Physics in Food Manufacturing 2020 Conference

15–17 January 2020
Weetwood Hall Estate, Leeds, UK

Organised by the IOP Liquids and Complex Fluids group
## Programme

**Wednesday 15 January**

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<tr>
<th>Time</th>
<th>Session</th>
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<tr>
<td>10:00</td>
<td><strong>Registration and coffee</strong>&lt;br&gt;Lawnswood Breakout</td>
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<tr>
<td>10:30</td>
<td><strong>Welcome remarks</strong>&lt;br&gt;Megan Povey, University of Leeds, UK&lt;br&gt;Lawnswood Suite</td>
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<tr>
<td>10:40</td>
<td><strong>Introduction to Physics in Food Manufacturing Group</strong>&lt;br&gt;John Bows FinstP, PepsiCO, UK&lt;br&gt;Lawnswood Suite</td>
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<tr>
<td>10:50</td>
<td><strong>Introduction to Liquids and Complex Fluids Group</strong>&lt;br&gt;Sergey Lishchuk, Sheffield Hallam University, UK&lt;br&gt;Lawnswood Suite</td>
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**Session 1: Complex fluids and rheology including surface rheology, extensional flows and oral processing**

**Chair:** Anwesha Sarkar, University of Leeds, UK  
**Location:** Lawnswood Suite

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<th>Time</th>
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<tbody>
<tr>
<td>11:00</td>
<td><em>(Plenary)</em> The physics of chocolate conching&lt;br&gt;Wilson Poon, University of Edinburgh, UK</td>
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<tr>
<td>11:40</td>
<td><em>(Invited)</em> Clustering of triacylglycerols in the molten state&lt;br&gt;Michael Rappolt, University of Leeds, UK</td>
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<tr>
<td>12:00</td>
<td><em>(Invited)</em> Understanding the biomechanics of swallowing to develop safer and more pleasurable food and drinks to meet the needs of older people&lt;br&gt;Marco Ramioli, University of Paris-Saclay, France</td>
</tr>
<tr>
<td>12:20</td>
<td><em>(Postgraduate)</em> Tribology and rheology of thermodynamically incompatible biopolymer mixtures&lt;br&gt;Kwan-Mo You, University of Leeds, UK</td>
</tr>
<tr>
<td>12:35</td>
<td><em>(Invited)</em> Models of surface viscosities of particle-laden fluid interfaces&lt;br&gt;Sergey Lishchuk, Sheffield Hallam University, UK</td>
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<tr>
<td>13:00</td>
<td><strong>Lunch</strong>&lt;br&gt;Woodlands Suite Restaurant</td>
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Session 2: Complex fluids and rheology including surface rheology, extensional flows and oral processing  
**Chair:** Wilson Poon, University of Edinburgh, UK  
**Location:** Lawnswood Suite

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<th>Time</th>
<th>Session</th>
<th>Speaker</th>
<th>Institution</th>
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</table>
| 14:00  | **Plenary** Soft colloidal microgels as bio-functional materials: from mouth to gut  
          Anwesha Sarkar, University of Leeds, UK |                                   |                                      |
| 14:40  | **Postgraduate** In vitro gastric digestion of pea protein microgel particle stabilized Pickering emulsions coated by cellulose nanocrystals  
          Shuning Zhang, University of Leeds, UK |                                   |                                      |
| 14:55  | **Postgraduate** Modification of rheological and tribological performance of continuum by addition of microgels  
          Efren Alberto Andablo Reyes, University of Leeds, UK |                                   |                                      |
| 15:10  | **Postgraduate** A self-assembled binary protein model explains high-performance salivary lubrication from macro to nanoscale  
          Evangelos Liamas, University of Leeds, UK |                                   |                                      |
| 15:30  | **Coffee Break**  
          Lawnswood Breakout |                                   |                                      |

Session 3: Extensional flows in food and modelling  
**Chair:** Sergey Lishchuk, Sheffield Hallam University, UK  
**Location:** Lawnswood Suite

| Time   | Session                                                                 | Speaker                          | Institution                          |
|--------|-------------------------------------------------------------------------|----------------------------------|                                      |
| 16:00  | **Plenary** Modelling the viscosity of mixtures of Na-caseinate stabilised droplets using a soft particle approach  
          Bill Frith, Unilever, UK |                                   |                                      |
| 16:40  | **Invited** On the origins and possibilities of biocompatible bijels  
          Brent Murray, University of Leeds, UK |                                   |                                      |
| 17:00  | **Invited** Particle based modelling in industrial processing  
          Matt Sinnott, CSIRO Data61, Australia |                                   |                                      |
| 17:20  | **Industrial pitch**  
          Lawnswood Suite |                                   |                                      |
| 17:30  | **IOP Physics in Food Manufacturing Group AGM**  
          Lawnswood Suite |                                   |                                      |
| 18:00  | **Poster session and drinks/light buffet**  
          Bramley Room |                                   |                                      |
| 21:00  | **Close** |                                   |                                      |
## Thursday 16 January

<table>
<thead>
<tr>
<th>Time</th>
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| 08:00 | Registration and coffee  
*Lawnswood Breakout*                                                   |
| 08:30 | Introduction to the IOP  
*Lawnswood Suite*                                                         |

### Session 4: Crystallisation, rheological changes, thickening, gelation

**Chair:** Megan Povey, University of Leeds, UK  
**Location:** Lawnswood Suite

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<th>Time</th>
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| 08:40 | *(Plenary)* Crystal engineering approaches for the food industry  
Elena Simone, University of Leeds, UK                                         |
| 09:20 | *(Invited)* Biopolymer-based “soft matter” assemblies and their influence on percolated bio-networks  
Francisco Goycoolea, University of Leeds, UK                                 |
| 09:40 | *(Invited)* Impact of pressure on the crystallisation of Cocoa Butter  
Marjorie Ladd Parada, Stockholm University, Sweden                           |
| 10:00 | *(Postgraduate)* Optimal control of ice morphology: targeting microstructure in crystallisation processes  
Estefania Lopez-Quiroga, University of Birmingham, UK                         |
| 10:15 | *(Invited)* Kinetic theory of shear thickening  
John Melrose, Jacobs Douwe Egberts, UK                                        |
| 10:40 | Coffee break  
*Lawnswood Breakout*                                                          |

### Session 5: Measurement science challenges and physical measurements in foods

**Chair:** Valerie Pinfield, Loughborough University, UK  
**Location:** Lawnswood Suite

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<th>Time</th>
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| 11:00 | *(Plenary)* Intelligent sensing labels for food safety assurance  
Xiaobo Zou, Jiangsu University, China                                            |
| 11:40 | *(Invited)* Rapid and non-destructive detection of agri-product and food  
Shi Jiyong, Jiangsu University, China                                            |
| 12:00 | *(Invited)* Particle characterisation of food formulations  
Stephen Ward-Smith, Malvern Panalytical Ltd, UK                                 |
| 12:20 | *(Invited)* Process engineering and manufacture (including powder handling, conveying, in-line rheology, rheological changes, thickening, gelatinisation)  
Graham Worrall, Centre for Process Innovation, UK                             |
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<tr>
<th>Time</th>
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<tr>
<td>12:40</td>
<td><strong>Industrial pitch: Campden BRI</strong></td>
<td>Martin Whitworth, Campden BRI, UK</td>
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<td><em>Lawnswood Suite</em></td>
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<td>12:55</td>
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<td>13:05</td>
<td><strong>Lunch</strong></td>
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<td><em>Woodlands Restaurant</em></td>
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<td><strong>Session 6: Physics in digestion including flow</strong></td>
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<td><strong>Chair:</strong> Alessandro Gianfrancesco, Nestlé Product Technology Centre, UK</td>
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<td><strong>Location:</strong> Lawnswood Suite</td>
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<td>14:00</td>
<td><em>(Invited) Modelling the structure and chemistry of iron supplements and intravenous iron drugs</em></td>
<td>Helen Chappell, University of Leeds, UK</td>
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<td>14:20</td>
<td><em>(Invited) The physics of digestion</em></td>
<td>Alan Mackie, University of Leeds, UK</td>
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<td>14:40</td>
<td><em>(Invited) Impact of salt on the structural properties of bread using X-ray and neutron tomography</em></td>
<td>Emanuel Larsson, RISE Research Institutes of Sweden, Sweden</td>
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<td>15:00</td>
<td><strong>Coffee break</strong></td>
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<td><em>Lawnswood Breakout</em></td>
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<td>15:40</td>
<td><strong>Industrial pitch: Malvern Panalytical</strong></td>
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<td>15:50</td>
<td><strong>Early careers panel discussion – re-imagining food production in a sustainable world</strong></td>
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<td>17:00</td>
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<td>18:00</td>
<td><strong>Gala dinner: after dinner speaker</strong></td>
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<td><em>Woodlands Suite Restaurant</em></td>
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<tr>
<td>08:00</td>
<td><strong>Registration and coffee</strong></td>
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<td><em>Lawnswood Breakout</em></td>
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<tr>
<td>08:30</td>
<td><em>(Plenary)</em> Acoustic measurement in food processing</td>
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<td></td>
<td>Valerie Pinfield, Loughborough University, UK</td>
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<td>09:10</td>
<td><em>(Postgraduate)</em> Conjugated soy protein as food emulsifiers</td>
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<td>Yue Ding, University of Leeds, UK</td>
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<td>09:25</td>
<td><em>(Postgraduate)</em> Effect of amylose to amylopectin ratio on oral behaviour of OSA-modified starch stabilised emulsion</td>
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<td>Mingdou Mu, University of Leeds, UK</td>
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<td>09:40</td>
<td><em>(Postgraduate)</em> Chocolate to couscous: a unified treatment of granulation and suspension rheology</td>
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<td>Daniel Hodgson, University of Edinburgh, UK</td>
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<td>09:55</td>
<td><em>(Postgraduate)</em> Functional properties of African orphan amaranth and finger millet</td>
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<td>Andreia Silva, University of Edinburgh, UK</td>
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<td><em>Lawnswood Breakout</em></td>
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<td>11:30</td>
<td><em>(Plenary)</em> The role of moisture in manufacturing of food powders: current challenges and future trends</td>
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<td>Alessandro Gianfrancesco, Nestlé Product Technology Centre, UK</td>
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<td>12:10</td>
<td><em>(Invited)</em> Physics of dairy powder manufacture: Use of advanced microscopy and ultrasound spectroscopy</td>
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<td>Zachary Glover, University of Southern Denmark, Denmark</td>
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<td>12:30</td>
<td><em>(Invited)</em> Applying physics of “macromolecules at interfaces” to the design of novel food colloid emulsifiers and stabilisers</td>
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<td>Rammile Ettefaie, University of Leeds, UK</td>
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<td><strong>Industrial pitch</strong></td>
<td><em>Lawnwood Suite</em></td>
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<td>13:00</td>
<td><strong>Announcement of poster prize and closing remarks</strong></td>
<td><em>Lawnwood Suite</em></td>
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<td>13:20</td>
<td><strong>Lunch</strong></td>
<td><em>Woodlands Suite Restaurant</em></td>
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<td>P1</td>
<td>Evaluating the fate of microgel-stabilised Pickering emulsions during in vitro gastric digestion: role of electrostatic coating versus covalent-conjugation with polymers</td>
<td>Andrea Araiza-Calahorra, University of Leeds, UK</td>
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<td>P2</td>
<td>A theoretical technique for screening protein fragments with potential as suitable emulsifiers and stabilisers</td>
<td>Cuizhen Chen, University of Leeds, UK</td>
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<td>P3</td>
<td>High resolution microscopy and spatial correlation analysis applied to dairy gels</td>
<td>Adam Cohen Simonsen, University of Southern Denmark, Denmark</td>
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<td>P4</td>
<td>Cryogenic applications in food industry</td>
<td>Eleni Fitsiou, Lancaster University, UK</td>
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<td>P5</td>
<td>High speed, simultaneous absorbance, transmission and fluorescence excitation emission spectroscopy (A-TEEM) makes complex food analysis practical and quantitative</td>
<td>Adam Holland, Horiba UK Ltd, UK</td>
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<tr>
<td>P6</td>
<td>Power spectrum measurements using ultrasound reflection spectroscopy</td>
<td>Liam Morris, University of Leeds, UK</td>
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<td>P7</td>
<td>Development of reduced fat powdered coffee creamer formulated with rice bran oil extracted from thai rice by-products</td>
<td>Linda Pravinata, University of Leeds, UK</td>
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<td>P8</td>
<td>The physics of chocolate conching</td>
<td>Wilson Poon, University of Edinburgh, UK</td>
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The physics of chocolate conching

Wilson Poon

University of Edinburgh, UK

The mixing of a powder of 10- to 50-μm primary particles into a liquid to form a dispersion with the highest possible solid content is a common industrial operation. Building on recent advances in the rheology of such “granular dispersions,” we study a paradigmatic example of such powder incorporation: the conching of chocolate, in which a homogeneous, flowing suspension is prepared from an inhomogeneous mixture of particulates, triglyceride oil, and dispersants. Studying the rheology of a simplified formulation, we find that the input of mechanical energy and staged addition of surfactants combine to effect a considerable shift in the jamming volume fraction of the system, thus increasing the maximum flowable solid content. We discuss the possible microscopic origins of this shift, and suggest that chocolate conching exemplifies a ubiquitous class of powder–liquid mixing.

Clustering of triacylglycerols in the molten state

Michael Rappolt1, Amin Sadeghpour1, 2, Marjorie Ladd Parada1, 3, Josélvio Vieira4 and Megan Povey1

1University of Leeds, UK, 2Swiss Federal Laboratories for Materials Science and Technology, Switzerland, 3University of Stockholm, Sweden, 4Nestlé Product Technology Centre, UK

The study of triacylglycerols (TAGs) in their molten state is of fundamental importance for a deeper understanding of TAG-crystallization processes being highly relevant for both, manufacturing and medical applications. Whilst different models have been proposed to explain the nanostructured nature of the fluid state of TAGs, none of them are fully satisfactory. We propose a new model consisting of positionally uncorrelated lamellar TAG-assemblies embedded in an isotropic medium, that assist as pre-nucleating structures. This model was validated by applying a novel global fitting method, resulting in excellent agreement with the small angle X-ray scattering data [1]. Deeper analysis of the scattering patterns at different temperatures, both in cooling and heating direction, allowed us further to detect crystalline traces of TAGs even after heating to 40 °C, and record on cooling the onset of crystallization at 30-25 °C. The application of the presented novel model not only explains the outstandingly structured fluid of molten TAGs, but also lays the basis for analyzing first crystallization steps in greater detail [2].

Understanding the biomechanics of swallowing to develop safer and more pleasurable food and drinks to meet the needs of older people

Marco Ramaioli
University of Paris-Saclay, France

Food oral processing (FOP) is a key step for a pleasurable eating experience, for a safe swallowing, for a comfortable digestion and for an adequate nutrition.

Swallowing disorders are a very common consequence of several pathologies, such as neurological disorders or cerebrovascular diseases, and can lead to an increased risk of aspiration pneumonia, malnutrition, dehydration, and isolation.

In recent studies, in vivo ultrasound observation [1], a simplified fluid mechanical analysis [2] and novel biomimicking experiments [3] are combined to understand better the different regimes governing the biomechanics of swallowing.

The effect of the bolus rheological characteristics on the bolus dynamics and cohesion were also investigated, supporting the development of novel products that have the potential of being both safer and more palatable [4].

A study of the flow of a bolus containing solid inclusions [5] will also be presented as well as research perspectives to understand better the oral processing of solid food products and to improve their structure to meet the specific needs of older people.


Tribology and rheology of thermodynamically incompatible biopolymer mixtures

Kwan-Mo You, Brent Murray and Anwesha Sarkar
University of Leeds, UK

Thermodynamically incompatible biopolymer mixtures are widely used in food, pharmaceuticals, tissue engineering, prosthetics and many other technological applications due to specific physical and mechanical properties. In this study, we investigated the rheological and soft tribological properties of biopolymer mixtures using optimized concentrations of waxy corn starch (WCN) and κ-carrageenan (KCN). The biopolymer mixtures were prepared by mixing WCN (0.5-10.0 wt%) and KCN (0.05-1.0 wt%) at 90 °C followed by cooling down to room temperature (22 °C). Apparent viscosity results reveal that both WCN and KCN on their own had shear-thinning behavior. The WCN + KCN mixtures also showed a non-Newtonian behavior with viscosity values being the average of the individual equilibrium phases at corresponding shear rates. In terms of soft tribological properties, KCN decreased friction coefficients in the mixed lubrication regime depending upon concentration (≥ 0.5 wt%) and interestingly KCN at such high concentration also showed hydrodynamic regimes. Striking behavior was shown by WCN, which not only decreased friction coefficients in mixed regime but also in the boundary regime (≥5 wt%), this might be attributed to the
presence of ghost granules that somehow were entrained in the contact region. Surprisingly, unlike the rheology results, tribological properties of WCN + KCN mixtures followed the behavior of WCN in the boundary regime decreasing the friction in the lower entrainment speeds (0.005-0.05 m/s), whereas the mixed and hydrodynamic regime was dominated by KCN. Preliminary results from this new study show opportunities for designing new aqueous lubricants with tunable frictional properties by optimizing the concentrations of the individual biopolymers.


Acknowledgements

The European Research Council is acknowledged for its financial support (Funding scheme, ERC Starting Grant 2017, Project number 757993) for this work

Models of surface viscosities of particle-laden fluid interfaces

Sergey Lishchuk
Sheffield Hallam University, UK

From the point of view of physics, many foods and beverages are soft interface-dominated materials. A particular class of such materials are those which contain food-grade particles adsorbed at fluid interfaces. Surface rheology of particle-laden fluid interfaces plays an important role in formation, stability, and dynamics of food emulsions and foams as well as in sensory physics. Although a vast amount of experimental studies of rheology of particle-laden interfaces in different conditions has been accumulated, theoretical description of the rheology lags behind. Many studies tend to use general phenomenological constitutive models which are fitted to experimental data and have little link to the structure and processes which occur in these complex systems.

I will focus on the microscopically based description of the viscous part of viscoelastic rheological response of particle-laden fluid interfaces. The theoretical models of surface viscosities to be presented are based on generalising Albert Einstein’s approach to calculating the effective shear viscosity of suspensions to the case of a flat interface separating two immiscible fluids laden with monodisperse rigid spherical non-Brownian particles. This approach has allowed calculation of the following effective viscous properties of this system: (i) dilatational viscosity in the limit of small and large concentration of the adsorbed particles [1,2], (ii) dependence of the dilatational viscosity upon the contact angle between the two fluids and the particle surface [3], (iii) surface shear viscosity at small concentration of particles [1], and (iv) the modification of the surface shear viscosity due to the presence of an incompressible surfactant layer [4].

Soft colloidal microgels as bio-functional materials: From mouth to gut

Anwesha Sarkar
University of Leeds, UK

Soft colloidal microgels are hydrogel microparticles with well-defined deformability that arises from the swollen nature of the integral biopolymeric network. Although practical applications ranging from drug delivery to diagnostics to tissue engineering are mainly fueling the research momentum of microgels, these colloidal gel particles are also extremely important to address fundamental biophysical research questions. Using a combination of multiscale experimental techniques and theoretical considerations, this talk will present an overview on the bio-functional performances of soft biopolymeric microgels (Fig. 1). Specifically, two case studies will focus on the oral lubrication properties\textsuperscript{1-3} of the microgels, where these microgels present in the bulk phase act as viscosity modifiers. Soft microgels designed using a top-down approach with proteins or starches of variable cross-linking degrees with or without containing oil droplets will be discussed. Their ability to act as high performance lubricating agents in elastomeric contact surfaces (with different wetting properties) emulating oral surfaces will be examined under the influence of oral shear and/or salivary enzymes. Some of these microgels showing aqueous ‘ball-bearing’ abilities depending upon their volume fraction will be highlighted\textsuperscript{2-3}. Also, a case study will be presented on how these proteinaceous microgels can be fused when present at the oil-water interface to alter lipid digestion kinetics\textsuperscript{4} of Pickering emulsion droplets. These recent advances on bio-functional properties of microgels hold promise for designing foods in the future with tailored properties.

Fig 1: Microgels in bulk phase for oral lubrication (a) and at the interface for altering lipid digestion properties (b) performances, respectively.

\textsuperscript{[1]} A. Sarkar, et al., Curr. Opin. Colloid Interface Sci 39, 61-75 (2019)
\textsuperscript{[2]} A. Sarkar, et al., Langmuir 33, 14699-14708 (2017)

Acknowledgements

The European Research Council is acknowledged for its financial support (Funding scheme, ERC Starting Grant 2017, Project number 757993) for this work.
In vitro gastric digestion of pea protein microgel particle stabilized pickering emulsions coated by cellulose nanocrystals

Shuning Zhang¹, Melvin Holmes¹, Rammile Ettelaie¹, Brent Murray, Anwesha Sarkar¹

¹University of Leeds, UK

Gastric flocculation and often coalescence of protein or protein-particle stabilized emulsions due to the interfacial proteolysis by pepsin are key reasons of destabilization in human physiology and offer as a challenge to deliver lipophillic bioactive compounds to targeted intestinal sites¹,². In this study, we designed Pickering emulsions stabilized by pea protein microgel particles (PPM, 1 wt%), coated with cellulose nanocrystals (CNCs) (1–3 wt%), latter being unresponsive to human proteolytic enzymes³. The hypothesis was that a secondary layer of CNCs at the PPM-stabilized oil-water interface (O/W) could protect the interfacial protein gel particle layer against in vitro gastric digestion by pepsin at 37°C. A combination of confocal microscopy, ζ-potential measurements and interfacial shear viscosity measurements suggested the presence of CNCs and PPM together at the O/W interface, owing to the electrostatic attraction between complementarily charged PPM and CNCs at pH 3.0. Microstructural analysis and droplet sizing revealed that the presence of CNCs increased the resistance of the interfacial proteinaceous microgel film to rupture by pepsin, thus inhibiting droplet coalescence in the gastric phase, which occurs rapidly in an emulsion stabilized by PPM alone. It appeared that there was an optimum concentration of CNCs at the interface for such barrier effects. In addition to adsorption of CNCs to the protein gel particle-coated droplets to form more rigid layers, there is also the possibility that network formation by the CNCs in the bulk (continuous) phase reduced the overall kinetics of proteolysis. Nevertheless, structuring emulsions with mixed protein particle-particle layers could be an effective strategy to tune and control interfacial barrier properties during gastric passage of emulsions.


Modification of rheological and tribological performance of continuum by addition of microgels

Efren Andablo-Reyes, Evangelos Llamas, Simon Connell and Anwesha Sarkar

University of Leeds, UK

Biocompatible microgels are envisioned as substitute lubricants in physiological processes, for example, to preserve oral lubrication properties on decreasing fat content.¹ In particular, aqueous dispersions of whey protein microgels (WPM) have demonstrated their capacity to reduce friction in soft contacts mimicking biological tissue.² In this work,³ the lubrication performance of soft (G’ ~ 100.0 Pa) and hard (G’ ~ 10.0 kPa) WPM dispersed in Newtonian (phosphate buffer and 50 – 75 wt% corn syrup solutions) or complex (0.5 - 1 wt% xanthan gum solutions) continuum was studied. Dynamic light scattering and atomic force microscopy estimated the particle diameter at around 100 nm. Rheological characterisation was performed using rotational rheometry (cone-plate) covering shear rates from 0.1 to 1000.0s⁻¹. Lubrication performance of WPM was studied using a rolling/sliding soft contact formed by an elastomeric ball and disc tribopair supporting a load of 2.0 N. Both, soft and hard microgels acted as thickeners in low viscosity Newtonian continuum, decreasing friction in the soft contact. Friction reduction was larger for the harder WPM.
Surprisingly, in high viscosity continuum, microgels acted as thinning agents decreasing viscosity and increasing measured friction. In the lubrication limit, microgels in buffer or corn syrup solutions behaved as Newtonian fluids with effective viscosity corresponding to their second Newtonian plateau value ($\eta_\infty$). The lubrication performance of the microgels dispersed in the complex fluid could not be described quantitatively. For the low viscosity xanthan gum, the microgels had no influence on friction. Nevertheless, for the high viscosity counterparts, the soft microgels acted as thinning agents whilst the hard microgels accelerated the onset of elastohydrodynamic regime. As summarised in figure 1, viscosity modification by microgels influences lubrication, depending upon the interplay of rheological properties of the particles and continuum, which has implications for microstructural design of future food formulation of reduced fat content.

Fig 1: Friction coefficient as function of WPM viscosity and AFM image of WPM.

[1.] A. Sarkar, E. Andablo-Reyes et. al., Current Opinion in Colloid and Interface Science, 2019 39, 61-75
[3.] E. Andablo-Reyes et. al., Soft Matter, 2019, DOI: 10.1039/C9SM01802F Acknowledgements: The European Research Council is acknowledged for its financial support (Funding scheme, ERC Starting Grant 2017, Project number 757993) for this work

A self-assembled binary protein model explains high-performance salivary lubrication from macro to nanoscale

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Salivary pellicle is a spontaneously-formed macromolecular architecture in the oral cavity1. Although saliva-mediated lubrication is pivotal to food oral processing and sensory perception, the molecular mechanism behind such lubrication remains elusive. Here, we report an unprecedented lubrication mechanism² associated with binary salivary protein that self-assemble via an electrostatic mechanism. From macro-to-nano scale, our results reveal the similarity between the lubrication properties of binary salivary-protein assembly and that of human saliva. Friction coefficients ($\mu$) associated with the multi-layered architecture of binary salivary-proteins, i.e. negatively charged bovine submaxillary mucin (BSM, 2.6 MDa) and positively charged salivary protein, lactoferrin (LF, 80 kDa) was assessed in orally relevant conditions (1-10 mM NaCl, pH 6.8, 37 °C) from macro to nanoscale using ball-on-disc tribometer, pin-on-plate set up, nano-grafting-assisted atomic force microscopy (AFM), with applied forces ranging from 1 nN to 1 N. Self-assembly formation was investigated using Quartz Crystal Microbalance with Dissipation Monitoring (QCM-D) employing various surfaces, e.g. polydimethylsiloxane (PDMS)-coated, gold-coated and positively-charged gold-coated sensors. In addition, self-consistent field theory (SCF) calculations were applied to investigate
the interactions between LF and BSM. Remarkably, QCM-D results revealed non-monotonic growth of BSM/LF multi-layered architecture with adsorption of \( \approx 25-45 \text{ mg m}^{-2} \) and associated formation kinetics of \( 0.1-0.15 \text{ mg m}^{-2} \text{ s}^{-1} \), which uniquely resemble those of human saliva. Also, BSM/LF architecture with 4.5 bilayers rendered hydrophobic PDMS sensors (static contact angle \( 102.7^\circ \)) to a substantial hydrophilicity \( (<10^\circ) \). Our findings show that performance of the binary BSM/LF system were comparable to those of human saliva, with dramatic reduction in \( \mu \) at both boundary and fluid film regimes. Experimental results supported by SCF theory highlights that low concentrations of small molecular positively charged proteins, such as LF in real human salivary pellicle acts as a 'molecular glue' that catalyse mucin-mucin networking as well as mucin-surface binding. Mucin controls the macromolecular hydration lubrication, and lactoferrin acting as the molecular glue aids boundary lubrication. Insights generated by our study brings new thinking for designing future nature-mimetic saliva substitutes for dry mouth patients and helps emulating just-right artificial salivary formulation for colloidal scientists.


Acknowledgements

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Modelling the viscosity of mixtures of Na-caseinate and Na-caseinate stabilised droplets using a soft particle approach

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Products such as ice cream present unique challenges in terms of manufacturing and formulation. Such materials comprise many phases in varying degrees of disequilibrium, the main ones being ice, air, oil/fat and aqueous solution(s). Their texture, flavour and quality are dependent on the microstructure of these phases, most of which will evolve over time during manufacture and storage. Also crucial to achieving a desirable product are the properties of the unfrozen aqueous phase in which the ice and air phases reside. This liquid matrix is a complex mixture of dissolved biopolymers, proteins, micelles and droplets and understanding the contributions of these components to the matrix rheology is a still unsolved challenge.

One of the major constituents of the matrix in ice-cream is a dairy protein stabilised emulsion, either in the form of milk or separately emulsified vegetable fat. In this talk I will present our investigations of the rheology of model protein stabilised emulsions and the development of a predictive model for their behaviour. Such protein-stabilised emulsions can be seen as mixtures of unadsorbed proteins and of protein-stabilised droplets. To identify the contributions of these two components to the overall viscosity of sodium caseinate o/w emulsions, the rheological behaviour of pure suspensions of proteins and droplets were characterised, and their properties used to model the behaviour of their mixtures. These materials are conveniently studied in the framework developed for soft colloids. Here, the use of viscosity models for the two types of pure suspensions allows the development of a semi-empirical predictive model that relates the viscosity of protein-stabilised emulsions to their composition.

On the origins and possibilities of biocompatible bijels

Brent Murray and Rammile Ettelaie

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Bijels can arise from the phase separation of biopolymer solutions via spinodal decomposition and where a particle acts as a Pickering stabilizer of the water-water (W/W) interface. Such systems have all sorts of potential uses, such as for controlled release and templating for high interfacial area materials. So far there seem to be few examples of such systems that are truly completely biocompatible, e.g., consisting of food-based materials, that would ease development of their applications for drugs, cosmetics, etc., as well as in foods themselves.

Through modelling of biopolymer phase separation, and determination of the interfacial tension between the two co-existing phases and a solid surface via self-consistent field calculations, it is possible to explain the difficulties in achieving biopolymer-based bijels. The computed difference in the free energy per unit area (i.e., interfacial tension) between a solid (non-interacting) particle surface and the co-existing polymer solutions is extremely low (~ 1 N/m) and results simply from the differences in the change in the free energy due to polymer depletion close to the surface of the particles. Although this value may seem rather small, this difference is more than capable of inducing the strong partitioning of particles of 100 nm in size (or larger) into the phase with the lower interfacial free energy, as opposed to the one with the higher value.

By examining the density profile variation of the polymers close to the surface, when the incompatibility between the two polymers is sufficiently large, the contact angle for a particle at the W/W interface is predicted to always be close to 90° and potentially this could lead to bijels – with the proviso that the particles must be larger than the width of the W/W interface, which is significant for the extremely low W/W
interfacial tensions (confirmed experimentally by other workers). This explains why the only practical biopolymer bijels that have been seen so far seem to involve fairly large particles, such as protein microgels, stable emulsion droplets or even bacterial cells, and thus leads to fairly large characteristic bijel layer spacings.

Particle based modelling in industrial processing

Matt Sinnott, Paul Cleary, Sharen Cummins, Gerald Pereira, James Hilton and Simon Harrison
CSIRO Data61, Australia

Industrial processing typically involves multiple sequential unit processes (either batch or continuous) to turn raw ingredient materials into edible products at a commercial scale for a consumer market. These processes can involve handling of complex materials such as granular powders or multiphase fluid suspensions or deformable solids interacting with moving equipment with complex 3D geometries. Physics based computational models can provide insights into a given process that may inform design or operational choices to improve efficiency or manage manufacturing issues where direct measurement is limited or difficult. The requirements for such models may also need to predict the effects of heat treatment, gas transport, phase change, and chemical reactions. For many of these systems, there are benefits in using particle based models over traditional grid based continuum methods.

In particle-based models the physics of interest is resolved at the spatial scale of a “particle” which may represent an individual powder grain or a small volume of food product in the case of continuum materials. They are well suited to fully transient problems with moving boundaries. Since each simulation provides the full history of each local part of the system, these approaches are more straightforward to include additional physics such as complex material behavior or chemistry. Here we introduce Discrete Element Method for modelling granular materials and Smoothed Particle Hydrodynamics for continuum materials. Examples (some of which are shown in Fig. 1) will demonstrate the use of particle based modelling for bulk material handling, mixing/blending, interactions with gas flow, thermal processing, and bubbly flows.

Fig 1: (a) Cohesive powder in a ribbon blender; (b) Mixing/extrusion of a fluid paste (the 2nd screw impeller at the top is not shown); (c) Dust generation from conveying grains through a transfer chute
Crystal engineering approaches for the food industry

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Many food products such as salt, sugar and butter are crystalline and several complex food structures such as ice cream contain crystals. Crystal properties such as shape, size and polymorphism can have a dramatic effect on the properties of these food products in terms of stability, taste and texture as well as bioavailability. Therefore, tailoring crystal properties and controlling crystal nucleation and growth during food product manufacturing is essential to ensure the final product quality.

The Crystal Engineering Approach utilises the understanding of the intermolecular interactions within a crystalline structure for the predictive, systematic design of crystals with specifically tailored properties as well as the crystallization processes necessary to achieve such crystals.

Our group is applying this crystal engineering approach for: (1) better understanding the effect of crystal properties on the quality of complex food structures; (2) designing optimal manufacturing processes to control crystal nucleation and growth and obtained crystals with the desired properties; (3) developing novel control technologies and strategies to monitor crystallization processes. Few examples of our work include molecular modelling of flavonoid structures, foam stabilization using fat crystals and complex soft structure characterization using ultrasound, rheology and X-ray tomography.

Figure 1 shows selected examples of the work currently being carried out in the group.

Fig 1: (a) Design, monitoring and control of crystallization processes; (b) Design of crystals for soft structures stabilization; (c) Fluid dynamic modelling of particle fractionation; (d) Molecular modelling of hydrate structures.
Biopolymer-based “soft matter” assemblies and their influence on percolated bio-networks

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Biopolymer-based nanocapsules (d.~200 nm; \( \mu \text{-potential} \approx +60 \text{ mV} \)) formed by spontaneous emulsification and electrostatic self-assembly, under a phase separation-driven process, can be furnished with a wide range of chitosans of varying degree of N-acetylation and molar mass. These “soft matter” systems are amenable to be loaded with lipophilic phytochemicals that occur in food and traditional medicinal plants, namely capsaicin, \textit{trans}-cinnamaldehyde, (quercetin, capsaicin, curcumin). In general, these systems associate more than 60% of the active payload and are stable against aggregation in biological media. Our results show consistently that the association of different type of bioactive phytochemicals results in enhanced anti-QS, anti-biofilm, and anti-adhesion of bacteria. Also, enhanced permeability, antiproliferative, enhanced motility and cytoprotective effects, have all been evidenced on different mammalian cell lines. Although the precise mechanisms of action of these systems are yet to be fully uncovered, we have demonstrated both experimentally and using computational simulations, that chitosan nanocapsules bind “stoichiometrically” to the bacterial envelope of \textit{E. coli}, and this leads to bacterial aggregation. Drug-free genipin-crosslinked nanoparticles have also been examined. They are able to disrupt bacterial network communication. In other studies, we have been addressing the use of chitin nanofibers to fill insect protein gels, as a strategy to modulate the mechanical properties. We have attempted to explain these phenomena in the context of network percolation theory. The potential application of these findings to conceive novel sustainable food systems is yet to be fully realised.

The impacts of pressure on the crystallisation of fats

Marjorie Ladd Parada
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Fats have been studied for decades, especially their crystallisation because by controlling it one can achieve new textures, not to mention promote stability in a wide variety of products, especially in confectionery. Most studies have focused on the effects of thermal treatment, that is, cooling and heating rates, and crystallisation temperatures, or in their composition either regarding fatty acids or the overall triglycerides. However, the effects of pressure specifically on crystallisation are yet to be fully understood, particularly at pressures that allow for continuous treatment of the fats, rather than batch processing. This talk aims to provide a brief overview on the current status artis, as well as present some of our most recent data where we pressurised cooled cocoa butter below 1000 bar and followed the crystallisation process in situ using X-ray diffraction.
Optimal control of ice morphology: targeting microstructure in crystallisation processes

Estefania Lopez-Quiroga
University of Birmingham, UK

Crystallisation processes are involved in a wide range of manufacturing methods and applications in food processing. For example, the first stage of lyophilisation processes – i.e. freezing and thus crystal formation – is key to determine a well-interconnected porous structure that will allow for drying and further rehydration or dissolution of the dried product. Chocolate manufacture also involves a step – tempering – that determines fat crystallisation and with it texture too. And texture in ice cream is given by targeting a certain crystal size/distribution that enhances mouth feel. In all these cases, crystal/microstructure formation determines both quality and function of the final products, so optimal control of the operating conditions that lead to crystal formation and growth is critical.

In this context, this work presents a model-based strategy to define optimal operating conditions for ice crystal formation in food model systems. The core of the optimisation problem is a Phase Field model that couples heat and mass transfer phenomena and describes the evolution of the solid/liquid interface, predicting the evolution of phase change interfacial kinetics and the formation of the product microstructure. The model was used to evaluate different operating scenarios - i.e. range of degrees of supercooling and seeding/nucleation conditions - and determine which combination of freezing temperature and freezing times resulted in a targeted final microstructure, which was defined as a given crystal size distribution. Cases considering both constant and time-dependent freezing conditions were studied, and the resulting dynamic optimisation problem solved using a Control Vector Parametrisation (CVP) method. The obtained optimal control policies where then simulated and compared, revealing the potential of model-based strategies to control and optimize structuring processes like crystallisation. This provides a powerful (virtual) tool to support process design in food manufacture.

Kinetic theory of Shear thickening

John Melrose and Robert Farr
Jacobs Douwe Egberts, UK

Farr et al (1997) published a kinetic theory of the many body physics underlying the jamming of dense suspensions under applied shear. The theory was based on the Smoluchowski aggregation equation with a sum kernel modelling the growth of rod-like clusters around the compression axis of flow. It will be shown that by adding a break-up term based on contact relaxation, steady-states can be generated with these equations and these both show thickening effects and thickening transitions. Although highly simplified with respect the geometry of particle packing, this does give a predictive theoretical basis beyond many particle simulations by which to approach this problem.

Intelligent sensing labels for food safety assurance

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Intelligent food packaging is designed to test the quality and safety of packaged foods in the supply chain. As miniaturized and portable sensors, intelligent sensing labels have received widespread attention in intelligent packaging. According to the different response signals of intelligent sensing labels, they can be divided into electronic labels and optical labels. Electronic labels generally transmit the electrical signals generated by the sensors to the receiver through a radio frequency identification system. Optical labels generally produce visible light color changes or fluorescence changes. At present, there are some commercial products of electronic sensing labels in intelligent food packaging, such as the time-temperature recorder [1, 2]. Although optical sensing labels for oxygen have commercial available products, optical sensing labels for food freshness are mostly at the research level. The key and difficult point in developing food freshness sensing labels is that the safety, stability, selectivity and sensitivity of the sensors must meet the requirements at the same time. This report mainly introduces the research progress of several electronic and optical sensing labels in intelligent food packaging and some research of our research group in this field. We believe that with the continuous optimization and development of various sensors, more industrialized sensing labels will be born in the future.


Rapid & nondestructive detection of agri-product & food

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The quality and safety of food and agro-products is a growing concern in global trade. In recent years, nondestructive methods of quality and safety evaluation have gained momentum and a considerable attempt has been made to develop them for their objective, consistently, and accurately test. Much research is now being directed toward the development of nondestructive measurement devices that are versatile, economical, and simple to use.

Machine vision, near infrared (NIR) spectroscopy and hyperspectral imaging are powerful techniques to extract and quantify features for food and agro-products assessment and control. In our study, machine vision was used to detect the color, shape and defect of fruit and vegetables. Online detection machine vision systems were developed for grading apples, oranges and tomatoes. NIR was used for qualitative and quantitative detection of agri-product & food quality, such as soluble solids content (SSC) in apples and nutrient components in edible birds’ nest. Hyperspectral imaging is a powerful technique for providing high-quality spectral and spatial information on samples. Hyperspectral imaging was used to estimate the distribution map of quality index in agri-product & food. The chlorophyll concentration distribution maps of cucumber leaves were detected and the chlorophyll distribution features were extracted for rapid and nondestructive detection of nutrient deficiencies successfully.
Material characterisation of food formulations

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Malvern Panalytical Ltd, UK

In 2016, UK Government figures put the size of the UK’s food and drink manufacturing industry at £29.5 billion, employing 390,000 people. Many food and drink companies use formulation processes to ensure the delivery of good-quality products made in a reproducible way.

Many raw materials are pre-treated in some way before they are used. Raw ingredients, including flour, nuts, cocoa and sugar, are often ground before incorporation, and emulsions will usually be homogenised. The effectiveness of these processes can be monitored by characterising the resulting material.

The particle size, shape and charge of food ingredients are key parameters in the final product. Particle size and shape influence how a material flows (smaller material tends to be stickier than larger material – think of the difference between icing sugar and granulated sugar), its rate of dissolution, and its mouthfeel. The human tongue is an extremely sensitive particle sizing device in its own right, detecting any particle larger than 30 microns in diameter as having a gritty or greasy texture.

The charge on a material is normally indicative of its shelf-life, with a change in pH or salt concentration often employed to induce electrostatic stabilisation, thus preventing coalescence or separation. Another option is the addition of a thickening agent, which will promote steric stabilisation, but this may also lead to a change in the product’s rheological behaviour.

On occasion, there could also be contamination in our food – this can have devastating consequences, as evidenced in the Chinese milk powder scandal.
For all these reasons and many more, materials characterisation technologies are a key component of any food formulator’s toolset. This talk will cover some of the processes and their associated challenges in food product manufacture, and the characterisation techniques that can be used to address them.

**Process engineering and manufacture (including powder handling, conveying, in-line rheology, rheological changes, thickening, gelatinisation)**

Graham Worrall
Centre for Process Innovation, UK

Commissioning of In-line Viscometer

The overall objective of the PROSPECT CL processing facility is to develop and deploy NEW and INDUSTRIALLY relevant capability to assist the UK formulation industry in the manufacture of liquid products. The novel research infrastructure created consists of a simple, flexible, multi-scaled liquid processing loop to which a range of measurement techniques can be integrated in order to allow in-line measurement during formulation processing. This will be housed at CPI’s National Formulation Centre and will be available as an open-access capability.

The primary end-use will be in:

1. Developing, validating and utilising new process analytics capability
2. Understanding the dynamics of manufacturing formulations at different scales with a view to manufacturing to an ENDPOINT rather than manufacturing to a RECIPE

Essential to achieve the above is the requirement to accurately determine key processing analytics which can be used to provide feedback control to the process. Incorporation of in-line viscosity measurement provides one such control metric in this open access facility. The need to validate such measurement devices against conventional off-line measurements is fundamental to this approach. CPI have installed an in-line viscometer in the manufacturing processing loop, presented here is the approach taken, highlighting equipment modifications necessary obtain reproducible results.
Iron Deficiency Anaemia (IDA) effects nearly a sixth of the global population and accounts for numerous disabling symptoms including fatigue, impaired immune responses, poor cognitive development in children, and ultimately maternal and child mortality. Treatment options for IDA include dietary modification, home fortification, oral iron supplementation (e.g. IHAT, Figure 1) and intravenous iron (IV). These latter two treatment options, which have been in use for many decades and are based on the use of nanoparticulate iron-oxides (Figure 1), are particularly interesting from a structural chemistry perspective.

Here we use the Near and Intermediate Range Diffractometer (NIMROD, ISIS) to carry out neutron diffraction studies, and first principles computational methods to provide insight into these experimental results. Using Pair Distribution Function analysis, we combine the experimental and theoretical data to elucidate details of the iron-oxide phases and the detailed chemical structure of ferrihydrite, the main inorganic iron-oxide phase of both the supplements and IV irons. Computational modelling is then employed to characterize the interactions between the iron-oxide particle surface and the organic ligands of the coating.

The results include an improved ferrihydrite structure [1], and an elaboration of the chemical bonding of the organic ligands to the IHAT (Figure 1b) nanoparticle core. We have also shown how the character of this bonding changes through the digestive tract as pH alters, and provide an explanation for the late absorption of this novel iron supplement, which remains intact through the stomach and small intestine.

As the only nutritional deficiency that effects both developing and developed populations, new treatment options that can overcome potentially dangerous side effects are increasingly important and any chemical insight that can improve the rational design of these drugs will be immensely valuable.

Fig 1: (a) Intravenous iron gluconate [2]; (b) Oral iron supplement, Iron Hydroxide Adipate Tartrate (IHAT) [3].

The physics of digestion

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The digestion of food is a complex multi-scaled process ranging from separation behaviour over many centimetres in the stomach to nanometre scale molecular rearrangements allowing enzymatic hydrolysis. In this talk, I will cover some of the recent attempts to model gastric behaviour as well as evidence of phase separation gathered using magnetic resonance imaging. The modelling includes fluid dynamic approaches¹-² and assessment of buffering behaviour in protein gels. Finally in the intestine calcium plays an important dual role in lipid digestion, promoting removal of long-chain fatty acids from the oil–water interface by forming insoluble calcium soaps while also limiting their bioaccessibility. This becomes more significant in food containing high calcium concentration, such as dairy products³. Finally, the mixed micelles formed are carried to the surface of the mucus layer by mixing but must diffuse to the underlying epithelial cells⁴ where they disassemble and many of the constituents are absorbed. The role of both bile and soluble fibre in altering the properties, especially the permeability, of intestinal mucus are now becoming apparent but the high degree of heterogeneity makes a full understanding of transport in this part of the intestine highly challenging⁵-⁶. Surprisingly the transport phenomena in the gut are still not a well-understood process.


Impact of salt on the structural properties of bread using x-ray and neutron tomography

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Today we consume more salt than needed, by eating pre-prepared food, e.g. meat, bread and cheese products. Too much salt can lead to health issues, e.g. heart attack, heart failure, stroke and kidney damage [1]. WHO aims to reduce the global population’s intake of salt by 30% by 2025 [2]. By reducing the salt consumption to recommended levels an estimated 2.5 million lives can be saved [2]. The salt thus needs to be reduced, while maintaining the taste, quality, and processability, as required by both the consumers and producers. By exchanging many fine-grained salts with fewer layer-distributed coarser-grained salt, one can even obtain an increased sense of saltiness.
In this industrial-relevant work, we studied the impact of fine-grained and coarse-grained salt on the structural properties of bread using X-ray microtomography and Neutron tomography, whereas the latter is a sensitive technique to detect salt in bread and the former provides a high-resolution image of the bread structure. Studies have been performed at both at the IMAGINE beamline the at the Orphée reactor at Laboratoire Léon Brillouin, Saclay, France [3], [4], as well as at the D50 NeXT beamline at Institut Laue-Langevin, Grenoble, France [5], [6].

The tomographic data sets were used to extract a set of quantitative 3D descriptors about the bread, e.g. pore size, the thickness of the beams and crust and connectivity index. By quantifying the microstructure of food products, it is possible to optimize the processing parameters and thereby obtain an improved food quality [7]. A better understanding of the 3D distribution of salt and its impact on the structural properties of bread, and consequently also the tastiness and quality, will help to develop healthier food products with reduced total salt content.


Session 7: Colloid science in foods

Acoustic measurement in food processing

Valerie Pinfield
Loughborough University, UK

Acoustic measurements offer unique insights for food processing, both for understanding the materials themselves and for process monitoring and control. Primarily working in the long wavelength, non-resonant frequency region, measurements of sound speed and attenuation (often as spectra) have been used as the basis for characterising a variety of food systems or equivalent media. These are commonly, but not exclusively, systems of particles in liquids, either emulsions or suspensions, in the colloidal size range and using ultrasonic, Megahertz frequencies. A brief introduction to the acoustic measurement techniques and the accessible material properties will lead on to an overview of recent developments in this field. Recent progress in our understanding of the relationship between microstructure and the measured acoustic properties will be illustrated for concentrated particle dispersions, aggregated systems, and for multi-species systems based on multiple scattering and effective medium models. The results of finite element modelling will also be shown, demonstrating the effects of microstructure (in this case particle clusters), leading to interactions of the thermal and shear fields between nearby particles, thereby affecting the predicted
attenuation. The forthcoming challenges that still need to be addressed and the opportunities for acoustic measurement for food systems will be discussed.

Fig 1: Illustration of the effect of microstructure on predicted attenuation in an emulsion of sunflower oil in water of 0.25 μm diameter at 3 MHz. As the particles cluster together, the attenuation is reduced due to thermal interactions between particles.


Conjugated soy protein as food emulsifiers

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In food colloid formulations, one of the most critical functional ingredients, namely food emulsifiers and stabilizers, frequently tend to be animal-derived proteins, for example, milk proteins. Recent trends concerning green issues and sustainability, as well as a significant increase in the number of vegan consumers, have encouraged enormous research efforts on the use of plant-derived materials as possible substitutes for animal-based materials in common food products. However, the use of vegetable proteins as effective emulsifying agents is limited by their rather poor solubility, compact globular nature and their slow unfolding and adsorption kinetics.

The current study used a combination of two techniques (i.e. controlled enzymatic hydrolysis followed by conjugation with polysaccharides) to modify soy protein isolate (SPI) to be a better emulsifier and stabilizer. Two enzymes with relatively higher and lower specificity (i.e. Trypsin and Alcalase) were applied to hydrolyze SPI. In order to see the emulsifying and stabilizing abilities that solely come from soy protein itself, a relatively small and non-ionic polysaccharide Maltodextrin (i.e. MD19 of 8.9 kDa) without any gelling or emulsifying ability was chosen to attach onto protein fragments. Then the stability of O/W emulsion at neutral and acidic pH conditions were evaluated over a storage time of 60 days.
The two enzymes have generated protein fragments with remarkably different properties and Trypsin was found to be more suitable in regards of the modification of emulsifying property of SPI. Fresh emulsion with submicron-sized droplets (D4,3=0.638μm) was able to be produced by conjugated SPI hydrolysates.

In the evaluation of the storage stability of O/W emulsions, it was surprised to see that coalescence of oil droplets happened significantly in soy protein-based emulsions, particularly at pH7.5 where proteins as coating materials were highly charged and provided strong electrostatic repulsions. While at acidic conditions, e.g. pH4.5 and pH3.0 where oil droplets were in a flocculated state and close to one another, coalescence was on the contrary occurring at a much less extent. And also only when higher-molecularweight polysaccharides (i.e. dextran of 500 kDa) were attached with protein molecules, coalescence at pH7.5 was able to be effectively prevented.

**Fig 1:** Appearance of SPI hydrolysates by Trypsin and Alcalase

**Fig 2:** Structure and size distribution of emulsions made by conjugated SPI hydrolysates at pH7.5 and pH4.5 before and after a storage of 60 days

**Effect of Amylose to Amylopectin Ratio on Oral Behaviour of OSA-Modified Starch Stabilised Emulsion**

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Hydrophobically modified starch has been proven as an effective emulsifier and is used as such in the food industry. Octenyl succinic anhydride (OSA) is the only legally permitted modifying agent for this purpose, limited to 3% hydrophobic modification with respect to the weight of starch. Amphiphilic OSA-starch is obtained by esterification of hydrophilic starch and OSA containing a long hydrophobic chain. In recent years, possible preferential attachment of OSA has attracted increasing attention. Among all considered factors, starch composition and structures are by far the most influential ones on emulsifying properties. Composition-wise, it was found experimentally that there is a preference in OSA attachment for amylose as compared to amylopectin. The two starch molecules also show differences in adsorption behaviour onto the surfaces of oil droplets. In this work, corn starches with varying amylose contents were modified with the same amount of OSA (3%) to obtain emulsifiers, which were then used in fabricating 10% oil-in-water emulsions. Amylose content of the OSA-starches affected the stability of droplets against electrolyte concentration increase and pH changes, as well as the oral digestion behaviours of the emulsion. The low and medium amylose content OSA-starch stabilised emulsions both exhibited acceptable levels of resistance to destabilisation over a suitably wide range of pH values, at low background electrolyte levels. In contrast, the low amylose content modified starch provided a distinctly greater level of stability against increased electrolyte concentration. Oral behaviour of each emulsion stabilised by OSA-starch was
independent of the oral processing method employed. Both emulsions stabilised by low and medium amylose content modified starch were destabilised by the starch-digesting enzyme, amylase. However, destabilisation of the low amylose one was more clearly associated with coalescence, whereas that of medium content showed greater tendency for flocculation. This behaviour is rationalised in terms of more rapid hydrolysis of highly branched chains of amylopectin on the surface of droplets, in the low amylose content system.

Chocolate to couscous: a unified treatment of granulation and suspension rheology

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Incorporating a small amount of liquid into dry powders is a ubiquitous unit operation in food manufacture. In some cases, a minimal amount of liquid is used to produce matt solid granules. Such ‘wet granulation’ [1] has long been used in the manufacturing of, e.g. couscous, baby foods and other dehydrated convenience foods [2]. When too much liquid is added, the mixture becomes (in granulation jargon) ‘overwet’ [3]: it turns into a flowing suspension and granulation fails. In other cases, a larger amount of liquid is added, and the mixture turns into a flowing suspension e.g. liquid chocolate. If insufficient liquid is added in these systems, flow fails – they fracture and jam, e.g., near constrictions. To date, applied research into these two areas has proceeded separately: where the interest of one community ends (‘overwet’), the attention of the other begins. However, in terms of the amount of liquid incorporated, the preparation of granules and high-solid dispersions form a continuum. Thus, these two areas of industrial practice may be amenable to a single, unified description, with insights from each enriching the understanding of the other.

We perform experiments on these phenomena using Spheriglass, an industrially-realistic model powder [4]. Drawing on recent advances in understanding friction-induced shear thickening and jamming in suspensions, we offer a unified description of granulation and suspension rheology. A ‘liquid incorporation phase diagram’ explains the existence of permanent and transient granules and the observed increase of granule size with liquid content. Our results point to rheology-based design principles for industrial granulation.


Functional properties of African orphan crops amaranth and finger millet

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Amaranth and finger millet are African orphan crops, that due to their nutritional content show high potential to become innovative and nutritional ingredients in processed foods and drinks. Amaranth grains present a protein content of 14 to 19%, including albumins, globulins, and glutelin. Finger millet contains 5 to 8% of proteins, containing prolamins, albumins, globulins and glutelin. Furthermore, finger millet grains have the capacity to grow in extreme conditions.

In this study, we experimentally quantify how pH and salts (NaCl and CaCl₂) affect the protein’s interactions and consequently their functional properties. Amaranth protein isolate (API) and finger millet protein isolate (FMPI) were dispersed in buffer solutions with pH values ranging from 2 to 10, in distilled water, and NaCl and CaCl₂ water solutions with different concentrations. Solubility experiments show that FMPI have higher
solubility than API, especially when dispersed in NaCl. Dynamic light scattering and zeta potential experiments reveal that even the addition of small concentrations of CaCl₂ promote aggregation of FMPI. Aggregates are larger for both protein isolates at pH values close to the proteins isoelectric point. Both API and FMPI stabilize the water-air/oil interface, and oil in water emulsions form a cream layer within minutes. Another noteworthy observation is the ability of API to form gels.

There is a growing demand of healthy food products that are easy to consume and present a good nutritional balance. The identification of the functional properties of API and FMPI under different pH and salt conditions is crucial for the effective incorporation in existing processed food products as well as for the development of new food products using those grains.

**Key words:** Proteins, Amaranth, Finger millet, Functional properties

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The role of moisture in manufacturing of food powders: current challenges and future trends

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A huge variety of food products is found in powder form, from ingredients like milk, cocoa or fruits to finished products like soluble beverages, soups or instant coffee among many others. The moisture content is one of the key parameters to be controlled during production and shelf-life of powdered food, as it impacts the final stability of the product as well as the efficiency of the production process.

Many different technologies are available to manufacture food powders. The choice of the right process depends on the desired end-use and on the characteristics of the product. In this talk, we are going to analyze the role of moisture in three examples of processing: spray drying, freeze-drying and roller compaction.

The drying process has the obvious goal of removing water until a desired target, but also of generating specific powder structures (e.g. size, porosity, sphericity…) and of preserving sensorial properties like color, taste or nutrient content. The knowledge of the evolution of moisture content along processing is essential in order to define the best process parameters, which allow reaching the final moisture and structure with maximal energetic efficiency.

In spray drying, it is important to avoid fouling of the tower and cyclone by taking into account the stickiness of the product. This is a function of the adhesiveness of the surface, which depends on the local moisture content and can be related to the glass transition temperature. However, the concept of glass transition is not always sufficient to explain the development of adhesive behavior as a function of moisture content. We will discuss an example based on the drying of a liquid solution of sucrose and skimmed milk in different proportion, where the glass transition temperature is the same.

Freeze-drying is one of the processes used to produce soluble coffee. Due to the high volumes to be manufactured, it is necessary to guarantee a very fast drying kinetics while avoiding the collapse of the structure due to overheating. After freezing, the drying occurs in two main steps: the sublimation of ice crystals (primary drying) and the convective drying of the water bound to the cryo-concentrated matrix (secondary drying). Moisture plays a key role in the whole process. Depending on the product characteristics, the parameters applied during the freezing cycle will influence the amount and size of ice crystals generated, with direct impact on the final reconstitution properties (porosity). On the other hand, knowledge of moisture evolution during secondary drying allows optimizing the heating cycle in order to maximize water diffusion and reduce processing time.

Finally, we are going to discuss the dry granulation process by roller compaction. Granules are produced to improve physical characteristics of the powder, like flowability and dispersion in water. The main parameters to be controlled include roller pressure, gap between the rolls and screw feed velocity. The moisture content of the initial powder has an impact on the plasticity of the material during compaction, where the temperature increases locally because of the pressure applied. We will show how the understanding of the adhesive behavior as a function of moisture content can be used to optimize roller compaction of sensitive food powders like fruits.
Physics of dairy powder manufacture: Use of advanced microscopy and ultrasound spectroscopy

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The manufacture, transport and reconstitution of dairy derived powders are of industrial interest on a global scale. The production process needs to be understood and characterised physically in order to deliver a final product that has the same qualities as those made from native fresh milks. A combination of Super-resolution imaging and Ultrasound spectroscopy provides an overview of the dynamics, interactions and structural properties of reconstituted dairy products. Stimulated Emission Depletion (STED) microscopy has been used to image dairy gels, where protein structures have been resolved to under 100 nm using an imaging protocol, that aside from dye addition is non-perturbative. Quantitative image analysis has been developed using an empirically validated model to extract the size of protein domains, inter-pore distance and fractal dimension of the protein network. These three parameters can discriminate between gels produced from fresh or reconstituted milk and between gels induced by acidification or renneting. In two colour images fat droplet size can be extracted and the distance between fat droplets and local maximum protein distribution can be determined. The distance between fat and protein changes significantly following homogenisation. The distribution and quantity of protein around individual fat droplets can be determined with region specific analysis. Coherent Anti-Stokes Raman Scattering (CARS) microscopy provides a label free negative control for the use of a fluorescent dye in STED imaging.

Fluorescence time-life imaging provides spatially resolved micro-viscosity measurements within dairy gels and can be correlated to the different levels of moisture binding in gels produced from milks with different thermal processing histories.

The speed of sound and acoustic attenuation are dependent on a materials physical properties. Ultrasound Reflection Spectroscopy has been used to monitor the changes in the dissolutions of dairy powders and protein aggregation under changing pH. The attenuation spectrum provides can be used to size particles in concentrated colloidal dispersions without dilution and is sensitive to long order rehydration kinetics in reconstituting systems. Effective combination of these techniques provides a unique insight into the entire process of characterising a reconstituted dairy derived gel with unprecedented temporal and spatial resolutions for given conditions.


Applying physics of “macromolecules at interfaces” to the design of novel food colloid emulsifiers and stabilisers

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Behaviour of macromolecules adsorbed at different types of interfaces remains an area of great fundamental interest to Physical chemists, Chemical Physicists and Colloid scientists, alike. Understanding the adsorption behaviour of biomacromolecules, and in particular proteins, is also crucial in a more intelligent approach to the design of new and superior edible emulsifiers, for use in food colloid formulations. There are two major recent incentives driving the desire for such novel food grade emulsifiers. Firstly, a need for much better food colloid stabilisers than those currently available, in order to facilitate the realisation of the much promised bottom-up type approach to design of food structure. Secondly, sustainability and green issues shitting the food industry away from animal based proteins towards the use of plant based proteins. This latter in particular poses a challenging problem for food colloid scientists, since vegetable proteins have an inherently inferior emulsion stabilising properties compared to their animal counterparts.

In this talk we review and present some recent advances in realising efficient vegetable based food grade emulsifiers. Several different strategies, including Millard conjugation of proteins with polysaccharides, use of large polypeptide fragments derived from vegetable proteins and hydrophobic modification of polysaccharides, will be considered. We provide experimental and theoretical results from our current studies to exemplify what may be achievable in near future. These studies also highlight the underlying mechanisms by which each method allows the vegetable derived macromolecules to acquire good emulsification and colloid stabilising properties. Various options forward for improving each of the presented techniques are also suggested by our results and will briefly be discussed, too.
Evaluating the fate of microgel-stabilised Pickering emulsions during in vitro gastric digestion: Role of electrostatic coating versus covalent-conjugation with polymers

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Recently, particle-stabilized emulsions, also referred to as Pickering emulsions (PEs) have attracted significant research attention. However, designing PEs as delivery vehicles using natural, biodegradable particles that offer gastric stability faces new challenges in food colloid science. The aim of this study was to design novel protein-based soft gel particles as Pickering stabilizers for specifically generating ‘gastric-stable’ PEs. By using electrostatic and covalent interactions between whey protein and biopolymer (dextran), a bottom-up approach was used to design three different types of Pickering emulsions (20 wt% oil); whey protein nanogel particle-stabilized PE1, dextran sulphate (DxS)-coated whey protein nanogel-stabilized PE2 (using electrostatic coating of DxS of 40-500 kDa molecular weights), and whey protein-dextran conjugate microgel particle-stabilized PE3, latter using different degrees of conjugation with dextran before the microgel creation. Complimentary techniques ranging from static and dynamic light scattering, interfacial shear rheology, confocal and cryo-scanning electron microscopy, zeta-potential, sodium dodecyl sulphate polyacrylamide gel electrophoresis (SDS-PAGE), ortho-phthaldialdehyde (OPA), pH-stat to cross-correlation image analysis of the particles and the PEs allowed to compare the behaviour of the PEs, pre- and post-digestion. Under in vitro gastric conditions, both electrostatically-coated PE2 and conjugate microgel-stabilized PE3 presented a diffusive barrier to the pepsin-hydrolysis of the underlying protein particle-laden interface, which consequently delayed the interfacial proteolysis. In both PE2 and PE3, droplets were resilient to gastric coalescence unlike the unmodified PE1. Under in vitro intestinal conditions, the amount of free fatty acid (FFA) released was similar (p > 0.05) for all the three PEs, whilst, the rate constant and half time of the electrostatically-coated PE2 was the highest and lowest, respectively (p < 0.05) owing to droplet aggregation and reduction in surface area for lipase to bind. This study provides new design principles to create PEs with tailored barrier properties to allow developing delivery systems for lipophilic compounds that require targeted intestinal release.


P2 A theoretical technique for screening protein fragments with potential as suitable emulsifiers and stabilisers

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The protein hydrolysis method has been widely considered as a potentially useful way to improve the emulsification property of proteins and in particular vegetable proteins. However, common enzymes, such as trypsin and papain, with relatively broad specificity, can break proteins into various unpredictable fragments at different degrees of hydrolysis. Based on this prerequisite, the degree of hydrolysis should be precisely controlled in preparing emulsifiers for stabilising oil-in-water emulsions. The purpose of the current study was to identify theoretically the protein fragments which play a critical and predominant role in the process of stabilisation by a combined self-consistent field (SCF) calculation method and newly developed selection techniques. This study also suggests a feasible way to obtain a specified fragment from an intact protein for future experimental investigation.

P3 High resolution microscopy and spatial correlation analysis applied to dairy gels

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The characterization of dairy gels with high spatial resolution combined with tailor made image analysis algorithms are strong tools for understanding the microstructure of dairy products and optimizing processing conditions. Here, we provide an overview of recent results obtained on cheese gels made from reconstituted powders.

Stimulated Emission Depletion (STED) microscopy has been used to image dairy gels, where protein structures have been resolved to under 100 nm using an imaging protocol, that aside from dye addition is non-perturbative. Quantitative image analysis has been developed using an empirically validated model to extract the size of protein domains, inter-pore distance and fractal dimension of the protein network. These three parameters can discriminate between gels produced from fresh or reconstituted milk and between gels induced by acidification or renneting. In two colour images fat droplet size can be extracted and the distance between fat droplets and local maximum protein distribution can be determined. The distance between fat and protein changes significantly following homogenisation. The distribution and quantity of protein around individual fat droplets can be determined with region specific analysis. Coherent Anti-Stokes Raman Scattering (CARS) microscopy provides a label free negative control for the use of a fluorescent dye in STED imaging. Fluorescence time-life imaging provides spatially resolved micro-viscosity measurements within dairy gels and can be correlated to the different levels of moisture binding in gels produced from milks with different thermal processing histories.


Cryogenic applications in food industry

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Novel freezing technologies have been proposed for commercial use in the food industry [1]. However, it is challenging to access their benefits since there is a plethora of different frozen food products with varying size and composition. Additionally, the need for applying safety regulations (from ‘field to fork’ [2]) and get acceptance of the proposed technique from the consumers is imperative.

Cryogenics has numerous applications, ranging from space technology to biology, being also an established technology in the food industry. Cryogenics describes the science and technology dealing with the behaviour of materials at very low temperatures and should drive the development of new ideas that can lead to practical purposes [3]. In the food sector, cryogenic freezing systems are used to preserve food, extend its self-life, protect its size, texture and quality, and retain its flavour and aromas (enhance its organoleptic properties). It also shows substantial operational benefits in the industry by reducing costs and showing a higher throughput [1].

Not all food is suitable for freezing as it can cause physical and chemical changes. Common foods suitable for cryogenic freezing are those that contain considerable amounts of water, i.e., meat, poultry, seafood, vegetables and dough [4]. The main aim of the freezing process is to control how and where ice crystals are formed and it is generally accepted that fast freezing rates produce more and smaller ice crystals. Therefore, any physical damage to the structure of the food is reduced. However, there are arguments in literature with regards to the benefits in the organoleptic quality of the products (e.g., for meat products) [4].

The understanding of physical phenomena such as heat and mass transfer, alongside ice formation during the freezing process is essential. Whether the freezing rate shows advantages in new products and how much it affects their quality and organoleptic characteristics, is an area of interest. Lancaster University has successfully commissioned the production of a ColdBox, which is a big vessel that uses liquid nitrogen to cool down large products (down to approximately 100K without submerging into liquid nitrogen, with a cooling rate of 60K/hour). The ColdBox is fitted with monitoring equipment for observing the process in real-time. The benefit of monitoring the properties of large products or big batches of products is unique. The ColdBox is part of the proposed Lancaster Multipurpose Cryogenic Facility that aims to enhance the research activities between industry and academia.

High speed, simultaneous absorbance, transmission and fluorescence excitation emission spectroscopy (A-TEEM) makes complex food analysis practical and quantitative

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It has long been recognized that fluorescence and in particular excitation-emission matrices have an important part to play in the analysis of changes in foods during storage [1]. However, traditional measurement techniques have been slow, so techniques such as synchronous scanning have been used to get limited information in an acceptable timescale. HORIBA has recently introduced a patented [2] measurement technique that not only collects the full excitation-emission matrix but it also simultaneously records absorbance-transmission data, all within 30 seconds. The absorbance data is used to correct for the inner filter effect in the fluorescence data resulting NIST-traceable A-TEEM fingerprints that can be evaluated using multivariate statistics such as PARAFAC (Parallel Factor Analysis), PCA (Principal Components Analysis), CLS (Classical least Squares) and PLS (Partial least Squares Analysis).

Two specific application examples will be presented

Classification and phenolics analysis of red wines. Of the hundreds of different compounds that have been identified in grapes, it is the phenolic content of ripening grape berries that fundamentally determines the quality of a wine [3]. The A-TEEM data can be used to evaluate lot-to-lot, regional, and varietal characteristics. (See Figure 1). CLS analysis yields the relative contribution of 9 phenolic compounds.

Changes with time due to oxidation will also be shown.

Additionally, A-TEEM is also shown to easily distinguish between Extra Virgin Olive Oil brands, varietals, and provides a quantitative evaluation of blends using 3-D PARAFAC and PLS chemometric analysis.

Figure 1: An array of A_TEEM molecular fingerprints of various wines

Ultrasound Reflection Spectroscopy (URS) is a low power (<10 W/m²), non-destructive, non-material altering technique for the characterisation of food grade materials. URS has been used previously to investigate phase changes in aqueous and non-aqueous crystal systems. These measurements utilised measurements in time, with acoustic attenuation of a sample sound pulse coupled with the speed of sound of that pulse to determine parameters such as cloud point, clear point and melting point.

Another mode of measurement for URS is to measure the power spectrum of the target pulse, giving valuable information on how the frequencies within the target pulse are changing over time, temperature or material composition. A change in characteristics of the propagation medium, such as the occurrence of a phase change, should be reflected in the outputted power spectrum output due to scattering effects. Fast Fourier Transform (FFT) is used to determine the power spectrum.

URS and specifically power spectrum measurements offer a powerful tool in the investigation of phase transitions, there is no need for dilution of sample, and ultrasound techniques are economically viable.
Novel powdered coffee creamers were formulated using rice bran oil (17 wt.%) produced from a specific Thai rice variety (Khao Dawk Mali 105 or Hom Mali) as a dairy fat replacement with 50 wt.% fat reduction. The creamers were produced via spray drying (SD) and hot air (HA) methods. SD was found to be poorly soluble and it formed large aggregates when dispersed in water (2 wt. % solution) at room temperature (RT) or high temperature (at 80°C), unlike HA. This study aimed to determine the microstructure and physical properties of these powders and to underpin the root cause of the dissolution problem in SD when dispersed in water. The SD powder is more ‘airy’, it has lower bulk density than HA (ca. 0.41 vs. 0.65 g/ml, respectively). There was no difference in particle sizes of the hydrated HA and SD when dispersed in water at RT, i.e. d32 were < 10 μm as measured via Mastersizer. CLSM was employed to locate the oil droplets and the water-soluble constituents and the results showed a clear distinction between SD and HA powders at microstructural level (Figure 1). The oil droplets in SD tend to cluster together within themselves and forming hydrophobic patches, while in HA the oil droplets are integrated into the water-soluble of proteins and/or starch matrices. In addition, the SD powder showed presence of large vacuoles of occluded air (possibly formed during the atomization) with oil droplets adsorbed at the A/W interface. With the presence of hydrophobic patches and occluded air covered with oil droplets in the SD powder, these may explain the underlying causes of its poor dissolution behaviour.

Fig 1: CLSM images of hot-air drying (HA) vs spray dried (SD) reduced fat powdered coffee creamers at 20x magnification, stained with Nile Red and Fast Green to highlight the oil droplets (red) and protein and/or starch (green), respectively. The scale bar represents 50 μm length
The mixing of a powder of 10- to 50-μm primary particles into a liquid to form a dispersion with the highest possible solid content is a common industrial operation. Building on recent advances in the rheology of such “granular dispersions,” we study a paradigmatic example of such powder incorporation: the conching of chocolate, in which a homogeneous, flowing suspension is prepared from an inhomogeneous mixture of particulates, triglyceride oil, and dispersants. Studying the rheology of a simplified formulation, we find that the input of mechanical energy and staged addition of surfactants combine to effect a considerable shift in the jamming volume fraction of the system, thus increasing the maximum flowable solid content. We discuss the possible microscopic origins of this shift, and suggest that chocolate conching exemplifies a ubiquitous class of powder-liquid mixing.