of the interfacial microstructure with atomic force microscopy on Langmuir-Blodgett films revealed the formation of highly heterogeneous interfaces. The combination of dilatational surface rheology and microstructure imaging indicated that asparagus proteins formed disordered viscoelastic solid-like interfaces. The asparagus proteins showed high foamability and stability, which could be related to the air bubble size. The removal of small molecule impurities and insoluble particles was found to improve the foaming properties. This study identified critical factors for interface and foam stabilizing properties of asparagus proteins and revealed the great potential of mildly processed asparagus bud extracts for application in food foams.

C2. Exploring Milk Fat / Water interface colonisation and organisation: How Surface Tension Measurement can reveal hidden insights

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The dairy industry commonly uses high-pressure homogenisation to achieve a homogeneous and stable dispersion of fat in formulated milk products. This process involves splitting milk fat globules into smaller units and increasing the surface area of the interface between the lipidic and aqueous phases. As a result, the surface-active molecules naturally present in milk such as phospholipids, whey proteins, and caseins, adsorb on the interface. The change of fat globule size and interface composition can in turn modify the textural and sensory properties of the dairy products in which it is dispersed. Despite the widespread use of this technique, the colonisation process of oil/water interfaces during homogenisation remains a complex and poorly understood phenomenon.

The aim of this research is to investigate the underlying colonisation mechanisms of these molecules in order to unlock knowledge of this sub-step of milk homogenisation. To do so, modelling is combined with a surface tension measurement dual approach. Those experiments are carried out in order to simulate the adsorption of the various surfactants (proteins and phospholipids, bulk concentration varying from 10-7 to 10-2 mol/m3) on a static single fat droplet (purified anhydrous milk fat). From the surface tension measurements, through modelling by the Ward-Torday approach, the diffusion and adsorption characteristics (saturation surface excess, critical concentration at which half of the surface is covered by the surfactant, diffusivity) were determined assuming the Langmuir model for adsorption isotherm. Modelling can be extended to predict not only the phenomena of individual adsorption of surface-active molecules but also interactions between surfactants.

Nevertheless, monitoring the interface colonisation by combining surface tension measurements and modelling highlights colonisation is not only diffusivity driven. According to the literature, many reorganisation hypotheses leading to changes of the surface area covered by these molecules are expressed, including multilayer organisations and orientation of molecules. A study of the dilatational interfacial rheology was carried out to investigate the mechanistic lack. The dynamic study of the surface tension was carried out using the sinusoidal deformation technique during the whole surface tension decrease, from the initial diffusion-colonisation of the interface phase to the reorganisation phase. Finally, the interfacial properties were studied in the non-linear viscoelastic regime by large amplitude oscillatory surface rheology (Lissajous plot) to highlight specific behaviour. The measurements and the modelling approach consider scenarios of increasing complexity, from the effect of a single surfactant, to the simultaneous adsorption of multiple surfactants and the sequential adsorption of surfactants. It provided insights into the complex mechanisms involved in the colonisation of milk fat globules during homogenisation.

C3. The impact of disulfide bonds of whey protein β-lactoglobulin on the structural conformation and adsorption behavior at the oil/water interface

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During the stabilization of food emulsion, amphiphilic proteins, like the whey protein β-lactoglobulin (bLG), adsorb at the oil-water interface in three phases: (I) migration through the bulk phase, (II) adsorption at the oil-water interface and (III) interfacial rearrangement with structural change followed by the formation of a protein film. For all these, relevant structural features of bLG range from secondary to quaternary structure, especially disulfide bonds. Disulfide bonds are built between two cysteine residues of the amino acid chain. bLG possesses five native cysteine residues that have a strong influence on the conformation of the tertiary structure. One free cysteine is available and two intramolecular disulfide bonds exist, one located in the inner core molecule and one at the surface of the molecule.

In this context, the use of recombinantly produced bLG, whose cysteine residues can be selectively exchanged by alanine residues, can help to better understand the role of protein structure in stabilizing oil-water interfaces. The aim of this work was to investigate the effects of disulfide bonds in the protein structure on the three phases (I -III) at the oil-water interface. For this purpose, structural variants of bLG were recombinantly produced in Escherichia coli as previously described in [1,2]. One or more of the five cysteines present in the native bLG were selectively substituted.

The behavior of the structural variants of bLG during phases (I) and (II) was investigated by interfacial tension measurements and during phase (III) by measurements of the response of the protein film to dilatational forces (both with drop tensiometer). Along with the experimental data, molecular dynamic (MD) simulations were used to have insight into the molecular and structural level of the adsorption of the bLG variants.
In general, the substitution of cysteines had a significant effect on the monomer/dimer ratio of bLG and, by that, affected the adsorption kinetics. The native quaternary structure of bLG is mainly a dimer at neutral pH, while the deletion of the disulfide bond near the molecular surface resulted in a shift toward a monomeric state [2]. This further resulted in a faster migration through the bulk (I) and faster adsorption (II) with predominantly elastic properties of the protein film (III). In contrast, the deletion of the intermolecular disulfide bond caused a higher elastic modulus E' (deformation amplitude 0.7%), which means the interfacial protein film was more elastic. When substituting all cysteines, the bLG resulted in a disordered and unfolded secondary structure, which led to stronger adsorption to the interface, which both was confirmed by MD simulations (stage (III)).

This research is the first step toward a fundamental understanding of protein structure during the interfacial stabilization mechanism at the oil-water interface, which is necessary to characterize and control the emulsification process.

C4. Cruciferin versus napin – air-water interface and foam stabilizing properties of rapeseed storage proteins

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Rapeseed protein extract has high nutritional value and good techno-functionalities, e.g. in foam systems. Yet, its exact interface and foam stabilizing mechanisms are not well understood. Rapeseed proteins comprise mainly of cruciferin and napin. Our aim was to systematically investigate the interface stabilization behaviors of cruciferin, napin and combinations of both at air-water interfaces. We used surface (dilatational and shear) rheology and microstructure imaging (AFM) and linked their behavior to their foaming properties. We observed that napin adsorbed faster at the air-water interface than cruciferin due to its smaller size, leading to 90% higher foam overrun than cruciferin (320%). The interfaces showed distinct differences in structure and mechanical properties, as cruciferin formed stiff solid-like interfaces with Ed' = 72.5 mN/m and Gi' = 9.0·10-3 Pa·m, leading to high foam stability (half-life time 220 min). Napin formed weaker less stretchable interfaces (Ed' = 61.8 mN/m; Gi' =6.7·10-3 Pa·m), leading to substantially lower foam stability (half-life time 23 min). Cruciferin and napin were also mixed at 3:1, 1:1 and 1:3 (w/w) ratios. Napin increased the foamability of all mixtures with foam overrun between 400-420%. The mixture at 3:1 cruciferin-to-napin ratio had comparable foam stability with pure cruciferin since cruciferin dominated the mechanical properties of the air-water interface. Higher napin contents largely decreased foam stability with half-life time decreasing to 80 min. These findings provide a comprehensive understanding of the behaviors of rapeseed proteins at air-water interfaces and their link to foaming properties, which can be used to tailor the properties of aerated products stabilized by rapeseed proteins.

C5. (Non)linear interfacial rheology of protein-phospholipid stabilized oil-water interfaces: Role of the molecular structure of phospholipids on the interfacial viscoelasticity

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Within the production and/or the further processing of oil-water emulsions, the interface of oil droplets is mechanically and thermally stressed, which can lead to droplet aggregation and coalescence. The choice of emulsifier is crucial as it influences the interfacial viscoelasticity (viscous and elastic portions) and thus, the process stability of an emulsion. Proteins such as β-lactoglobulin (β -LG) form strong and highly viscoelastic networks on the interface, resulting in a high stability during processing. In literature, it is often assumed that low molecular weight emulsifiers (LME) such as phospholipids (PL) form weak and highly viscous interfaces which are unstable towards stress. Two phenomena were previously shown in food systems where proteins and PL are present simultaneously: (1) The replacement of proteins by phospholipids, leading to a weak LME-dominated interface, thus a low process stability; (2) Interactions between proteins and LME, leading to an increased interfacial viscoelasticity. The molecular structure (head group, fatty acid chain) of PL seems to impact the interfacial composition and the resulting interfacial properties, which is not yet fully understood. Additionally, the emulsion undergoes temperature cycles during processing which can affect interfacial properties due to the melting/crystallisation of the emulsifier/dispersed phase. This temperature impact has often been neglected in the literature.

The aim of this study was to analyse the effects of the molecular structure of PL on the interaction with β-LG at the oil-water interface. PL with varying head groups (choline, ethanolamine) and fatty acid chains (C18:0, C18:1) were used. The interfacial properties were investigated within and outside of the linear viscoelastic regime by means of dilatational measurements and interfacial shear rheological measurements. A temperature sweep has been used to consider melting/crystallisation phenomena and their effect on interfacial viscoelasticity.

In presence of the β-LG, an increase in the storage modulus was measured in case of the C18:0 PL, while the interface behaved predominated viscous in case of the C18:1 PL (high loss modulus). For the C18:0 PL with ethanolamine as a head group, the increase in storage modulus was the highest. The fatty acid chain determined whether interactions between PL and β-LG take place, while the head group determined the strength of interactions. Currently, we are investigating to what extent the fatty acid composition of the disperse phase has an influence on the interfacial viscosity.

D Tribology

Tuesday, 13th of June 2023, Lijsterbeszaal

D1. Tailoring the mouthfeel of dairy-based beverages with polysaccharides: bridging tribology with sensory perception

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Dairy-based beverages contain many functional ingredients, such as protein, fat, and polysaccharides, which affect their sensorial attributes and palatability. Polysaccharides play a significant role in determining the sensorial properties of foods, which is partly related to the lubrication they provide. To identify the most relevant structural features related to the lubrication ability of polysaccharide solutions, we first investigated the lubrication performance of polysaccharides with different characteristics (i.e. molecular weight, conformation, stiffness, and charge density). Rigid rod-like polysaccharides (xanthan gum) showed a higher capacity in reducing friction coefficient and better resistance to changes in pH and ionic strength compared to semi-flexible (pectin and carboxymethyl cellulose) and flexible polysaccharides (guar gum). In combination with protein particles or oil droplets, different polysaccharides also gave a different microstructure, which had a large effect on the frictional properties of these mixtures. For these model systems of dairy-based beverages systems, we also varied the emulsifier of the oil droplets. In the presence of polysaccharides, systems containing soy lecithin-stabilized droplets showed a high degree of aggregation and coalescence, whereas systems containing whey protein-stabilized droplets were less sensitive to such phenomena. Aggregation was especially observed for mixtures containing xanthan, whereas a more homogeneous structure was mainly observed for mixtures with guar gum. Systems with high level of aggregation showed higher friction coefficients and tribological curve with multiple regimes. Various parameters in the different sliding speed regimes were correlated with the sensory perception of these model systems. Systems with a relatively low degree of shear-thinning gave higher values for the attributes creamy, thick, and fatty. This was mostly observed for homogeneous structures. The attributes slippery, dry, and mouthcoating were strongly correlated with the slope of frictional curves in different regimes. This presentation will provide an overview of how structure, the tribological profile and sensory perception are related.

D2. Oleosomes: natural oil droplets for dairy alternatives studied by tribology

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Oleosomes (a.k.a. lipid droplets or oil bodies) are omnipresent natural oil droplets in plant seeds. In this work, we extracted these oleosomes from rapeseeds and studied their lubricating behaviour by tribology. Proper lubrication (i.e. friction reduction) during oral processing is crucial in the sensory perception of food products, especially dairy alternatives. We have studied the friction-reducing properties of oleosomes by using an Anton Paar Tribocell setup. In addition, we created full-fat and skimmed dairy alternative model systems by creating oleosome-whey protein and oleosome-pea protein mixtures. The tribological properties of these systems were evaluated and compared to pure pea protein emulsion droplets. The aim of this work is to show the potential of oleosomes as a substitute for milk fat globules in dairy alternatives.

We discovered that oleosomes can reduce friction by a mechanism called ball bearing (i.e. rolling). Oleosomes seem to remain largely intact after the analysis (shown with CLSM), which can be related to their special membrane containing proteins and phospholipids. In a mixture with proteins (at neutral pH), the proteins can dominate the lubricating properties in a skimmed dairy alternative system, while the oleosomes dominate the full-fat system. At an acidic pH of 4, the oleosomes possess a great feature, as the droplets remain single droplets, while pea protein emulsions flocculate. This gives oleosomes significantly better lubricating properties.

In summary, oleosomes have excellent lubricating properties and possess the potential to substitute fat globules and even plant protein-stabilised oil droplets in dairy alternatives.

D3. Predicting oily mouthcoating of pure vegetable oils using tribology

Maria Tecuanhuey, Alicia Girardi, Mark Ambühl, Marine Devezeaux de Lavergne

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Friction of oils is often measured in food systems but seldom on pure oils. To maintain the organoleptic qualities of fat systems, with a focus on oily mouthcoating, we propose a tribological approach to predict oily perception in pure oils and fat blends. Five pure vegetable oils and four oil blends were studied: canola oil, high oleic sunflower oil (HOSO), coconut oil, shea stearin, cocoa butter, and blends at 70/30 of HOSO/shea, canola/coconut; canola/cocoa, and canola/shea. The oils were chosen to have samples with a variety of fatty acid and triglyceride composition. Friction and high shear ($\eta\infty$ = 1000 1/s) viscosity were measured at 30°C and 60°C. The tribological tests were carried out using a ball-on-three-pin test configuration, speed was increased logarithmically from 1x10-8 to 0.2 m/s. Polarized static microscopy at 30°C and 60°C was done using an objective lens of 20x magnification. Solid Fat Content (SFC) was analysed with low-field NMR. All the oils and blends were heated at 60°C and evaluated by an untrained sensory panel (N=10) for several attributes including "oily mouthcoating" perception. Tribology revealed that friction of all oils and blends is lower at 30°C than at 60°C. For all oils and blends, except shea stearin, no difference in friction was observed anymore when the tribology data was scaled with the high shear rate viscosity, in the mixed regime. Static microscopy revealed that few small crystals were present at 30°C only for cocoa butter, canola/shea and HOSO/shea. For shea stearin, the difference in friction at 30°C and 60°C could not be explained by the viscosity, and static microscopy revealed a large extent of crystallization at 30°C. Therefore, we hypothesize that the lower friction at 30°C for shea stearin is due to a ball-bearing lubrication mechanism. These results would suggest that SFC, and therefore the presence of crystals, can decrease friction of edible oils. An inverse correlation was found between SFC at 30°C and friction at 1x10-2 m/s at 30°C (-0.89 Pearson correlation). Friction at 1x10-2 m/s and 30°C and viscosity at 30°C correlated with "oily mouthcoating" perception (-0.82 and -0.80 Pearson correlation, respectively), suggesting that friction is the best parameter to predict fat related perception of simple oil systems.

These results confirm that friction is a good predictor of "oily mouthcoating" perception of pure oil systems. Moreover, viscosity and crystallization can modulate friction and therefore guide the development of oil/fat containing food products with optimal sensory characteristics.

D4. New tools for accessing powder rheology of food systems

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Many food products are either in the form of powder or are a powder during a processing stage. The flow behaviour of powder and granular media is governed by a variety of adhesive and repulsive forces, which depend on internal and external influences. Therefore, measurements of powder flow are crucial for a better understanding of the processes involved. Powders can be subjected to very different loading condition, from high loads such as in silos, bins, and hoppers, to low loading conditions and fluidized powders in pipe flow or blending. Different measurement techniques are used depending on the loading condition and cohesion level of the powder. A powder flow cell works for powders with low cohesion strength under low loading and in aerated or fluidized conditions and determines the flow behavior, while a powder shear cell is used on cohesive powders under high to low loading and measures the yield limit of a consolidated bulk material.

Both a powder flow cell and a shear cell are designed for use in a standard rheometer. The flow cell consists of a glass cup in which a concentric bob or a stirrer is connected to the rheometer motor coupling. In a standard procedure the cohesion strength of a powder or the resistance of a powder to a flow can be determined. A sufficiently large air flow through the powder removes the powder memory and generates a gas fluidized bed. After the air flow is stopped, a constant rotational speed is applied and the torque is measured. Once a steady flow is reached, the torque value is converted into a stress representing the cohesion strength. The shear cell is used to characterize consolidated powders in a temperature and humidity-controlled environment. Examples measurements on food powders demonstrate the versatility and usefulness of these techniques.

The shear cell can also be used to study the stick-slip behavior in consolidated powders through a new mode called slip-stick. In this mode, the lower motor rotates at a given speed and the upper motor mimics a mechanical spring, i.e., a recoil torque is applied that is proportional to the deflection angle. When the contact “sticks” the friction deflect the upper part of the contact until the force exceeds a certain value and it “slips” back to its initial position, then the process begins again. Since the spring is not mechanical, the spring constant can be easily changed by software, making the setup a versatile stick-slip tester. Many powders exhibit stick-slip characteristic when sheared. While this is a friction or tribology effect, the causes of stick-slip in powders are at the particle level.

D5. Impact of emulsifying salt and intact casein levels on wear behavior, microstructure, and shredding performance of process cheese

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Material loss during slicing/shredding a block of cheese is a common industrial problem which can occur due to sticking of cheese to the moving parts of machinery or crumbling during high-speed operations. Wear testing measures penetration depth and mass loss during rotational shear motion. As per our hypothesis, extent of mass removal obtained from wear testing of cheese should give an indication of shredding performance. We used processed cheese (PC) as a model system to investigate impact of the PC formulation on wear, structural, and shredding properties. PC samples in 18 batches were prepared using full factorial design varying trisodium citrate (TSC) levels (2-3%) and weighted average ages of natural cheese (1-101.8 days) indicating different amount of intact casein levels. Microstructure of the PC samples was characterized using confocal laser scanning microscopy (CLSM) and transmission electron microscopy (TEM). Wear measurements were performed at 5°C using a pin-in-disk attachment to MCR 302 Rheometer, using 1N tribological force and 50mm/s sliding speed. Shredding performance was evaluated at 5°C as work needed to grate and crumbliness. CLSM and TEM micrographs of PC indicated that size of fat globules decreased with increasing both age of natural cheese and TSC concentration. Wear parameters. penetration depth and mass loss decreased linearly ($p<0.05$) as intact casein levels increased. Cheeses with lower amount of the intact casein exhibited lower ($p<0.05$) values of hardness and gumminess, suggesting softer PC matrix. With age of natural cheese, work needed to grate a block of PC increased, and crumbliness decreased, indicating loss of shredding property. Effect of TSC on these properties was not significant, as solid fat at 5°C, largely dominated the material characteristics. The findings of this study provide useful information to cheese manufacturers for optimizing formulation and processing condition to attain desirable shredding property with minimal material loss.

E1. Start-up shear of natural near-critical gels made of gluten proteins

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Gluten is a complex protein network crucial for the breadmaking quality, and widely used in the food industry as a texturing agent. It forms a natural transient network that include physical bonds (hydrogen bonds) and dynamic covalent bonds (disulfide bonds). The study of various gluten protein dispersions previously showed that samples can be regarded as polymeric near critical gels characterized by rheological parameters, elastic plateau, and characteristic relaxation time, which are related to one another, as a consequence of self-similarity, and span several orders of magnitude when changing the parameters such as protein concentration1, ageing time1 and solvent quality2. For its part, the protein composition controls both the distance to the gel point3 and the critical exponent of the critical state.

In order to investigate the non-linear visco-elastic properties of gluten, a series of pre-gel samples satisfying the time-curing superposition over 5 decades of time in the linear regime, were investigated by successive start-up shear and relaxations at different shear rates. Master curves of maximum stress and strain values as function of a shifted shear rate could be plotted and a transient characteristic time of samples, different from the characteristic time defined in the linear regime, could be defined. In addition, samples totally relaxed the stress generated by very large shear strain in a relaxation time independent of the initial gelation state of samples, while the initial linear viscoelastic properties are recovered after the non-linear protocol. The molecular mechanisms at the origin of this singular behaviours will be discussed in light of our knowledge of the gluten microstructure.

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E2. Probing carrageenan gel syneresis: from macroscopic to microscopic scale

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Syneresis is a process where gels expulse a fraction of the water they contain, leading to their shrinkage. The so-induced changes in gel dimensions and structure are problematic for applications. Despite syneresis being a common feature of many gels, experiments probing its microscopic mechanisms are still scarce.[1]

We have developed three space-resolved dynamic light scattering (DLS) tools to examine the behaviour of carrageenan hydrogels during syneresis: (i) a set-up coupled to a white light source allowing us to simultaneously perform DLS experiments and directly visualise syneresis during the gelling of a millimetre-size drop, (ii) a set-up probing a full vertical slice of a sample contained in a vial, and (iii) a set-up probing a smaller volume of the sample but with a finer space-resolution than (ii). We have used the above set-ups to investigate the behaviour of carrageenan gels prepared at different concentrations and in vials where gel adhesion to the walls was either promoted or supressed. With set-up (i), we showed that, in boundary-free conditions, syneresis occurs in a spatially and temporarily heterogeneous way, i.e. starting from one region of the gel bead and spreading throughout it. With set-ups (ii) and (iii), we showed that, in the case where wall adhesion is suppressed, syneresis occurs freely. DLS measurements showed fastening of the polymer gel network microscopic dynamics which we could assign to macroscopic events such as the onset of gel motion due to water expulsion from either the bottom or top of the sample. Furthermore, DLS revealed two relaxation times – a fast and a slower one, that could be attributed to the radial and the vertical strains, respectively, induced by gel shrinking. Last but not least, network relaxation dynamics were fairly homogeneous across the sample.

By contrast, when gel adhesion to the walls was promoted, syneresis was significantly hindered and limited to the exposed surface at the very top of the sample. No speeding-up of the polymer gel network dynamics was observed, consistent with the absence of major macroscopic event and with the behaviour of syneresis-free hydrogels. The relaxation dynamics in the gels were significantly slower than those measured in the samples able to undergo syneresis and they were more spatially heterogeneous, with fast dynamics observed only at the top, where syneresis occurs.

To our knowledge, it is the first time that such a clear relationship between microscopic dynamics and macroscopic events at work during syneresis could be established when boundary conditions are properly controlled. The use of multiple set-ups also allowed us to interpret the relaxation phenomena probed by DLS.

[1]. S. Mizrahi, Syneresis in food gels and its implications for food quality, In Chemical Deterioration and Physical Instability of Food and Beverages, Woodhead Publishing, 2010, pp. 324-348.

E3. Protein-based gels produced by fermentation of faba bean and pea

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Plant protein-based gels can have an important structuring role in the development of products such as high protein dairy alternatives, however their production and properties under different conditions is still largely unknown. In this work, the effect of initial pH (i.e., pH prior to fermentation), inoculum dose, protein concentration, and pre-heat treatment on the gelling potential and properties of the resulting fermentation-induced gels prepared from faba bean protein isolate (FPI) and pea protein isolate (PPI) emulsion were studied. Two FPIs were used, having 77% and 84% protein content, while PPI had 84% protein content. Emulsions were prepared with a final protein content of 10% and 10% rapeseed oil. Results indicated the initial pH of the material as determinant factor in gel formation. The 77% protein FPI had low initial pH (≈5.2) and was not able to form a gel at any of the investigated conditions unless the pH was adjusted to >7 prior to fermentation. The effect of protein pre-heating on gel properties was less pronounced. A general trend to increase elasticity in small deformation experiments (rheometer) was observed, however the effect on hardness in large deformation experiments (texture) was marginal. Using double the inoculum dose (from 0.2% to 0.4%) also had marginal effect on the gel structure. In general, FPI appeared to produce harder (by ≈25%) gels on all investigated conditions. The effect of storage on the rheological properties of the gels was also studied and appeared to depend on the final pH reached after fermentation, although more work is required to verify the results. In the 77% protein FPI, where the initial pH was adjusted to ≈7.2 and final pH was high (≈5.5), storage appeared to reduce the storage and loss moduli of the gel. On all other occasions, where the final pH was ≈4.3, storage marginally affected the rheological properties of the gel. Overall, this work shows the potential of faba bean and pea proteins to form fermentation-induced gels with possible applications in the development of dairy alternatives.

E4. Gelation of protein from mealworm (Tenebrio molitor): A study on structural changes and rheological properties

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The farming of insects only requires a fraction of land and water compared to the production of other animal derived proteins. This makes insects – such as mealworm – a very promising and sustainable source of alternative protein for food and other applications. With regard to these applications, it is important to understand the techno-functional properties (e.g. gelation) of insect protein in general and mealworm protein in specific. Moreover, since other constituents such as e.g. (divalent) cations may influence the structure and rheological properties of corresponding gels, their influence should also be considered. However, despite the potential of insect protein as sustainable protein source, research on the gelation of mealworm protein is just developing and so far, only a limited number of publications exist, especially, when it comes to the influence of (divalent) cations. With the presented study we contribute to the general understanding of the structure formation and gel properties in heat-induced gelation of mealworm protein. Additionally, we investigate, how these properties are influenced by the addition of a divalent cation ($ZnSO_4$).

Our methodical approach focussed on:

- applying rheological time sweeps to describe the gelation kinetics
- elucidating the involved types of interactions via gel solubility experiments
- approaching the microstructure of the gels from a microscopic, as well as rheological perspective
- using FT-IR measurements to consider changes in the structure of the protein

Results showed gel formation in all examined samples. Combining results from gel solubility experiments and gelation kinetics we conclude, that hydrophobic interactions and hydrogen bonds are the dominant types of interaction in all samples. Investigation of concentration dependent rheology revealed the overall network structures of samples without $ZnSO_4$ to be in the transition regime where both inter- and intrafloc links contribute to the gel's elasticity. The addition of $ZnSO_4$ influenced the structure of the protein, both before and after heating, and led to additional stabilisation of the gels via electrostatic and/or covalent interactions. Moreover, the corresponding gels showed distinctly different rheological properties from the gels without $ZnSO_4$ (i.e. higher storage modulus, shorter linear viscoelastic regime, an increasing contribution of intrafloc links and more pronounced intracycle strain stiffening at deformations outside the linear viscoelastic regime). This could be related to a less homogenous and more particulate gel structure as confirmed via scan electron microscopy. Based on the obtained results, strategies for the customisation of gel properties and gel composition may be derived, that can advance the potential for future utilisation of mealworm protein as a sustainable protein source in food and other applications.

E5. Impact of adjunct strains in co-culture with a commercial starter on rheological properties of stirred yogurt

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Exopolysaccharides (EPS) produced naturally by lactic acid bacteria (LAB) have often been suggested as natural stabilizers in yogurts. The type of EPS produced during the fermentation is known to modulate rheological properties of yogurts. The aim of this work was to identify promising adjunct cultures for EPS production such as Lactobacillus, Lacticaseibacillus, Levilactobacillus, Lactiplantibacillus, Streptoccocus, and Leuconostoc to be used in co-culture with a commercial starter to improve rheological properties of stirred yogurts.

Eight EPS-producing strains from a collection of 537 LAB were selected based on genomic characteristics, ropiness properties (filament stretchability) and known EPS structural features from the literature (linkages, branching and charges). Stirred yogurts were prepared at a laboratory scale by using double jacketed stainless steel fermentation vats. The system was coupled with a pumping, shearing and smoothing system to simulate industrial productions. The fermentation was performed at 43°C during 5.5 h ± 30 min, until pH reached 4.6 by using a commercial starter and an adjunct inoculation rate of 107 CFU/mL-1 in reconstituted skim milk (3.8% proteins, 4% sucrose). At the end of the fermentation, the yogurt was smoothed at 20°C and stored at 4°C. Rheological properties of stirred yogurt were measured after one and seven days of storage at 4°C with a rheometer (AR-G2) using a cup and bob geometry. The flow curves were obtained by recording viscosity and shear stress with increasing shear rate linearly from 0.1 to 250 s-1 and decreasing to 0.1 s-1. Apparent viscosity at 250 s-1 and consistency index were calculated by using the power law modelization. The thixotropy was evaluated by calculating the hysteresis loop area between the upward and the downward curve.

Rheological results have shown that Lacticaseibacillus paracasei LMA-1793 induced higher values for thixotropy, viscosity and consistency index: 24%, 15% and 15% of increase, respectively, compared to the control without adjunct. The EPS concentration in yogurt produced with this adjunct was up to 159 mg/kg.

The positive effect of L. paracasei LMA-1793 as an adjunct on rheological properties of yogurt may be related to EPS structural features. Two different EPS were isolated and characterized by gel-permeation chromatography after fermentation in acidified milk at 43°C. Further work is ongoing to investigate the structural characteristics of the EPS produced by this strain as molecular weight, monomer composition, charge and linkage.

E6. Effect of pH and addition of calcium on the textural properties and temperature sensitivity of heated casein gels

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Changes in calcium content in cheese milk regulate the structure of casein micelles, affecting the mechanical and functional properties of the final product. How calcium content affects the texture and temperature sensitivity of heated rennet-induced casein gels (model systems for cheeses like Halloumi) is still unclear. Therefore, the aim of this study was to investigate the effect of removal and addition of calcium to the textural properties of such gels. To obtain calcium-depleted gels, skimmed milk was acidified to pH values ranging from 6.5 to 5.9, corresponding to calcium contents in the gel from 3.2% to 2.4%. To obtain calcium-supplemented gels, calcium chloride was added to milk (pH adjusted to 6.3) at concentrations from 1.5 to 12 mmol/L, corresponding to calcium contents in the final gels ranging from 3.0% to 4.2%. Composition, rheological properties at small deformations, and texture profile analysis of the obtained samples were investigated. Heating the casein gels increased their dry matter due to syneresis. With decreasing pH, a higher dry matter and a more fused casein network were observed, corresponding to a higher storage modulus and a lower critical strain at small deformation, and a higher hardness and resilience at large deformation. Also calcium addition enhanced syneresis, and thus led to an increase in dry matter. Although calcium addition had a limited effect on the structure of the gels, changes in the properties similar to those of the pH series were observed. Based on these results, we concluded that the mechanical properties of the gels were mainly determined by the dry matter content, and were not much affected by calcium content.

For heated casein gels, also the temperature sensitivity is important, and this property was strongly correlated with calcium content. Temperature sensitivity was characterised based on the ratio between storage modulus at 20 and 90 °C and tan δ. For gels with reduced pH (i.e., reduced calcium content in the casein micelles), a higher temperature sensitivity was observed, whereas in the case of calcium addition, temperature sensitivity decreased. Our results provide insights to alter the structure of heated casein gels and temperature sensitivity upon baking or frying of dairy products like Halloumi cheese.

E7. Double gels made of interpenetrating colloidal networks

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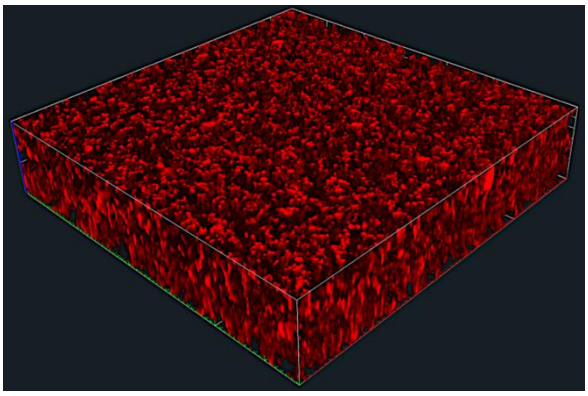
The advancement of new methods for assembling soft matter results in the creation of fascinating structures with valuable material properties. In this study, we present the development of double gels networks composed of interpenetrating colloidal networks made of cellulose microfibrils and protein. Our findings show that the mechanical properties of these double gels vary based on the composition of the colloidal species, resulting in stiffer double gels with similar elasticity compared to single gel counterparts. Further analysis of the gels' microstructure suggest that the peculiar mechanics of the double gels is a result of mutual steric hindrance and restricted particle mobility due to interpenetration of gel networks.



3D real space imaging of an apolar colloidal silica gel

E8. Effect of Ca2+ and pH on thermal gelation behavior of soluble pea protein

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The aggregation and gelation behavior of plant based proteins is important to understand for many food applications, including e.g. meat analogs (Mistry et al., 2022) or using various functionalities of protein microparticles (Schwestka & Stoger, 2021). Ca2+ is often added to food products as a nutritional fortification as well as to control their structure. However, knowledge on Ca2+ induced gelation properties of soluble pea protein is still limited. It is also known that the addition of Ca2+ results in a decrease in pH. In this paper, thermal gels preceded by Ca2+-induced aggregation, or preceded by pH change, were compared. The particle size distribution, ζ-potential, rheological properties and microstructural characteristics were analysed before and after heating, either following a step of Ca2+ addition or a step of pH change. It was found that the addition of Ca2+ would increase the particle diameter of the pea protein dispersion and thereby increase the gel strength within the range of 0-20 mM concentration. Excessive Ca2+ addition (50-100 mM), however, led to stronger aggregation of pea globulins before any thermal treatment applied, which was detrimental to and the thermal gelation behaviour. Similar to the effect of calcium ions, a decrease in pH below 5.9 prior to thermal treatment decreased the strength of the subsequently formed thermal gels. It was also observed that the gels preceded by pH-induced aggregation exhibited higher viscoelasticity compared to the gels preceded by Ca2+-induced aggregation. These results provide useful information for the design and preparation of pea protein gels for food applications.

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E9. Phase behaviour of ternary mixtures

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Phase separation in biopolymer mixtures is a common phenomenon in foods. In recent papers, we analysed the Edmond-Ogston virial model for binary mixtures. A number of novel analytical results combined with some simple numerical procedures to predict the binary phase diagrams were presented [1,2]. The binary phase diagram can be predicted using the three virial coefficients as an input, and these three virial coefficients can be extracted from a set of experimentally determined critical points of mixtures with shared components [3].

Recently, the model was extended to describe phase separation in ternary mixtures (three biopolymers in a common solvent). The ternary phase diagrams, characterised by six virial coefficients, and show a more complex phase behaviour compared to binary mixtures. In this contribution, we present new results on ternary mixtures, and their relation to earlier work:

- a necessary condition for the virial coefficients for the occurrence of phase separation in two or three phases
- an analysis of the different regions of (local) thermodynamic instability using the Descartes sign rule
- an expression for the critical curves
- a relation between the slopes in points along the critical curve
- the relation between the concentration of components in the different phases according to the so-called Lambert-W function

These results lead to a deeper insight in the phase behaviour of ternary mixtures and are expected to serve as a steppingstone towards modelling phase separation in polydisperse and multi-component mixtures.

[1] Dewi et al, Food Hydrocolloids 101, 105546 (2020)
[2] Bot et al, ACS Omega 6, 7862-7878 (2021); ibid 6, 20086-20087 (2021)
[3] Bot et al, Food Hydrocolloids 126, 107473 (2022)

E10. Manipulating the mechanical and rheological properties of ethylcellulose oleogels: the role of chemical structure of small molecular weight surfactants

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Oleogels have raised great interest among food scientists as a promising approach for developing functional fat mimetics and alternatives to animal-derived lipids with enhanced nutritional benefits. Ethylcellulose (EC) is unique amongst oleogelators, as it is the only directly-dispersible polymeric gelling agent yet identified. However, these gels generally display limited plasticity due to poor solvent/polymer interactions. This work seeks to expand the scope of EC as a functional oleogelator by characterizing the impact of incorporating various amphiphilic small molecule surfactants. Canola oil-based oleogels were prepared with 8 wt% EC (45cP) and a 2:1 EC/surfactant ratio. Seven surfactants were evaluated to contrast differences in the chemical structure (i.e., both tail and head groups). Of these, monoacylglycerol (MAG), stearic acid (StAc), sodium stearoyl lactylate (SSL), and citric acid esters of monoglycerides (CIRTEM) all enhanced the mechanical properties of the EC oleogels to varying degrees, with 18-, 17-, 11-, and 11-fold increase in storage moduli (G'), respectively. MAG and StAc also significantly decreased the gelation/melting point of EC. In addition, CITREM and SSL produced the most dramatic increase in strain stiffening in the large amplitude oscillatory shear response, while in the thixotropic tests, CITREM showed the highest G' recovery (18.8%) immediately after the shear strain was reduced from 100% to 0.1%, which was ~6 times greater than that of the surfactant-free control (2.6%). In contrast, MAG and StAc decreased thixotropic shear recovery to ~0.15%, indicating the formation of more brittle gels. Diacetyl tartaric acid esters of monoglycerides (DATEM) was the only surfactant investigated which diminished gel strength, but it did not significantly impact the rheological behavior. Crystals with varying morphology were observed in micrographs of oleogels containing MAG, StAc, and SSL, suggesting the substantial impact of these molecules on EC oleogels may be attributed to the formation of a crystalline network, and may have further contributed to strengthening the gels. In comparison, DATEM produced large spherulite-like crystals of varying size (up to ~30 μm) randomly scattered throughout the gel, but no space-filling crystalline network was observed. The formation of these crystals could be attributed to surfactant self-assembly in the oil phase, which may inhibit the formation of the EC polymer network and further decrease the mechanical properties of these gels. In contrast, no crystals were observed with the addition of CIRTEM, but small spheroid-like structures (~5 μm) were formed uniformly throughout the gels and cooperatively interacted with the EC polymers. Compared to MAG, the CIRTEM headgroup contains carboxylic acid functional groups, which potentially interacts with EC polymers more strongly, and consequently affect the polymer network in a positive way. These results suggest the mechanism of gel formation is different for EC-CIRTEM and EC-MAG/-StAc gels. The precise molecular mechanisms by which these surfactants interact with the EC network and their roles in changing gel properties are continuing to be investigated and will be discussed in relation to developing functional polymer-based oleogel as fat mimetics.

E11. Effect of processing conditions on structure and sorption capability of whey protein aerogels

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Aerogels are nanostructured materials with low density, high surface area (>150 m2/g) and open porosity (95-99.99%), typically obtained by solvent removal from gels while preserving network structure. Protein hydrogels are optimal sources for the preparation of food-grade aerogels with a high capacity for entrapping nutrients and/or bioactive molecules within their porous structure. Although the research on food applications of aerogels is still pioneering, this evidence supports the promising role of protein aerogels as functional ingredients. The functionality of aerogels is strictly dependent on their structural features, which are driven by the preparation technology.

In this study, the effect of the preparation procedure on the structural properties and sorption capability of whey protein (WP) aerogels was studied. To this aim, WP hydrogels (20% w/w, pH 5.7) were subjected to three different procedures: i) freeze-drying (FD) to obtain FD aerogel monoliths; ii) supercritical-CO2-drying (SCD) to obtain SCD aerogel monoliths; iii) grinding, water-to-ethanol substitution and supercritical-CO2-drying to obtain SCD aerogel particles. The physical properties (density, mechanical properties, isotherm, bulk and BET porosity, SEM microstructure) of the aerogels were studied. Results indicate that, depending on processing parameters, aerogels with different porosity were obtained. FD aerogel monoliths presented pores with 2-5 μm diameter. By contrast, SCD monoliths and particles presented a denser network, with pores <1 μm in diameter. In all cases, the aerogels showed glass transition at 161±4 °C and maintained their original porosity at 20 °C at an equilibrium relative humidity <80%.

Based on their high porosity, the obtained aerogels easily absorbed both oil and water by capillary forces. Nevertheless, the kinetics of oil and water uptake was slower in the SCD aerogel monoliths than in the FD ones. While water absorption caused aerogel destructuring, the oil-loaded aerogels retained their integrity, generating materials containing up to 80% (w/w) oil and high physical stability (96% oil holding capacity). When oil was loaded into the SDC aerogel particles, an oleogel with rheological properties analogous to those of traditional hard fats was obtained. These peculiar mechanical properties were attributed to the formation of weak hydrophilic interactions among aerogel particles in an oil environment so that oil was not only absorbed into the particles but also retained in the spaces among them.

This study demonstrates that the choice of preparation technology can steer aerogel performances in view of multiple uses. If a fast dissolution of the protein aerogel in water is the target, FD aerogels should be selected. Conversely, when oil should be retained within the aerogel for in-food and in-vivo delivery, the use of SCD protein aerogels, in the form of both monoliths and particles could be an option. Finally, if fat replacement is the issue, plastic oleogels could be obtained by using SCD aerogel particles.

E12. The dual functionality of di-acylglycerides in lipid systems

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Oleogels, semi-solid gels made of vegetable oil and structuring agent, were proposed as healthier alternatives to conventional fats due to their high unsaturated fatty acid content, providing an improved nutritional profile while maintaining similar physical and sensory attributes as fats. Low-molecular-weight oil gelators are commonly used in oleogels due to their ability to mimic the texture and functionality of solid fats while maintaining a healthier nutritional profile. While extensive research on mono-acylglycerides and tri-acylglycerides as single oil gelator or in combination with other gelators is available, only little information can be found in the literature on di-acylglycerides (DAG) and its oil structuring performance.

The current research aims to investigate the dual functionality of DAG as oil structuring agent and as interfacial stabilizer of water-in-oleogel systems. For that, di-stearin (DS) oleogels and water-in-oleogel systems were prepared using various DS concentrations and water/oleogel ratios. DS formed stable oleogels with a minimum concentration of 10 %wt. and stable emulsion gels with a maximum water content of 30 %wt. DS-based emulsion gels with 30 %wt. water showed a significantly higher gel strength compared with oleogels with the same DS concentration. Interestingly, oleogels demonstrated behavior similar to ideally elastic materials characterized by a zero slope in a frequency sweep test, while emulsion gels exhibited a frequency dependent behavior with a slightly positive slope. Both oleogels and the corresponding emulsion gels exhibited typical sol-gel transitions characterized by a cross-over between the storage and loss modulus and thermo-reversible behavior. DS crystallization in oil revealed the formation of large spherulite crystals, whereas water addition and homogenization reduced the crystal size. Moreover, aggregated crystals were found around the wtare/oil interface, implying on the involvement of DS crystals in the interfacial stabilization of the biphasic system. Overall, saturated DAGs can be used as oil structuring agent as well as surface active agent in emulsion gel systems. Such ability can be exploited while developing new food products based on biphasic systems like margarine and mayonnaise.

F1. Effect of fibre orientation on the shear stress and normal stress responses of meat (analogue)

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In recent years, the demand for sustainable food options has greatly increased. In this regard, alternatives to meat products have gained much attention due to the environmental impact of animal agriculture, resulting in a wide range of commercially available meat analogues. However, the development of meat analogues that mimic real whole cuts is still scarce, with few studies assessing the rheological response of whole cut meat analogues, and the influence of their fibre orientations. In this study we aimed to provide new insights on the role of fibres on mechanical response of meat and meat analogues. We compared the rheological response of meat cuts (beef, chicken) with meat analogues (made with soy protein or pea protein in combination with wheat gluten using shear and heat-induced structuring1), both in the linear viscoelastic regime using small amplitude oscillatory shear (SAOS), and in large deformations using large amplitude oscillatory shear (LAOS). Furthermore, we also studied the normal force response during shear, which is studied in other fields such as material sciences, but largely ignored in foods.

Our results show that fibre orientations parallel to the shear direction result in lower shear moduli compared to perpendicular orientations in small deformations. At large deformations, the meat analogues showed a higher dissipation ratio than meat cuts, while real meat exhibited large intracycle strain stiffening. From the oscillatory normal force response, a Poynting modulus was calculated, showing larger values in perpendicular orientations than parallel to the shear direction in meat cuts, while no differences were found for meat analogues, confirming the presence of weaker fibres in the meat analogues. We showed that studying the normal stress response, in addition to shear stress response provides a better mechanical characterization of the meat and meat analogue structures. These results provide a deeper understanding of structure in both meat and meat analogues during large deformations, which are applicable to large and complex deformations such as mastication, and determine the textural traits which are desirable to be found in future meat analogues formulations.

F2. Combined FEM modeling and experimental characterization of die melt flow for prediction of phase separation and associated fibre formation during extrusion of high moisture meat analogues

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High Moisture Meat Analogues (HMMAs) are today commercially produced from soy, pea protein and wheat gluten protein, utilizing an extruder to form a protein melt at high moisture content, high temperature and high pressure with subsequent active cooling on exit Kyriakopoulou et al. (2021). Under suitable conditions this process render a fibrous structure similar to that of meat. An extruded fibrous structure is not unique for these proteins, but has also been observed and utilized in plastics production to improve material strength Ardakani et al. (2013). In both cases it is known how to produce the fibres, but not the mechanism governing their formation. Consequently, it is currently difficult to use other proteins and to utilize the full potential of this technique.

In addition to experimental extrusion trials with focus on characterization of the intrinsic properties of ingredients and their impact on microstructure, a proper physical understanding of the associated fibre formation in a given protein melt is necessary to identify a robust and efficient production window with reliable operating conditions. The situation calls for a simulation model of the extruder die, where the fibre formation process can be analyzed based on both wall cooling conditions and rheological properties, to avoid e.g. core-slip and non-fibrous regions.

We have developed a FEM model to predict fibre formation in the extruder die based on the phase separation approach developed by Murillo et al. (2019) with extended rheological modeling. The model couples the temperature, Navier Stokes and Cahn Hilliard equations with rheological assumptions and the solution is then obtained by combining both stationary and time dependent solvers in COMSOL Multiphysics. Flow properties are extracted from quantitative rheological measurements and are combined with qualitative information of the extrudate flow, whereas the more inaccessible Cahn Hilliard parameters and associated assumptions remain more speculative. We investigate and predict the impact of rheology, flow characteristics and Cahn Hilliard parameters on fibre formation for a realistic extruder die geometry.

As fibre formation is a complicated process and likely consisting of several phenomena, the main goal with the present work is to identify the strengths, but also limitations with the present model and thus give guidance for future work.

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F3. Towards better understanding of structure formation in extrusion

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Meat consumption is linked to environmental, ethical and health issues. The production of meat analogues is one of the possible approaches to facilitate consumers to switch from animal-based to plant-based products. Extrusion is a commonly used method to produce meat analogues with a fibrous structure that is comparable to meat. However, knowledge about the effect of different processing conditions and protein ingredients is rather empirical still. Our research is therefore aimed at gaining a more fundamental understanding of fibrous structure formation during extrusion. A first step to achieve this goal is finding the link between structure formation under well-defined flow, which is used in the shear cell, and under less defined flow conditions in the extruder. This has for example been done by investigating the effect of shear application during cooling in the shear cell to gain more insight in the processes taking place in the cooling die of the extruder. Additionally, the effect of mixing intensity before processing material in the shear cell has been investigated. In addition to the use of the shear cell, this project also explores the use of near-infrared spectroscopy to measure the composition and processing history of extrudates.

F4. Uni-directional freezing as food structuring tool for meat analogues

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Plant-based substitutes for meat and fish are in demand by many consumers who want to make food choices that lower their environmental footprint and limit animal suffering. The majority of plant-based meat analogue products are comminuted meats like burgers and sausages, or come in a form of strips or battered nuggets. These processed meats, however, are not the largest meat segment, as 75% of the meat market are whole-cuts. Producing an appealing whole-cut meat using plant-based ingredients is challenging. In part, this is due to the limited production methods available to make plant-based protein fibers. These methods often involve high temperatures and / or shear, which essentially pre-cookes the product, making it difficult to impart a raw appearance.

A potential alternative method is unidirectional freezing. This process removes heat from one side of a colloidal suspension or gel in a controllable way. Suspended particles or polymers are excluded from the moving ice front and begin to concentrate around the ice crystals. Typically, ice is then removed from the material by sublimation to obtain a scaffold with aligned pores. In the current work, uni-directional freezing is used to generate aligned, elongated pores in an agar-based scaffold. In the next step, a protein solution is allowed to diffuse into the pores. The resulting structure is a highly anisotropic structure, which generates protein fibers of several cm’s. By varying the cooling rate, and shape of the hydrogel, the diameter and length of the protein fibers can be controlled and be used to modify the textural properties of the final product. Unidirectional freezing is a flexible process in terms of ingredient choice, processing parameters as well as the shape of the products it generates, making it a suitable technique for developing whole-cut meat alternatives.

F5. The interplay between soy proteins and dietary fibre in determining structure formation of plant-based meat analogues produced with high moisture extrusion

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Plant-based meat analogues have been receiving increasing attention. Soybeans are a highly relevant raw material for producing such products because of their high protein content (40% of dry matter [dm]), well-balanced amino acid composition, and high dietary fibre (DF) content (15-20% of dm). An important quality attribute of meat analogues is their meat-like fibrous structure, which can be created through a process called high moisture extrusion (HME). In systems containing both proteins and DF such as soy protein concentrates, these constituents are believed to form separated phases due to their thermodynamical incompatibility, which in turn may contribute to the formation of such fibrous structures during HME. Still, the (joint) contribution of soy proteins and DFs in determining structure formation remains unclear. To fill this knowledge gap, freeze-dried products originating from locally grown soy, varying in protein and DF content, including a protein-rich fraction (soy protein isolate [SPI], 92% protein and almost no DF), a DF-rich fraction (DFF, 35% protein and 59% DF) and several SPI-DFF blends (46 to 81% protein and 48 to 15% DF) were prepared. Their bulk rheology (at 25 and 95 °C) and water distribution (determined by proton nuclear magnetic resonance [1H-NMR], at 25 °C) when hydrated to a moisture level of 60% v/w were studied. These raw materials were subjected to HME at the same moisture level and the structure and texture of the obtained extrudates was determined. Finally, a correlation analysis was applied to examine the relations between the rheological properties, the water distribution in the hydrated samples and the structural characteristics of the extrudates.

Increasing the DF proportion in the hydrated SPI-DFF blends led to significantly ($P < 0.05$) lower phase angles and a linear increase of the complex viscosity, both at 25 and 95 °C, indicating that soy DF caused the formation of a more elastic structure with higher resistance to deformation. Also, the mobility of weakly bound water protons decreased when the DF content increased, indicating that the soy DF-water interactions are stronger than soy protein-water interactions, which may lead to a different water distribution in hydrated SPI-DFF blends. This matched with the rheological observations. Extrudates produced from SPI-DFF blends containing >69% protein and <26% DF comprised layered structures, whereas those produced from samples with 61% protein and 33% DF contained fine and evenly distributed fibrous structures. No distinct fibres were observed in extrudates prepared from DFF. Textural analysis showed that the cutting strength of extrudates was higher for samples containing proportionally more protein. Interestingly, clear negative correlations ($R2= 0.952$ at 25 °C and 0.923 at 95 °C) were observed between the cutting strengths of the extrudates on the one hand and the complex viscosities of the hydrated SPI-DFF blends on the other hand. In conclusion, this research for the first time shows that the soy protein-DF ratio has a strong impact on the rheological properties and water distribution of soy protein-rich products, and is an important parameter that directs the structure of meat analogues produced via HME.

F6. Influence of oils on plant-based meat analogues: Assessing extrudate's mechanical properties to ensure quality and consumer acceptance

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Whilst the global demand for plant-based meat alternatives continues to grow rapidly, many consumers still lack diversity in marketed products, especially in terms of meat types and variety of protein sources. To meet consumers' demands, new products need to be developed that mimic taste, aroma and texture of meat as authentically as possible. However, various consumer surveys reveal that available products often do not meet consumers' expectations regarding several factors incl. texture, juiciness, and mouthfeel.

An important product feature for achieving a similar product to meat is the fibrous, anisotropic structure, which resembles the product texture of muscle meat. Additionally, to improve the texture and taste of extrudates, oils and fats are usually added to the formulation. Whilst increasing the juiciness perception of the extrudates, oil and fats can also disrupt the fibre formation and act as plasticizers. The degree of interference depends on the interactions between oil/fat and proteins and the proteins emulsifying properties. However, mechanisms involved in structure formation, oil stabilization, and interaction amongst ingredients are not fully understood and individual for every protein-oil combination. For this reason, this contribution addresses the application of a mechanical, rheological and tribological characterization as solution approach to gain insights into formulation and process parameters, facilitate product innovations and assure an objective evaluation of the resulting product.

The effect of oil on the textural properties of the meat substitutes in terms of customer acceptance were investigated by different techniques. Results show, how parameters such as oil concentration, protein source, and extruder die temperature define the fibre formation. The influence of these parameters on product structure were evaluated using electron microscopy and image analysis. To assess mouthfeel, rheological measurements and texture analysis were performed and compared to meat. This was evaluated using an electron microscope and image analysis. For the assessment of mouthfeel, rheological and tribological measurements and texture analysis tests were performed using a Thermo Scientific HAAKE Rheometer. Results show how these measured data can be related to product structure and used as quantitative measure to compare meat analogs' product properties.

F7. Legume-based meats with tuneable textures obtained using fermentation techniques

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A broad range of plant-based meats (chicken, beef, pork, fish etc) have been developed in recent years and are currently widely available. However, a consumer demand is arising for plant-based products with minimal processing and limited additives. This offers many scientific challenges and opportunities for innovation. As such, we explore the potential of legume fermentation as food materials that have consumer-acceptable flavour and textural attributes. Specifically, we aim to understand the physics and chemistry behind the ability of various binders to enhance the textural properties of fermented legumes. We carry out solid-state fermentation fava beans using the Rhizopus Oligosporus fungus. The resulting fermented legumes are combined with various binders including methylcellulose, gluten, egg whites and a commercially available vegan egg replacer in different ratios and moulded into a patty. Using this legume/binder matrix we explore the molecular properties of the binders contribute to the microscopic as well as the macroscopic structure of the fermented legumes. We present the results of our physical/chemical measurements (microscopy, texture profiling analysis (TPA), composition analysis, rheology etc.) as well as a sensory evaluation conducted by a trained sensory panel. The reference sample with no added binder results in a moist patty with limited cohesion, density, and firmness. The firmness and density become highest upon the addition of methylcellulose. This is in line with microscopy images which show a dense network when methylcellulose is added compared to the reference sample, as well as TPA results which show a ten-fold increase in the hardness. However, the moistness strongly decreases with the addition of methylcellulose compared to the reference sample according to the sensory panel. In contrast, both vegan egg replacer as well as gluten increase the moistness and improve the cohesiveness of the reference sample. The addition of animal-based egg does not appear to cause changes in texture compared to the reference sample. Furthermore, we show that combining binders in various ratios results in patties with higher moistness, cohesiveness, density as well as firmness than the reference samples and a higher degree of moistness and our instrumental measurements are in good agreement with our sensory results. Legume fermentation thus results in high-protein food products with limited processing and additives. A wide range of tunable textures making fermented legumes suitable candidates for a new generation of plant-based meats.

F8. Rheological assessment and design of protein-rich inks for the 3D printing of meat analogues

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Over the last years, research on plant-based protein sources for 3D food printing has emerged. As protein-rich inks for extrusion-based 3D food printing do not exhibit favourable printing properties of their own, hydrocolloids are often added. However, this complicates the scene of understanding the rheological properties of non-native printable inks. Previous studies either did not (i) investigate rheology, (ii) extract meaningful data, (iii) consider tuning the ink's rheology, or (iv) relate rheology to the 3D printing process. For that reason, this work focuses on increasing the knowledge of the rheological behaviour of plant-based protein-rich inks on all four fronts. Different protein sources are studied, of which soy and gluten. Not only is the effect of sample preparation for rheological testing investigated, but also different encountered problems during testing are discussed. This allows the establishment of efficient procedures and testing methods, which are shown in this study. Mainly step-change responses and flow curves are assessed, resulting in meaningful data regarding shear thinning, viscoelastic and thixotropic behaviours. Through the analysis of multiple hydrocolloidal ink formulations (including thickening and gelling agents), the first steps are taken to determine the relation between ink composition and rheological properties. Based on findings in the literature, the specific shear rates during 3D printing are determined and the corresponding viscosity is assessed. Therefore, a first relation between ink rheology and final shape quality of the 3D printed food is established, opening the path for a model-based design strategy for 3D food printing. Overall, this work provides the foundation (i) to customize the rheological properties of protein-rich inks for 3D printing applications and (ii) to obtain relevant rheological data. In future works, the relation between rheology and 3D printing can be deepened further, which will result in the possibility to design inks with tailored rheological properties and desirable print performance. These findings can help alternative meat production via extrusion-based 3D printing.

F9. Do's and don'ts in tensile testing of meat analogues

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Tensile testing is a widely utilized technique to obtain information on the mechanical properties of meat analogues. However, the potential of tensile tests for meat analogues is currently not fully realized. To date, parameters such as fracture stress, fracture strain, Young's Modulus and anisotropy index have been used to describe mechanical properties of meat analogues. By drawing inspiration from other disciplines outside of food science, we can learn to use tensile tests in a more advanced manner. In this presentation, we will describe how tensile testing can be employed to gain a comprehensive understanding of how to enhance meat analogue structures.

The fibrous structure in meat analogues is a result of the elongation of a phase-separated system. In such systems, the dispersed phase undergoes elongation during a heating and shearing process, typically using a high-temperature shear cell. When a force is applied to the system, the sample will fracture at the weakest points, which are typically located at the interfaces between the dispersed and continuous phases. Thus, comprehending the fracture behaviour of these systems can provide insight into the phase-separated structure of meat analogues.

We first comment on the specimen and test conditions to use (or not to use) during tensile testing of meat analogues. Four different tensile mold sizes and three different tensile test speeds were studied. To understand the link to meat analogue structure, we used one homogenous sample consisting of solely SPI and water, and a heterogenous, fibrous sample consisting of SPI, pectin and water. We found that smaller mold sizes and a slower test speed enable us to better interpret the fracturing behavior of the meat analogue samples. Additionally, we show the value of a dynamic anisotropic index, to further understand how anisotropy arises. The transition point from elastic to plastic behaviour and the toughness are introduced as new tensile parameters in the analysis of meat analogues. Furthermore, we showed that the stress-strain and force-deformation curves themselves can also provide us with information on the structure of our samples. Next, we comment on using a novel imaging technique to obtain a better understanding of the fracturing behaviour, and thus the structure, of the meat analogues. Here, we combine tensile testing with digital image correlation (DIC). DIC was used to analyse the crack initiation and propagation in homogenous gels as well as fibrous meat analogues. We propose the idea that number of cracks is related to visual fibrousness of meat analogues. We show that the cracking pattern of fibrous samples is a lot more heterogeneous than the cracking pattern of gel-like samples. Furthermore, we describe how to use true deformation data from DIC instead of using the raw deformation data of the texture analyser itself.

We postulated that an increase in the elongation of the dispersed phase would result in a perceptible enhancement of the fibrous structure and an increase in the number of cracks as observed through digital image correlation (DIC) analysis. Therefore, in this study, we investigate the intricate relationship between food structure and rheology.

F10. Structure formation and structure evolution during high moisture extrusion of soy proteins studied by scattering techniques

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Due to health and environmental factors, the food industry is looking for ways to introduce meat alternatives made from plant-based proteins to consumer markets. High-moisture extrusion (HME) is the most widely used method for converting proteins and polysaccharides into a meat alternative with a highly hierarchical fibrous structural organization. However, the HME process is usually considered as a "black box" with limited information about structure formation inside. The dead-stop operation is a feasible and effective method to collect material from different extruder and cooling die zones. In this study, scattering techniques were applied to study the evolution of meat analogue structure by sampling material after a dead-stop. By combining wide-, small-, and ultra-small-angle scattering methods, we have probed the structures of meat alternatives from the nano- to the micrometre range and established relationships between them. We have demonstrated that anisotropic structures were present already in the transition zone of the extruder and they developed further along the entire cooling die section. It also appeared that these anisotropic structures were formed at length scales of a few tens of nanometres with an increase of anisotropy at larger length scales. These findings represent a starting point in the understanding of the mechanisms of structure formation and evolution in the extruder and cooling die at several hierarchical levels.

F11. The impact of protein denaturation and solubility of soy protein isolates on structure formation during high moisture extrusion

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To sustainably feed the growing world population, a shift from animal to alternative protein-based foods is needed. In this context, there is an increasing demand for e.g. plant-based meat alternatives. High moisture extrusion (HME) is a technique frequently used to obtain products with a meat-like fibrous structure from plant protein. However, there is no consensus on the mechanisms underlying fiber formation during HME, which is crucial for successful product development. A notable knowledge gap in this regard concerns the relationship between the raw material physical properties and their behavior during HME. Accordingly, this study aims to investigate the impact of varying degrees of protein denaturation and solubility in soy protein isolates (SPI) on product texture and formed protein-protein interactions and reactions in extrudates produced from such SPIs. SPIs were produced in-house from locally cultivated soybeans and subjected to controlled hydrothermal treatments to obtain SPIs differing in degree of protein denaturation and solubility. After characterization of their water holding capacity (WHC) and rheological properties at a hydration level of 60% w/w, these SPI samples were used for HME processing using a lab-scale Process 11 Parallel Twin-Screw Extruder with cooled slit die at moisture contents of 50% and 60% w/w. Extrudate texture was determined with texture profile analysis and Warner-Bratzler analysis. To better understand structure formation during HME, formed protein-protein interactions and reactions during HME were evaluated by analysis of the protein extractability in different buffers, known to disrupt different types of interactions and chemical bonds. Molecular weight (MW) distributions of the obtained extracts were analyzed with size-exclusion high-performance liquid chromatography. Fully denatured SPI with a low solubility had a higher WHC and complex viscosity at low strain compared to native SPI. HME with this SPI as raw material also resulted in a softer extrudate texture compared to samples produced from native SPI, regardless of the moisture content. Disruption of non-covalent interactions resulted in similar protein extractability for extrudates produced from native SPI as well as from fully denatured SPI with low solubility. However, in the former case the obtained extract contained a relative higher amount of protein with a MW similar to 7S subunits, while in the latter case a higher 2S albumin content was detected. Notable is that when an extraction medium was used in which all non-covalent interactions and covalent bonds were broken, an increase in the level of protein with high MW (> 100 kDa) was detected for both samples. This could indicate that isopeptide bonds or alternative protein crosslinks are formed during HME, which has to the best of our knowledge not previously been reported. In conclusion, altering the physical properties of SPI resulted in altered protein-protein interactions and reactions that took place during HME as well as resulted in different HME product texture. As a next step, blends will be prepared from native and denatured SPI prior to rheological characterization and HME processing. This will allow even better understanding the impact of the degree of denaturation and protein solubility of SPI on its behavior during HME.

G1. Viscosity in Crystallizing Agitated Sucrose Dispersions

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If supersaturated sucrose solutions are cooled down sufficiently without disturbance or prior nucleation, they represent a meta-stable system in which agitation can induce rapid crystallization. This is intentionally used for example in the production of fondant, and represents an interesting process from a physical point of view: while simple in containing only water and sucrose, the system undergoes complex kinetic processes during nucleation and crystal growth which highly depend on concentration, temperature and agitation. Due to the complex nature of such non-equilibrium processes, the exact mechanisms remain unclear and scientific literature is scarce.

In this study, we investigate the course of the crystallization process and the according viscosity development over time in highly supersaturated sucrose solutions in a closed system. For this, temporal progression of the torque was measured in a temperature-controlled batch kneading machine and compared with rheological experiments of selected samples. Light microscopy to study crystal size distributions was performed additionally. Microscopy pictures showed that a high number of conglomerates formed and broke apart under shear, corresponding to a peak with subsequent decrease in the torque of the kneading system. We demonstrate that crystallization times at constant agitation speed correspond to classical nucleation theory. Furthermore, changes in apparent viscosity during crystallization are affected by concentration decrease of the liquid phase, growth of the crystalline phase as well as changes in particle size distribution. All three mechanisms influence measured apparent viscosity on different levels depending on the amount of completed crystallization and agitation geometry. These findings contribute to understanding the fundamental principles of such non-equilibrium systems, and offer possibilities to control crystal quality in various sucrose products in confectionery or pharmaceutical industries.

G2. Shaping the structure of blends from sunflower press cake and whey proteins through heat treatment and fermentation

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The current food system suffers from the inefficient use of resources, including the generation of side streams of low economic value, but still containing nutritional components. One potential approach to reach a more sustainable food system is to reintroduce such side streams into a circular value chain, and valorise them in novel food products, preferably in an unrefined or minimally refined manner. Moreover, blending side streams from different industries might be a suitable way to exploit functional synergies of structuring components such as proteins and polysaccharides, and to improve the nutritional value of the final food matrix. In this study, we combined the side streams from sunflower oil production (i.e., sunflower seed press cakes) and cheese manufacture (i.e., whey and whey proteins) to obtain novel food matrices containing valuable proteins, structuring polysaccharides, as well as lactose and minerals facilitating the fermentation.

Press cake, whey powder, and whey protein concentrate were dispersed in milk ultrafiltrate to prepare different blends with varying sunflower protein to whey protein ratio (100:0–0:100) but equal dry matter (~26%) and protein content (~10%). Structure formation as a function of heating temperature was studied by heating the blends to 80, 120, or 140 °C in a Rapid Visco Analyser (RVA), either undisturbed or under continuous, moderate shear applied by a paddle rotating at 160 rpm. The bulk viscosity of the unheated blends increased with increasing press cake concentration (0–22.5%) due to the higher polysaccharide to protein ratio. As observed from the torque profiles measured by the RVA, heat treatment at 120 and 140 °C increased the viscosity of the blends. A lower heating temperature, 80 °C, barely affected the sunflower components, but resulted in some denaturation of whey proteins, thereby increasing the viscosity of blends low in press cake and high in whey proteins. Confocal microscopy revealed that undisturbed heating at 120 and 140 °C resulted in the formation of a homogeneous gel network, whereas heating under moderate shear fostered the formation of a more heterogeneous structure, with protein aggregates dispersed in a continuous matrix.

Fermentation trials using three different co-cultures, each comprising one strain of lactic acid bacteria and one yeast strain, were conducted on the blend with the highest press cake content (22.5%) for maximum valorisation of the side stream. The samples were heated in an autoclave at 120 °C for 5 min without agitation prior to inoculation, as a homogeneous structure was assumed favourable for the fermentation. The pH development during fermentation at 26 °C was recorded, and samples were withdrawn for analysis after 12, 24, and 48 h. Small amplitude oscillatory shear rheology showed no significant changes in the storage modulus with fermentation, and confocal microscopy revealed a homogeneous microstructure for the unfermented and all fermented blends. This research provides important insights in the structure formation during processing of biomacromolecule blends and shows the potential of fermentation as a mean to stabilise side stream blends and modulate their sensory properties while only minimally affecting their physical appearance.

G3. Effect of the ink rheological properties and printing parameters on the filament spreading during 3D printing of Pectin

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The attraction toward 3D food printing is growing significantly since the development of extrusion-based 3D printing technology. Making food printable is one of the primary issues. To obtain printable consistency, biopolymers are frequently incorporated into the food ink formulation. The biopolymer-based inks' rheological properties significantly impact the ability of food products to be printed. Obtaining high-fidelity printed shapes is essential to raise consumers' interest in printed food. Therefore, it is vital to highlight the factors relevant to shape formation. Indeed, it is paramount to adequately control the dimensions of each printed line to form 3D printed objects correctly. This work investigates the impact of the food ink formulation, the effect of the biopolymer concentration and rheological behavior, and the impact of the processing parameters on the spreading of the deposited materials. The spreading is defined as the ratio between the measured printed line width and the targeted line width corresponding to the nozzle diameter. Low-methoxyl pectin was used in this study due to its wide-ranging usage as a gelling or thickening agent. Lines were printed using these pectin inks at varying concentrations of 0.5-12 w/w% to represent various rheological behaviors. It was highlighted that increasing the viscosity and the complex storage moduli or decreasing the loss tangent resulted in printed lines with a reduced overall spreading. Low viscosity inks' printing was demonstrated to be unsuitable as high spreading occurred due to their liquid-like behavior. Moreover, this study highlighted the importance of the printing parameters, i.e., the nozzle diameter, the applied pressure, the distance between the printing bed and the nozzle, the printing speed, and the temperature of the printing bed on the line width. Printing with a larger nozzle diameter resulted in an overall decrease in the spreading ratio, thus achieving better printing precision. Furthermore, it was demonstrated that increasing the printing speed and decreasing the applied pressure significantly reduced the width of the printing lines. Design of Experiments were carried out to develop models linking the rheological properties of the ink, the printing parameters, and the spreading of the printed lines. By continuing to enhance the knowledge in the field of 3D food printing. An increase in the number of food inks formulation can enable the development of tailored 3D printed foods with high fidelity for personalized nutrition.

G4. Towards Printability Predictions of Complex Food Inks: Formulation and Rheology

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3D printing technologies are gaining interest in food applications with the promise of personalization and digitalization of nutrition. A current challenge is to achieve a targeted shape and formulation avoiding trial and error. Rheology is often suggested as a robust tool to clarify the relation between material properties and printability. The ideal food ink should be easily extrudable while it adopts a shape fidelity following the extrusion, which translates to an adjustment of the shear thinning and the yielding properties. Accordingly, our aim is to express the printability of complex food ink formulations by their rheological properties, where we adopt a systematic formulation approach to elucidate the relation. The effects of macronutrient composition, moisture content and the moisturizing technique on ink rheology were investigated. Food inks were formulated using various combinations of pea fractions. Starch, protein, and fibre dominated samples were prepared at different water levels by two moisturizing methods, i.e., solid to water ratio and water holding capacity (WHC) based approach. Flow properties, namely the yield stress and shear thinning exponent, were obtained by the capillary rheometry (101-1031/s). A strain sweep (1-100%-1Hz) was utilized to obtain the flow point (crossover of G'-G") and the strain thinning index. Finally, post-printing recovery was evaluated by the step-shear experiments. Rheological properties were compared with the extrusion force and surface defect index to conclude on printability. Results revealed that WHC based moistening approach is better to provide systematic outputs. Water and protein content were found to have a dominant impact on the rheological properties. G' and yield stress appeared to correlate well with printability. Finally the shear and strain thinning indices were observed to hold a similarity under suitable conditions. Overall, this study offers a set of rheological parameters and a systematic approach to take a step forward in understanding printability. Hereby, the need for trial-and-error efforts to achieve a targeted design and formulation can be alleviated.

G5. Effects of Bran Pigmentation and Parboiling on Rheological Properties of Waxy Rice in Neutral and Acidic Environments

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Obesity is associated with serious health risks including type 2 diabetes and cardiovascular disease, and greater risk for severe illness during the COVID-19 pandemic. About 42.4% of U.S. adults and 13% adults worldwide were obese between 2017 and 2018. Overeating, one of the leading causes for obesity, is reflected by failure of mechanisms inducing satiety signals. High viscosity foods have been reported to increase the sense of satiety during meal consumption by delaying gastric emptying. Recent studies show that proanthocyanidins, a class of polyphenols, are shown to be capable of interacting with gluten protein (prolamins) in wheat, barley and rye to increase batter viscosity. Pigmented rice is a good source of polyphenols, particularly anthocyanins. Parboiling is a three-step hydrothermal process consisting of soaking, steaming and drying of rice to increase head rice yield and retain nutrients. Parboiling influences rice functional properties via starch gelatinization and protein denaturation, but its effects on protein-polyphenol interactions are unknown. The objective of this study was to investigate the rheological properties of pigmented (HB-1) and non-pigmented (Neches) waxy rice in neutral (water) and acidic (0.1 N HCl) environments as affected by soaking at room temperature or 5°C below onset gelatinization temperature (To) and steaming. Pasting properties of starch, whole grain and milled rice were determined before and after soaking using a Rapid ViscoAnalyser. Oscillatory frequency sweep was performed to investigate rheological properties of parboiled whole grain and milled waxy rice flours in neutral and acidic environments at 37°C to simulate human gastric condition. Whole grain rice and starch of the rice varieties Neches and HB-1 shared similar pasting viscosities, whereas milled HB-1 displayed a higher peak viscosity than its derived starch. Soaking at 5°C<To increased pasting viscosities of whole grain and milled Neches and whole grain HB-1, but decreased pasting viscosities of milled HB-1. When soaked at 5°C<To, parboiled whole grain and milled HB-1 showed restricted swelling in a neutral environment, but displayed enhanced viscoelastic properties in an acidic environment. The results provide evidence for the presence of inherent crosslinks between polyphenols and proteins in pigmented rice. These crosslinks may be strengthened from protein denaturation during parboiling but disrupted when exposed to acid to allow for increased starch swelling. Protein-polyphenol interactions may help develop functional foods with improved rheological properties for enhanced satiety that may reduce dietary caloric intake.

A12. Controlling chocolate surface properties and gloss formation potential by the choice of contact / mould material

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Gloss is a main criterion for chocolate quality and hence consumer acceptance. Today, gloss inhomogeneities, i.e. shiny and matt spots on the chocolate surface, remain a problem for manufacturers as these inhomogeneities occur even after apparently optimal pre-crystallization and cooling of the chocolate.

It is already known that chocolate gloss after demoulding is highly influenced by the degree of roughness and polarity (specifically the dispersive share) of the mould material [1]. However, the energetic interactions between chocolate components in the liquid state as influenced by the type of contact material have not yet been investigated. The presented research deals with clarifying the complex interactions between dark chocolate in contact with different mould materials (varying in polarity and roughness based on composition and production techniques), and focusing on changes in chocolate surface properties and structure. To get a deeper insight, we aim at identifying and characterizing using the appropriate length scale surface properties of both mould and chocolate at specific locations where gloss inhomogeneities occur. For this purpose, an improved method is being implemented to analyze local polarities at the µm scale using AFM equipped with functionalized tips.

Results on polarity, gloss, AFM surface topography and calculated roughness of chocolate and mould surface are presented. Based on these results, the relationship between surface properties of mould and chocolate as well as the occurrence of gloss inhomogeneities are described. Furthermore, the development of functionalized tips will be explained.

[1] Middendorf, D., Heinz V., Bindrich, U. & Franke, K. (2019) „The glossy chocolate bar: How can it be attained using the right mould", Manufacturing Confectioner, No. 99, S. 86–90.

A13. Rheology of semi-liquid shortenings in relation to composition, processing and storage temperature

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Semi-liquid shortenings are increasingly being investigated as alternatives for butter and margarine in industrial bakery applications driven by the health concerns related to the intake of saturated fatty acids. In semi-liquid shortenings, a low amount of lipid hardstock will form a (gelled) three dimensional network that entraps the liquid oil. Hereby, the ability to retain the liquid oil inside the network or the oil binding capacity, the overall rigidity and the ability to recover when sheared are of main importance. Before applying the semi-liquid shortenings to bakery applications, these are stored at a certain temperature. During this storage period, undesired changes like oiling off and/or softening might occur. Therefore, research about their structural changes upon storage is crucial. In this research, the stability of four dynamically produced semi-liquid shortenings was investigated. All semi-liquid shortenings contained 6% hardstock in rapeseed oil, two of them were triglycerides (TAG) and two were monoglycerides (MAG). Within each type (TAG – MAG), one was based on hydrogenated palm oil and one on hydrogenated rapeseed oil. The semi-liquid shortenings were stored at 5, 15 and 20°C for 8 week and analyzed after three time points (1 week, 4 weeks, 8 weeks) with rheology (amplitude sweep, flow measurements, thixotropy), polarized light microscopy (PLM), oil binding capacity (OBC) test, differential scanning calorimetry (DSC) and X-ray scattering techniques (XRS). It was found that the rigidity and rheology of the TAG-based shortenings was more impacted by storage temperature compared to the MAG-based ones. For the TAG-based shortenings, the rigidity was higher when stored at higher temperatures (G*20°C > G*15°C > G*5°C). Additionally, flow curves indicated higher values for the yield and flow stress. Storage at 5°C resulted in a flow curve with a traditional shear thinning behavior, while storage at 15 and 20°C resulted in the presence of an overshoot. When comparing the palm-based shortenings with the rapeseed-based ones, it was found that the former ones showed a higher rigidity and yield stress. However, they had a lower recovery capacity in the three step thixotropy test. Generally, no drastic decrease in the oil binding capacity and rigidity were found as function of the storage time. This showed that the semi-liquid shortenings were stable up to 8 weeks of storage, which is an important quality characteristic for their applicability in the food industry.

A14. Impact of time and applied shear during processing on fluid gels' rheology and dynamics

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Fluid gels are jammed gel particle suspensions which are obtained by applying shear to a gelling hydrocolloid during the sol-gel transition. They exhibit material properties (e.g., yield stress, shear thinning behaviour, viscoelastic response) tuneable via formulation and process parameters. Their flexible functionality enabled their application in several industries (e.g. as suspending agent, foam, and emulsion stabiliser).

The rheological properties of fluid gels are subject to a large uncertainty and evolve with time when left unperturbed (García et al., 2015). We established a methodology to study and quantify the fluctuations in the rheological properties, which were shown to be caused by the intrinsic granular matter nature of fluid gels. Thus, we have investigated in detail how time impacts the rheology of fluid gels. We have developed a 3-step cycle-based method consisting of: 1) rejuvenation in oscillatory mode to erase ageing effects while minimising the relaxing strain from this step; 2) waiting time (tw) where no stress was applied; 3) creep with a stress below the yield stress to retrieve the relaxation time τ. The results showed that τ(tw) increases until an asymptotic constant value $\tau\infty$, found for both an ionotropic fluid gel (using Ca2+ and low acyl gellan gum) and a non-ionic fluid gel (using agar). This behaviour has then been modelled for both systems. In addition, a method was developed to tune the density difference between the fluid gel particles and the continuous phase to study the influence of the density difference on ageing. Our findings suggest that the time changes in fluid gel rheology and dynamics are caused by physical ageing induced by the thermally activated colloidal arms surrounding the fluid gel particle core (D'Oria et al., 2023).

The newly developed methodologies enabled to characterise the impact of applied shear rate during processing, on the rheology and dynamics of fluid gels. We observed that the effect of time and number of cycles performed on G' causes a faster increase in storage modulus for samples processed at a higher shear rate. We hypothesised that fluid gels processed at a higher shear rate result in more porous structures caused by an overall fluid gel particle elongation. This hypothesis is corroborated by an increase in the fluid gel particle aspect ratio distribution for systems processed at higher shear rates. Finally, this study provides new tools to systematically analyse fluid gels' rheological properties by taking into account their granular and colloidal nature. This will not only help in a more precise characterisation of fluid gels for research purposes but, will also boost the industrial application of fluid gels by enabling a precise tuning of their properties.

García, M. C., Alfaro, M. C., & Muñoz, J. (2015). Yield stress and onset of nonlinear time-dependent rheological behaviour of gellan fluid gels. Journal of Food Engineering, 159, 42–47. D'Oria, G., Gunes, D. Z., Lequeux, F., Hartmann, C., Limbach, H. J., & Ahrné, L. (2023). Fluid gels' dual behaviour as granular matter and colloidal glass. Food Hydrocolloids, 137, 108401.

A15. Structure and rheology of oil-continuous capillary suspensions containing water-swellable cellulose beads and fibres

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Cellulose particles are used in a range of materials and applications. Here, we investigated the role of cellulose sorptivity on the rheology and microstructure of oil-continuous capillary suspensions consisting of cellulose beads and fibres at volume fractions of 0.3 to 0.65 and water at volume fractions up to 0.2. We hypothesised that, in spite of their water-absorbent nature, cellulose particles in oil would be structured by water added as an immiscible secondary phase. The interplay between cellulose bead load and water content was used to generate suspensions ranging from flowable to highly elastic with moduli near 1 MPa. While capillary bridging induced a liquid to semi-solid transition, nearer particle volume fractions of 0.65, we found a transition to a solid, wet-granular material. Liquid-solid transitions occurred even if the water was absorbed by the particles, suggesting that the increase in elasticity was not solely due to the formation of capillary bridges, but also a result of particle swelling and the associated increase in effective packing volume fraction. Swapping the beads with fibres increased stiffness by 4 orders whereas swapping half of the beads at the same water fraction increased material stiffness by 3 orders of magnitude. The two conclusions of this study are that, even for water-sorptive particles, capillary interactions can be used to structure oil-continuous suspensions, and that the use of high aspect ratio particles can increase the rigidity of oil-continuous capillary suspensions to a greater extent than beads at an equivalent volume fraction.

A16. Comparing structural differences between ice crystal-dominated and fat network-dominated ice cream by oscillatory rheology

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Ice cream is a food with a complex microstructure consisting of air cells, ice crystals, and a network of coalesced fat droplets entrapped in a thick continuous phase. This microstructure is responsible for its viscoelastic behavior. Due to such complex structure, it is difficult to gain insight into the relative importance of the different structural elements on viscoelastic properties, as changes of one microstructural feature will also affect others. This makes it challenging to understand the exact role of fat content, ice fraction, fat network and dispersed air cells in ice cream properties. Up to now, oscillatory rheology has not been extensively used in ice cream research. In this study, we investigated the individual effect of each microstructural parameter by changing separately overrun, fat destabilization degree or ice crystal size while keeping the other characteristics constant. This gave us the opportunity to relate specific structural features to the viscoelastic behavior of ice cream. To vary the degree of fat destabilization, we changed the emulsifier type; to modify overrun and ice crystal size, we used either a batch freezer or liquid nitrogen freezing upon whipping.

Depending on the composition of the ice cream, we identified two main types of structures: one dominated by ice crystals, and one dominated by a fat network. The viscoelastic properties of our samples were mainly determined by the ice crystal structure, and less by the fat network. In presence of an ice crystal structure, also overrun had a large effect, but overrun had less effect on ice cream with a fat network. Similar viscoelastic properties were observed upon melting for the ice creams with different microstructure in the frozen state. However, the presence of a fat network played a more dominant role than the ice crystal network on the melting process. After the ice crystal network had disappeared, the viscoelastic properties were determined by the degree of fat destabilization, and overrun played a limited role. In the case of ice cream with a more extensive ice crystal network, melting was a two-step process; (1) the early stage where overrun had limited effect, and (2) the later stages where the overrun slowed down the melting behavior once the structure of connecting ice crystals had disappeared. This research proved that oscillatory rheology can provide valuable information on the structural organization of ice cream and that different structural characteristics affect the properties of ice cream in a different way depending on specific microstructural features.

A 17. Effects of rapeseed oleosome membrane composition on curcumin encapsulation and oleosome stability

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A drive towards healthy and sustainable food find common ground in functional foods. These provide health benefits through the incorporation of bioactive compounds. To effectively administer these compounds in significant doses and improve their bioaccessibility they require a stable hydrophobic environment. In this study, rapeseed oleosomes with different membrane structures had their applicability as a carrier of the bioactive compound curcumin investigated. This applicability was tested with regards to encapsulation efficiency and oleosome stability. These parameters were determined for native oleosome emulsions and homogenized oleosome emulsions with reduced particle size and membrane density. Our analysis revealed that complete encapsulation could be achieved for both emulsions, with only a reduced efficiency (73.7 wt%) for homogenised oleosome emulsions at higher curcumin loads (1.4 mg/g oleosome as opposed to 0.14 mg/g oleosome). Interfacial dilatational rheology showed that the stability of homogenised oleosomes was much higher than native oleosomes, and that for both emulsions this stability was unaffected by the loading of curcumin. It further showed that curcumin has interactions with the phospholipids and/or proteins on the interface, leading to differences in viscoelastic behaviour between oleosomes with or without the presence of curcumin. The interfacial activity of curcumin might have also been the cause for the higher free fatty acid release observed for emulsions with curcumin. The particle size, however, was revealed to be a more important factor for this free fatty acid release, as fractions of 23.5 and 31.7 wt % were measured for emulsions of native and homogenised oleosomes, respectively. Overall, our results show that rapeseed oleosomes are effective as carriers of curcumin, and that the alterations of the native membrane structure, for instance by homogenisation, can lead to higher stability.