RSC INTEREST GROUP
FORMULATION SCIENCE & TECHNOLOGY

RSC INTEREST GROUP
PARTICLE CHARACTERISATION

Particle Dispersion in Liquid Formulation

Wednesday 16 December 2020

Online Meeting

Conference Booklet

Please visit the RSC-FST website at: http://formulation.org.uk/
Please visit the RSC-PC website at: http://my.rsc.org/groups/home/118
Please contact the RSC-FST: secetary@formulation.org.uk

Welcome to Particle Dispersion in Liquid Formulation

The Formulation Science & Technology (RSC-FST) and Particle Characterisation (RSC-PC) interest groups of the Royal Society of Chemistry are organising a conference this joint event. The RSC-FST is the leading scientific organisation dedicated to product formulation and acts as a community for the exchange of knowledge in formulation across many scientific disciplines and industrial applications, including pharmaceuticals, cosmetics, foods and detergents. The RSC-PC provides a forum for those with a common interest in ways to dimensionally characterise single or assemblies of particles, in the solids handling, solids fabrication and general solid particulate industries.

As a charitable organisation, both interest groups work for the benefits of their members and further the awareness of the wider chemical science community. They organise many events during the year, including conferences, training days, and networking events. This year we have moved to a virtual platform for dissemination. This has allowed for a wider global reach – please take advantage to network with the formulation and particle characterisation community from across the world during the meeting.

This meeting is based on the premise that for many liquid formulations which include particles, the final state of the particles is not just determined by classic colloid science particle dispersion considerations, but also by a number of other factors. Rarely are fluid flows so intense in liquid formulations that primary particles are broken up. The initial state of agglomeration of multiple primaries, their break-up in the fluid flow and potential coagulation through process, storage and application are all important in determining the final state of the formulated product and thus its properties. For coatings and inks they determine the final colour / opacity and other functional properties, for food they influence mouth feel, for cosmetics they influence skin feel, for drugs they influence efficacy etc. Our talks will focus on particle dispersion across formulation lifetime and we will have poster presentations on formulation processes, particle characterisation and the role of particle dispersion in industry.

Our meeting will have three themes:

- Particle dispersion - thermodynamics - colloid science - overview talk
- Particle dispersion - kinetics - particles in hydrodynamics - topic talks
- Particle characterisation - both current methods and new improved approaches - 3 talks focus on different stages in manufacture

Thank you for your participation. We hope you find the day both enjoyable and informative.

Organising Committee

Dr Simon Gibbon, AkzoNobel RD&I, UK

Simon is Secretary of the Formulation Science and Technology Group, he has an appreciation of the challenges of successful formulation, having spent over 30 years working on formulations from non-stick cookware, through personal care and cleaning, to oil recovery. He is currently Corrosion Protection Community of Practice Leader for AkzoNobel, responsible for knowledge management across the company, where advances in characterisation provide the mechanistic understanding which drive the continually improving corrosion protection formulations.

Dr Phil Gill, Cranfield University, UK

Phil is a committee member of the Formulation Science and Technology Group, and is a physical chemist specialising in characterising and formulation of solid propellant chemistry. He is a nationally recognised expert in the field of energetic materials with over 20 years' experience. His research team is focused on the life assessment, manufacture and formulation of solid propellants. Current research topics include: (i) resonant acoustic mixing (RAM) to formulate the next generation composite rocket propellants; (ii) understanding the swelling and gelling behaviour of nitrocellulose for an improved understanding of min-smoke propellants.

Sam Peel, International Flavors & Fragrances, The Netherlands

Sam is a committee member of the Formulation Science and Technology Group. He has a Masters' degree in Formulation Science from Greenwich and an undergraduate degree in Chemistry from Warwick. He lives in The Netherlands, where he currently works for International Flavors & Fragrances.

Dr Claudio Pereira da Fonte, The University of Manchester, UK

Claudio is a Lecturer at the Department of Chemical Engineering and Analytical Science of The University of Manchester. His research interests are on the fundamental understanding of flow and transport phenomena in industrial mixing processes, with special focus on operations that involve rheologically complex liquids exhibiting high viscosity, elasticity or solid-like properties (such as shampoo, hair conditioners, mayonnaise, etc.). His research applies a combination of theory, and advanced computational (CFD) and experimental techniques (PIV, PLIF, high-speed shadowgraphy) to develop new processes and to deliver practical solutions to industrial mixing challenges.

Dr Helen Ryder, The University of Manchester, UK

Helen is the Chair for the Formulation Science and Technology Group. Helen obtained her PhD at The University of Manchester working on 'The Behaviour of Surfactant Lamellar and Gel Phases Under Flow'. She is currently a Research and Facilities Manager for Henry Royce Institute, the UK's National Centre for Advanced Materials. Helen specifically supports the extensive and innovative imaging and characterisation facilities at The University of Manchester.

Dr Stephen Ward-Smith, Malvern Panalytical, UK

Steve has worked for Malvern Panalytical for 25 years, and is currently one of the segment leaders in the Pharma and Food area looking after the regulatory pharma, food, agrochemical and personal care areas. He is also chair of ISO TC281 in fine bubble technology and an expert and convenor in ISO TC24 SC4 in sizing methods. He has a BSc from the University of Manchester in Chemistry, a MSc in Biomolecular Technology from the Leicester University and a PhD in Chemistry from the University of Nottingham.

Programme (all times UK GMT)

LOCATION: Online – Zoom/YouTube Meeting	
09:45-10:00	**Please join the online event either Zoom or YouTube** – details in joining email
10:00-10:10	**Welcome - Dr Simon Gibbon**
Session 1 – Dr Simon Gibbon (Chair)	
10:10-11:10	Industrial Practices & Challenges Posters – **watch presentations online now –** facilitated by Dr Simon Gibbon
11:10-12:10	Equipment Supplier Posters **- watch presentations online now –** facilitated by Dr Stephen Ward-Smith
12:10-12:55	**Theme 1 – Particle dispersion – thermodynamics – colloid science** **Talk 1 –** Particle Dispersion for Formulators**,** Professor Steven Abbot, Steven Abbott TCNF Ltd
12:55-13:25	**Formulation Science and Technology Interest Group AGM** Dr Helen Ryder, RSC-FST Chair
13:25-14:00	**LUNCH BREAK**
Session 2 – Dr Claudio Fonte (Chair)	
14:00-14:30	**Theme 2 – Particle dispersion – kinetics – particles in hydrodynamics** **Talk 2.1 –** An investigation of the performance of mixing systems for viscous solid-liquid mixing using CFD-DEM Professor Bruno Blais, Polytechnique Montréal
14:30-15:00	**Talk 2.2 –** Dispersion of Fine Powders in Liquids Dr N. Gul Özcan-Taşkın, Loughborough University
Session 3 – Dr Helen Ryder (Chair)	
15:00-15:20	**Theme 3 – Particle characterisation – both current methods and new improved approaches** **Talk 3.1 –** Particle characterization in formulation, pharmaceutical particulate material quality control Dr Richard J Tweedie, Particulate Sensors Ltd
15:20-15:40	**Talk 3.2 –** Breaking from the laboratory – On line and at line characterisation of particles Dr Stephen Ward-Smith, Malvern Panalytical
15:40-16:00	**Talk 3.3 –** Combining Methods for Particle Size Dispersion Measurement in a Research and Development Environment Dr Kerstin Jurkschat, OMCS, University of Oxford
Session 4 – Dr Simon Gibbon (Chair)	
16:10-16:40	**Integrated Examples - "beginning to end"** **Talk 4.1 – Material 1 –** Sustainable mineral/ microfibrillated cellulose composite formulation additives: Properties and uses Dr David Skuse, FiberLean Technologies Limited
16:40-17:10	**Talk 4.2 – Material 2 –** From particles to an ice-repellent coating Dr Emily Haas, Promethean Particles
17:10-17:30	**Summary and close: Session chairs**

Abstracts - Industrial Practices & Challenges Posters (10:10-11:10)

Industrial Poster 1 - EXAMPLE PROCESS CHALLENGES IN NANOSUSPENSION FORMULATIONS AND APPLICATIONS

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Specialty inks and some niche pharma formulations can be presented in the form of suspensions of nano particles. When they are prepared in a top down approach, larger size particles are milled to the required size using appropriate energy input, in the presence of surfactants for surface stabilization. Such suspensions are either used as is or subjected to downstream process steps for use in final applications. Characteristics like particle size distribution, shelf-life, and rheology impact their performance during the end-use. For a given chemistry, the preparation method of the suspension, batch sizes, and the conditions of the downstream process steps can significantly impact the performance of the finished products.

This poster discusses a couple of example challenges faced in a real-world scenario related to getting a reproducible nanosuspension product at one scale and scaling it up. It shows how structured approaches characterizing the materials, process, and understanding the underlying materials-process interactions, help address these challenges and achieve consistency in the finished product performance. Attention to such details during early stages of process development in an industry can result in significant cost-savings downstream.

Watch the 3-minute flash poster presentation here.

Industrial Poster 2 - TiO2 nanoparticle dispersions studied by sedimentation analysis: Hansen Parameters vs. DLVO interpretations

Lucie Delforce[1], Evamaria Hofmann[2], Véronique Nardello-Rataj[1], Jean-Marie Aubry[1]

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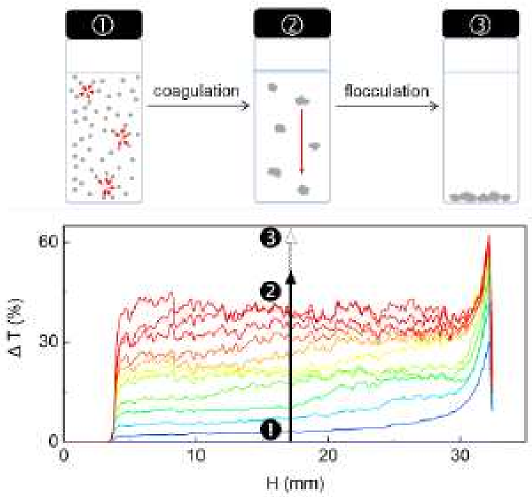
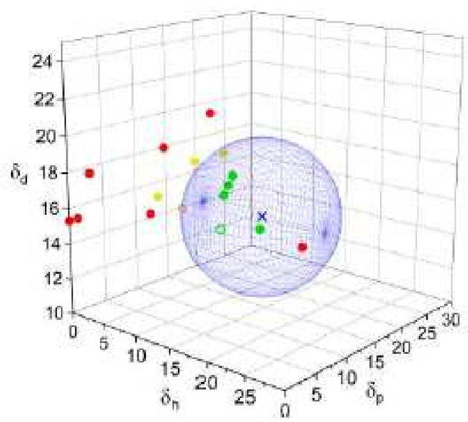
Titanium dioxide nanoparticles (TiO2 NPs) are among the most widely used NPs due to their interesting optical and catalytic properties. Most applications require their dispersion in fluid or solid matrixes. It is of first importance that NPs be and remain homogenously dispersed in the matrix to achieve optimal properties and stability. Hansen Dispersibility Parameters (HDP)[1] have been shown to be an effective approach for rationalizing and predicting the stability of various types of NP dispersions including TiO_2[1,2]. However, it is not excluded that interparticle electrostatic interactions, not considered in Hansen's approach, also play a significant role, especially for organic solvents having a notable dielectric constant[3–5]. In this work, we discuss the respective contributions of DLVO and non-DLVO interactions in the stability of TiO_2 P25 nanoparticles dispersions, with a special emphasis on the relevance of the HDP concept to rationalize non-DLVO interactions in organic solvents.

For that purpose, zeta potential measurements in aqueous media and in 20 organic solvents are carried out to identify dispersions for which the stability can be explained by the DLVO theory from those for which the stability results from more specific NP-solvent interaction. These latter solvents are used to build the Hansen Dispersibility Sphere of TiO_2 P25 with a Turbiscan as stability analyser. This light scattering method provides detailed information regarding the destabilization mechanisms of dispersions i.e. agglomeration and flocculation of NPs, which reflect the balance between interparticle attraction and electrostatic repulsion[6–8].



Transmitted light monitoring showing TiO_2 coagulation



TiO_2 P25 Hansen Dispersibility sphere

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(4) Lyklema, J. Principles of Interactions in Non-Aqueous Electrolyte Solutions. Curr. Opin. Colloid Interface Sci. 2013, 18 (2), 116–128.

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(6) Woo, S. H.; Min Gu, L.; Rhee, C. K. Sedimentation Properties of TiO 2 Nanoparticles in Organic Solvents. Solid State Phenom. 2007, 119, 267–270.

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Watch the 3-minute flash poster presentation here.

Industrial Poster 3 - The Importance of Dilution Process and Solvent Selection on Particle Size Measurements

Margaret Hwang, Jyo Lyn Hor, David Adrian

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Dispersion of solids drives the product quality for filled materials such as liquid silicone rubber (LSR) and antifoam (AF) compounds; both are made by blending silica into a silicone matrix. An important indicator of filler incorporation is particle size. However, when using laser diffraction to size particles in diluted samples, the selection of solvent and the dilution process can affect the apparent particle size of silica. In this presentation, we highlight some considerations for selecting a suitable solvent for measuring silica particles in dilute solution. Because sufficiently high shear stress may break down agglomerates and lead to smaller apparent particle sizes, physical properties to consider include the viscosity of the solvent and the shear stress imposed on the system during the dilution process. On the other hand, interactions between the solvent and the particles or between the solvent and the matrix can either lead to agglomeration or further dispersion of particles. Therefore, when a product is changed significantly, it is necessary to evaluate whether the current dilution and solvent combination is able to capture those changes.

Watch the 3-minute flash poster presentation here

Industrial Poster 4 - Challenges of assessing number-based particle concentration and the development of a quantitative method using DCS

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Number-based particle concentration is an important attribute of industrial particle formulations which enable assessment of product batch-to-batch variability, and measurement of dosage for therapeutic and medical applications. However, its measurement accuracy and precision remain a challenge to industry due to the lack of relevant reference materials and standards. Collaboration between OxSonics and NPL has resulted in the development of a method to measure number-based particle concentration using Differential Centrifugal Sedimentation (DCS), with lower variability than reported by Particle Tracking Analysis (PTA).

Watch the 3-minute flash poster presentation here

Industrial Poster 5 - Osmotic swelling as a key for perfect nanosheets dispersion: From water to organic solvents

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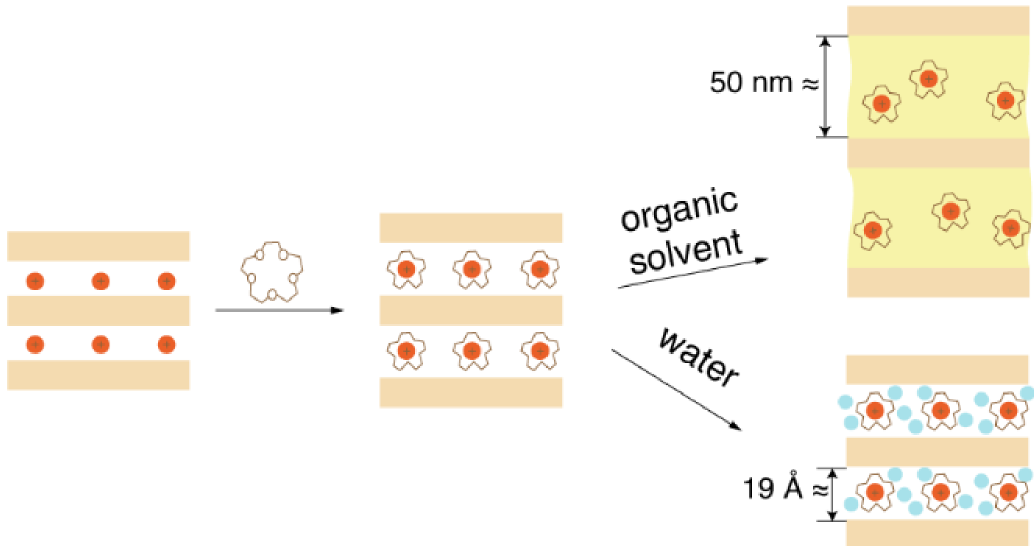
Layered silicates have a wide range of applications due to their unique platelet like structure, high aspect ratio, and natural abundance. Particularly in nanocomposites, where silicates improve mechanical properties, decrease gas permeability and significantly enhance flame retardancy and thermal properties. In order to obtain these desirable properties, the delamination into single layers is essential; otherwise, the obtained composite exhibits micro-composite structure with two separate phases without a synergistic enhancement in properties.

The conventional method of liquid-phase exfoliation are not suitable for the fabrication of high aspect ratio filler due to high shear force exceeded on the clay particles, which leads to the diminishing of the aspect ratio. Osmotic swelling is an alternative method, which allows a gentle thermodynamically allowed delamination with the preservation of aspect ratio of pristine mineral. So far the osmotic swelling were limited to the water systems and the range of applications would be further expanded if clay delamination could be achieved in non-aqueous solvents. For this purpose, so-called "organophilic" clays have previously been developed but, quite often, the materials thus prepared show poor delamination.

We are going to present the straightforward methodology to achieve perfect delamination to the single platelets in organic solvents and water making possible numerous new formulations. The resulting dispersion is infinitely stable due to electrostatic stabilisation and can be processed with numerous industrial methods including slot die and spray coatings.



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Industrial Poster 6 - Highly Dispersible Kaolin for Printing Inks

Anabelle Elton-Legrix, Andrew Curtis,
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The inherent hydrophilic nature of kaolin particles make them well suited for incorporation into water borne liquid systems, but it is also of interest to disperse fine kaolin particles for solvent based applications, in particular non polar and oil based offset inks. Here we focus on using fine platy kaolins (with median particle size down to 0.2 micron by sedimentation) in heatset inks used for printing high gloss magazines. The fine kaolin particles need to be well dispersed with absence of aggregates or agglomerates in order to reduce the surface roughness of the printed ink layer and maintain high final printing gloss (1).

Mechanical solutions including suitable grinding and milling of fine kaolin particles are commonly used in the minerals industry, allowing easier dispersion of kaolin particles to a suitable and acceptable fine size and reduced aggregates when incorporated in the oil or solvent. However, agglomerates can still be present due to the incompatibility of surface chemistry between the hydrophilic kaolin and the oleophilic dispersion medium. Suitable surface treatments can be used to increase the compatibility of the kaolin surface in oil and previous literature studies (for example 2) have shown promising results using long chain amines and polyisobutylene-based stabiliser with a succimide/amine head group for dispersion in non polar solvents.

Here we have initially used a simple and effective sedimentation test in the ink petroleum distillate to allow screening of a wider range of amine and silane chemistries, with the aim to find the optimum dosage and treatment for effective dispersion of particles and sedimentation of treated kaolin into a hard compact layer. The most promising treated kaolins were incorporated into the full ink varnish (based on phenolic modified rosin, alkyd, veg oil and mineral distillate) up to 15wt% addition and printed onto polyester film to measure print gloss compared to an unfilled ink varnish. High print gloss was confirmed for well dispersed ultrafine kaolins and examination of the printed surfaces using Scanning Electron Microscopy tilted images showed a significant reduction in surface micro-roughness. Full pigmented inks also gave better dispersion on the rollers of a triple roll mill and high print gloss at high kaolin content. Work is ongoing to consider the health and safety hazard of the treatments, minimise the viscosity for ease of addition onto dry kaolin and optimise the cost performance of the treated kaolin by considering blends of long chain and bulky amines as suggested in publications on other minerals (for example 3).

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Watch the 3-minute flash poster presentation here

Industrial Poster 7 - Powder Incorporation into Liquids using Ytron ZC1

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The introduction of nanoparticles in new formulations has resulted in enhanced product performance or entirely new product properties that cannot be achieved otherwise. Adhesives, coatings, packaging, catalysts, paints and inks, sunscreens are a few examples. The first step in the production of final or intermediate products in the form of nanoparticulate dispersions is the incorporation of a dry powder into a liquid, which is often performed using a stirred tank [1]. Previous work on powder incorporation has therefore focused on stirred tanks, with only one study which made use of a batch rotor-stator[2]. Incorporation is then followed by a deagglomeration stage with power-intensive devices, such as inline rotor-stators, which are commonly used for large scale manufacture of such products.

In this project, the powder dispersion performance of an in-line rotor-stator, Ytron ZC1, has been studied. The specific design of Ytron ZC1 allows the introduction of the powder directly into the rotor-stator head; subsequently the dispersion can be recirculated to achieve deagglomeration. The part of the study presented here is concerned with powder incorporation with the objective of establishing the performance of the device. Concentrations up to 10% w:w were studied by making incremental additions of 1% w:w silica powder into water, over a range of flow rates. Air velocity values at the powder inlet were also measured.

For a given rotor-stator head and speed, there exists a range of liquid flow rates over which the incorporation rate is highest. Initially, increasing the flow rate increases the incorporation rate and above an optimum range, incorporation slows down and eventually ceases. This is due to the liquid starting to flow up the powder inlet which can have severe consequences if the partly wetted powder forms a soft solid, blocking the powder inlet as this would require stopping the operation to dismantle and clean.

It was shown that using the Ytron ZC1, the incorporation rate can be maintained constant over a wide range of powder concentrations compared to surface additions (Figure 1). This is in agreement with previous findings reported with Ytron Y-Jet, a batch rotor-stator and holds as long as the increased concentration does not result in non-Newtonian rheology.

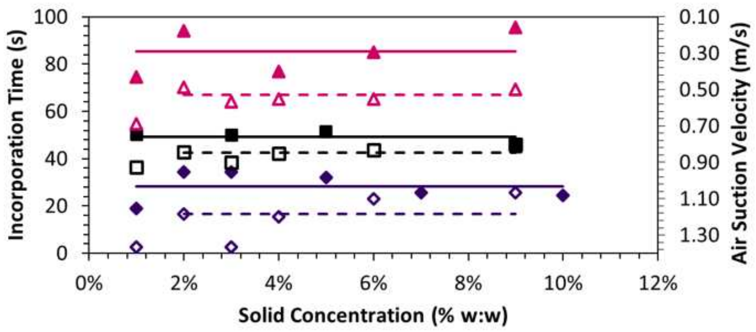

Figure 1 Effect of powder concentration and liquid flow rate on incorporation rate

♦:1.89 l s-1; ■: 2.28 l s-1; ▲:2.60 l s-1

It could be shown that the air suction velocity at the powder inlet is indicative of powder incorporation performance. This would be of significant benefit for industrial practice as trials can be run with just liquids to identify optimum operating conditions, saving time and costs associated with materials and waste.

References:

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Watch the 3-minute flash poster presentation here

Industrial Poster 8 - The importance of particle dispersions in paint formulations and the need for better characterization methods

Dr David Elliott

AkzoNobel Decorative Paints, Slough, UK

Paints comprise dispersed particles of several kinds having wide ranges of particle size from tens of nanometres to hundreds of microns and a variety of shapes and aspect ratios. These include polymer latex particles and Titanium Dioxide white pigment at the smaller (colloidal) size end of the range, with near spherical shapes and mineral extender particles with largest particles of more than 100 microns and with diverse shapes from blocks to fibres and plates. Much R&D effort in the paint industry is spent on formulating to achieve stability of these complex dispersions, so that long product shelf-lives can be offered and so colours can be made accurately and reproducibly.

Formulating work and problem solving usually takes the form of extensive designed experimentation and time-consuming stability testing. Attempts to gain deeper understanding and use more fundamental scientific approaches are hindered by the limitations of current characterisation methods, when it comes to complex, multi-modal, concentrated dispersions like paints, which typically have particle volume concentrations in the range of 20 to 50%.

Watch the 3-minute flash poster presentation here

Industrial Poster 9 - Particle incorporation in liquid – P&G's perspective

Ke-ming Quan

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Many P&G formulated products require particle incorporation in liquid. The quality of incorporation directly impacts product performance and stability. At present, process development for particle incorporation is conducted empirically. There is significant challenge to scale lab and pilot plant learnings to production.

A formulation in process typically has a mixture of particles, drops and gas bubbles. It is highly desired to have breakthroughs in inline measurement to enable multiphase particle size (distribution) characterization. At the same time, fundamental and/or semi-empirical models are also needed to guide process optimization and scalability. An example of particle incorporation will be given to show how measure and model can help to solve an industrial problem.

Watch the 3-minute flash poster presentation here

Industrial Poster 10 - A Statistical Approach to Ink Formulation

Anna-Marie Stobo, David Johnson, Lynn Donlon, Rachel Findlay, Jodie Clark, Adam Todd, Ross Urquhart, Martin Sondergaard

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LEE-BED is a Horizon 2020 project that aims to reduce development time of materials and process for the production of hybrid ele ctronics. CPI-Formulation's focus is on ink, adhesive and composite formulation, while CPI-Electronics are optimising testing procedures for roll-2-roll printed components. DTI is working on scaling up the synthesis of a range of nanoparticles, including copper nanoparticles. Other partners include Fraunhofer, TNO, ITENE, RISE, VSParticles, TPU, Swarovski, Maier, Acceona and Graphetic.

Watch the 3-minute flash poster presentation here

Abstracts - Equipment Supplier Posters (11:10-12:10)

Equipment Supplier Poster 1: Static Multiple Light Scattering for Monitoring the Stability and Particle Size of Concentrated Dispersions

Gordon Irvine[1], C. Tisserand[2], R. Ramsch[2], J. Cohen[2], P. Bru[1], G. Meunier[2]

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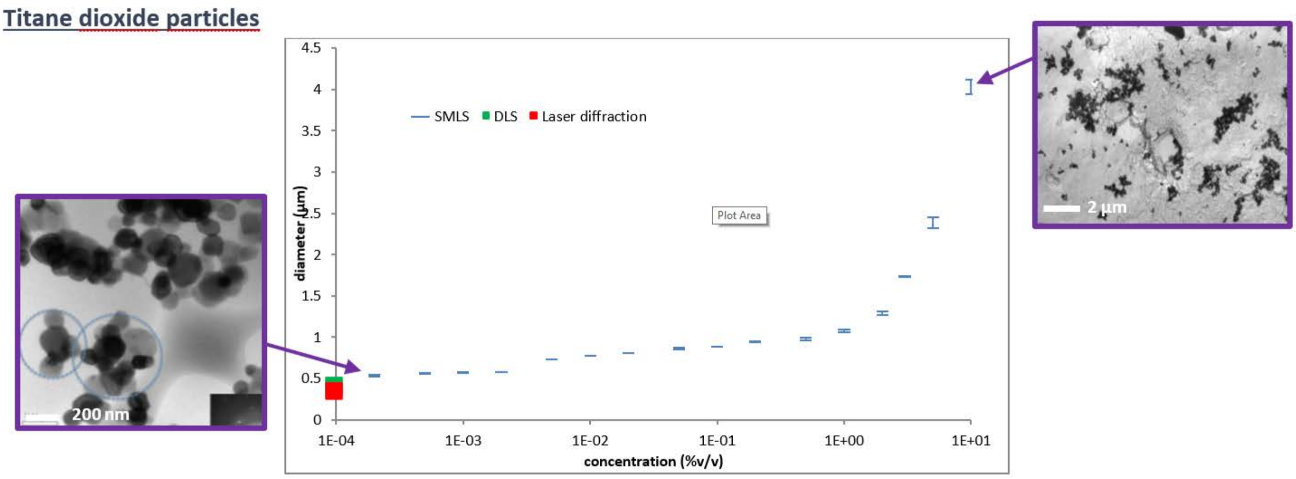
Characterization of colloidal systems and investigation of their stability and size evolution in their native state (i.e. without denaturation) is of prime importance for formulators to optimize the development of new products.

The Static Multiple Light Scattering (SMLS) technique utilized by the Turbiscan Lab instrument can monitor physical stability and measure mean particle size in concentrated dispersions between 0.0001 and 95 vol% for particle sizes between 10 nm and 100 µm. The Turbiscan uses an incident light source of 880 nm acquiring the backscattered and transmitted signal whose intensity is directly related to particle diameter and concentration. The signal intensity enables measurement of the mean spherical equivalent diameter when the refractive indices of continuous and dispersed phase as well as the particle concentration are known, according to the Mie theory.

This technique has the advantage to measure such properties in a single click without sample preparation or dilution the concentrated suspensions. Other optical techniques such as DLS or PTA can perform size measurement but only at a very high dilution which can denature agglomerates and give an erroneous size of the native particles. In this poster, we present a comprehensive outline of multiple studies where particle size is measured in concentrated dispersions. We also show how overall formulation stability, particle size, and particle migration kinetics can be determined from the same Turbiscan data set.



Mean size versus concentration (v/v) for titanium dioxide particles

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 2: Determination of Stability and Particle Size of Colloids in Lab or Online

André Giese, Kai Gossmann, Philipp Schreier, Margret Böck, Hanno Wachernig

Colloid Metrix GmbH, Meerbusch, Germany

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In addition to the final product properties, the main objective in the formulation of colloidal dispersions is the particle size and the stability against agglomeration.

The particle size is of great importance, but also their chemical environment.

For this purpose, the zeta potential, which is generally regarded as a measure of the mutual repulsion of the particles by electrostatic interactions, is a key parameter. The zeta potential and thus the stability of dispersions is influenced by the suspension properties. By autotitration, the formulator can investigate the influence of pH, salt concentration and additives on stability, and optimize the required quantities accordingly.

The determination of the particle surface charge is often also of great interest. It allows an estimation of free binding sites or adsorption capacity that can be used for the functionalization of the particles, be it for further stabilization against aging processes or for changing the final product properties.

This kind of characterization can be optimally performed with the streaming potential / streaming current method in combination with an autotitration, even at high particle concentrations. At such concentrations, the 180° heterodyne dynamic light scattering method is suitable for determining particle size, as it is less susceptible to multiple scattering effects.

On an industrial scale, inline particle size measurement can be interesting. The DLS method mentioned above can be extended to monitor top-down or bottom-up processes in real-time.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 3: Routine, ensemble characterisation of electrophoretic mobility in high and saturated ionic dispersions

Jake Austin, Diogo Fernandes, Matthew J. A. Ruszala, Natalie Hill, Jason CW Corbett

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With the industrialisation of nanoparticle manufacture, the pervasive incursion of nanoparticles into the environment, the need to characterise nano-scale pharmaceuticals and living systems in replicated in vivo conditions, the continuing development of new theories to describe the electro-kinetic behaviour of nano-particles in representative ionic strengths and numerous other applications, there is an urgent requirement to provide simple and effective experimental tools to validate these models and explore new systems. Micro-electrophoresis implemented with a diffusion barrier, which isolates the dispersed phase from the electrode surface, is demonstrated as enabling such measurements for the first time, preventing the catastrophic outgassing, precipitation and sample degradation observed when the dispersed phase is in close proximity to the electrode surface. Using a measurement of a few minute's duration in a standard laboratory light scattering instrument we reproduce the theoretically predicted phenomena of asymptotic, non-zero electrophoretic mobility with increasing ionic strength, the cationic Hofmeister series dependency, charge inversion and a continuously decreasing variation in mobility with pH as molarity increases.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 4: Colloidal Refractometry for Microstructure Characterization of Particle Dispersions

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This poster introduces a new structural probe for colloidal liquids in their neat unperturbed state. The technique combines two recent advances in refractometry - a generalised equation for the refractive index of particulate systems which contains the size and the volume concentration of the dispersed phase and the advent of digital refractometers which measure the refractive index of turbid dispersions with accuracy. Examples of its application include study of fat globules in milk, coagulation of polymer latex and lamellar dispersions.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 5: Vapour Sorption Techniques for Particle Engineering

Sabiyah Ahmed[1], Meishan Guo[1], Majid Naderi[1], Manaswini Acharya[1], and Daniel Burnett[2]

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Drug substances intended for drug delivery to the lungs typically require particle size reduction. High energy processes are typically utilized to produce particles smaller than 10 µm but these processes are also known to influence crystallinity, which can lead to a reduction in physical and sometimes even chemical stability. Therefore, these materials may be conditioned following micronization before further processing. Such a “deamorphization” step typically involves the treatment of the materials with an appropriate solvent that plasticizes the material and induces crystallization. While the selection of the solvent is critical, the degree of control over the deamorphization process is also very important. A treatment time that is too short may lead to incomplete crystallization of the material, while overtreatment may cause partial dissolution and agglomeration.

In order to estimate the appropriate treatment time, knowledge of the crystallization kinetics may be predicted from Dynamic Vapour Sorption (DVS) studies. Changes in surface chemistry; from a heterogeneous surface property to a homogeneous and low wettability surface property would affect the variability in powder flow behavior and agglomeration which may be monitored using Inverse Gas Chromatography Surface Energy Analyser (IGC-SEA).

Water vapor has been shown to induce crystallization for milled salbutamol sulphate, with one-step mechanism. DVS can be used to investigate moisture-induced crystallization kinetics, over a wide range of temperature and humidity conditions.

Silanisation process has clearly improved the flow properties of D-mannitol in a low-stress environment. iGC-SEA is a fast and accurate way to predict agglomeration and powder flow behavior, using surface energy heterogeneity and work of cohesion as the parameters.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 6: Application of NMR Relaxation to Determine the Hansen Solubility Parameter (HSP) of Particles

Ravi Sharma[1], Shin-Ichi Takeda[2] Dave Fairhurst[3], Stuart Prescott[4], Terence Cosgrove[5]

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[2] Colloid-Techno-Consultants,Ltd., Osaka, Japan
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[4] University of New South Wales, Australia
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The ease with which particles can be dispersed in a liquid is dependent on the details of the liquid to be used and the surface chemistry of the particles. The particle wettability, as controlled by dispersion forces, dipole-dipole forces and hydrogen bonding is the primary influence. Being able to predict the appropriate liquids to use for a given particle is of considerable interest to the formulator, and there has been much interest in semi-empirical routes to selecting the optimum solvent (or solvent mixture) for pigment particles.

The Hansen Solubility Parameters (HSP) approach, originally devised to study polymer-solvent compatibility, can be applied to solid materials. Determination of the particle HSP, traditionally by visual observation, is subjective, time-consuming, error-prone, and is only qualitative. Extinction profiles measured over centrifugation time are more quantitative; the influence of density and viscosity of different solvents employed must accounted for and the solids concentration must be chosen with care.

NMR relaxation measurements are sensitive to the same intermolecular forces between solvent and particles with which HSP are concerned. We will show relaxation data obtained using five different nano-size zinc oxide and aluminium oxide powders, each having a completely different surface chemistry, ranging from a high polar component (hydrophilic) to a lower polar component (hydrophobic).

Results suggest that a straightforward, quantitative, and fast instrumental approach to determining the HSP of a nanomaterial is feasible and, further, that NMR relaxation measurement can discriminate between suspensions that may initially appear similar but exhibiting different long-term colloidal stability. In addition NMR relaxation data may potentially be used as a method for quality control of particles. NMR relaxation measurements (made using a low cost, low field table-top device) can be made at almost any industrially relevant solids concentration without requiring further sample preparation; any hydrogen-containing solvent can be used.

The ability to project solid-liquid interactions obtained by NMR relaxation into Hansen space is powerful, is much simpler and easier than sedimentation and can potentially provide formulators with a time-saving method to optimize and select the liquid composition (solvent plus surfactant additives) for desired particle suspension performance.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 7: Preventing Crystallization with the Crystal16 for Successful Parenteral Formulations

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Many drug substances are formulated as parenteral formulations and administrated directly into the systemic circulation in animals and humans. In order to improve their stability and solubility, many parenteral formulations are formulated using a co-solvent. Ampule or vial dosage forms are often co-solvent concentrated formulations. These concentrates are finally diluted before administrated to the patient. Nevertheless, there are many cases known where dilution of drug formulations has the potential to generate conditions where drug concentrations are supersaturated. Under these circumstances, drug precipitation/crystallization is likely to take place. Drug precipitation after parenteral administration may cause mechanical or chemical irritation at the injection site, and potentially even more serious systemic effects. With the use of the Crystal16, solubility measurements may be performed to help avoid vulnerable formulations and foresee mechanical and chemical irritation at injection site. In the phase diagram of lorazepam was showed how high concentrations of the drug and low ratios of glucose lead to a stable solution in which crystallization does not occur.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 8: Shining a Light on Particle Characterisation at the Diamond Light Source

Claire Pizzey

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Diamond Light Source is the UK's national synchrotron science facility, located at the Harwell Science and Innovation Campus in Oxfordshire. Diamond generates brilliant beams of light from infra-red to X-rays which are used to investigate physical and chemical behaviour of materials and processes on the atomic, molecular and cellular scale. Clients range from multinationals to start-ups and SMEs working in sectors as diverse as drug discovery and development, food, electronics, formulation, energy, automotive and aerospace engineering. Investigations of microstructure of formulations are of particular interest to a variety of clients with small angle X-ray scattering among the available techniques used to non-destructively probe nanoparticle morphology and suspension microstructure. Case study examples of work done by Diamond's users and clients will be explored.

Watch the 3-minute flash poster presentation here

Equipment Supplier Poster 9: Use of a Closed Vial Milling System in Investigations of Carbon Black Ink Formulations

Stephen R Bysouth[1], Muhammad Ali[2]

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Experimentation in dispersion can be time consuming, messy, consume considerable amounts of materials and consequently, produce large volumes of waste. A product of the development of technology for high throughput experimentation is the Automaxion multi-sample milling system based on planetary milling, which facilitates bead milling in ordinary glass vials. It has been applied to particle size reduction of materials from refractories to pharmaceuticals, comminution of crop protectants, milling of heterogeneous catalysts, cocrystal screening, mechano-chemical preparation of MOF's and the dispersion of pigments in coatings and, as presented here, ink concentrates.

We will show that small samples of carbon black ink concentrates of about 10 ml can be prepared quickly and are suitably equivalent to larger samples prepared using a laboratory recirculating bead mill. Comparisons of particle size, zeta potential and rheology of samples prepared by both techniques are presented and their rheological stability over four weeks is compared.

Watch the 3-minute flash poster presentation here

Abstracts – Oral Presentations

12:10-12:55 - Particle Dispersion for Formulators

Professor Steven Abbott

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For too long, the particle formulation community has had to work with inadequate formulation tools. Things are now changing for two reasons. First, a lot of the knowledge is available in free apps, demonstrated live during the talks. Second, the community is realising that the solubility science approach is more powerful than the limited capabilities of classic “dispersion” or “colloid” science. We can now optimize the interactions of solvents, polymers and particles with tools such as Hansen Solubility Parameters and, in a recent development, with hands-on Scheutjens-Fleer theory. With the increading adoption of these new tools, the future for particle formulation science is looking very bright.

https://www.stevenabbott.co.uk

14:00-14:30 – An investigation of the performance of mixing systems for viscous solid-liquid mixing using CFD-DEM

Dr Bruno Blais, Department of Chemical Engineering, Polytechnique Montréal, Montréal. Canada

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Solid-liquid mixing is a significant process that is critical to pharmaceutical, biomechanical, food processing and other industries. Despite the relevance of viscous solid-liquid mixing applications, the physics governing the suspension of particles in agitated vessels is still poorly understood and historically much less studied in the laminar regime than in the turbulent regime. This can lead to sub-optimal designs and important energy waste or even an incapacity to achieve the desired mixing state. Important criteria that characterize solid-liquid mixing, like the just-suspended speed (Njs) and the homogeneity of the suspension, are difficult to predict. Consequently, engineers often rely on correlations to design solid-liquid mixing vessels. There is a need for more studies on solid-liquid mixing and, more particularly, for new and robust simulation models to predict suspension dynamics.

Complex geometries can be used to meet specific process requirements. For example, the double helical ribbon has shown to achieve good mixing in the laminar regime. However, the simulation of solid-liquid flow in such rotating systems is challenging. To deal with this issue, an innovative Euler-Lagrange model in a non-inertial frame of reference that is suitable to a wide range of geometries regardless of their complexity was developed. It is based on an unresolved CFD-DEM approach that combines the Discrete Element Method (DEM) for the solid particle dynamics and CFD techniques for the fluid phase [1].

First, the full model is introduced with a description of the coupling strategy for the fluid and the solid phases and the validation of the CFD-DEM model against experimental data and numerical studies previously carried in our lab [1,2], Then, using this model, several agitator geometries commonly used for viscous liquid, such as the double helical ribbon, the anchor or the Paravisc, are compared. This comparison is based on typical solid-liquid mixing parameter such as the minimum agitation speed required to obtain a complete suspension (Njs), the cloud height (CH) or the power number (Np). Finally, we conclude on the role of numerical modeling in investigating multiphase mixing.

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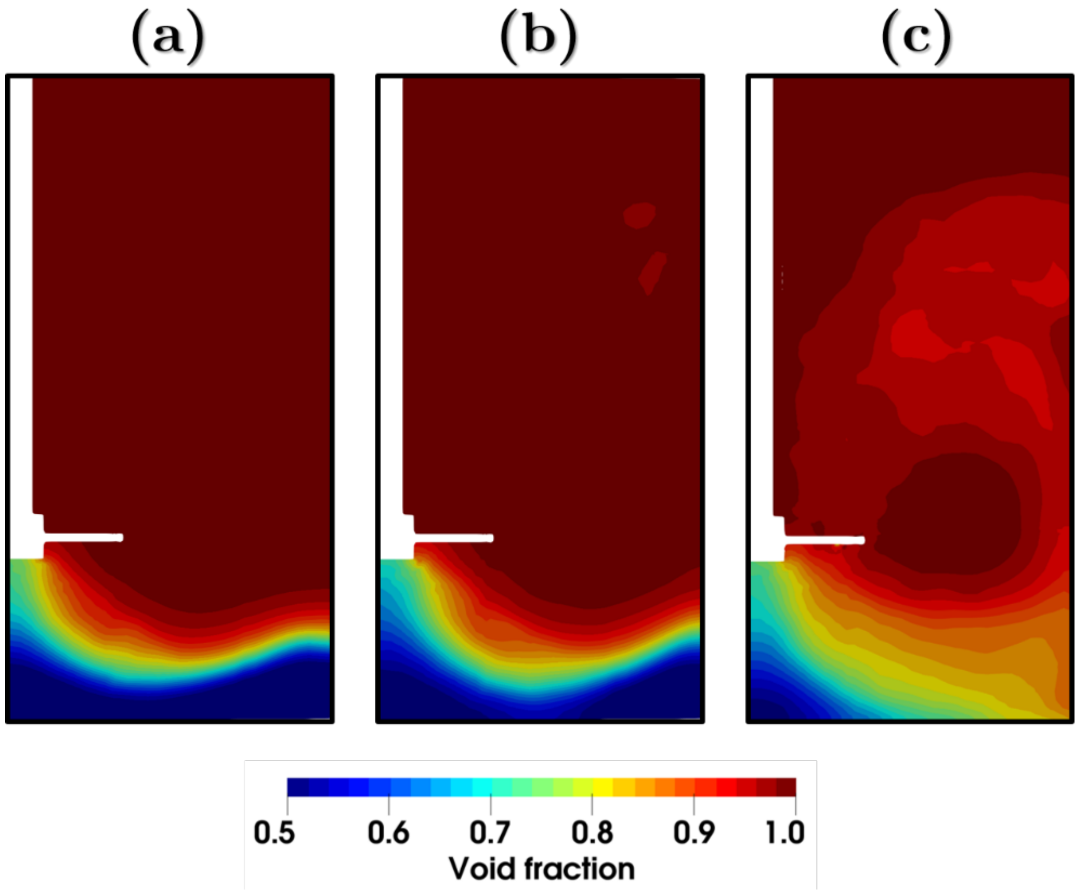

Figure 1 : Liquid volume fraction profile for a solid-liquid mixing operation agitated by a pitched blade turbine at an impeller velocity of (a) 250 RPM (b) 350 RPM (c) 450 RPM

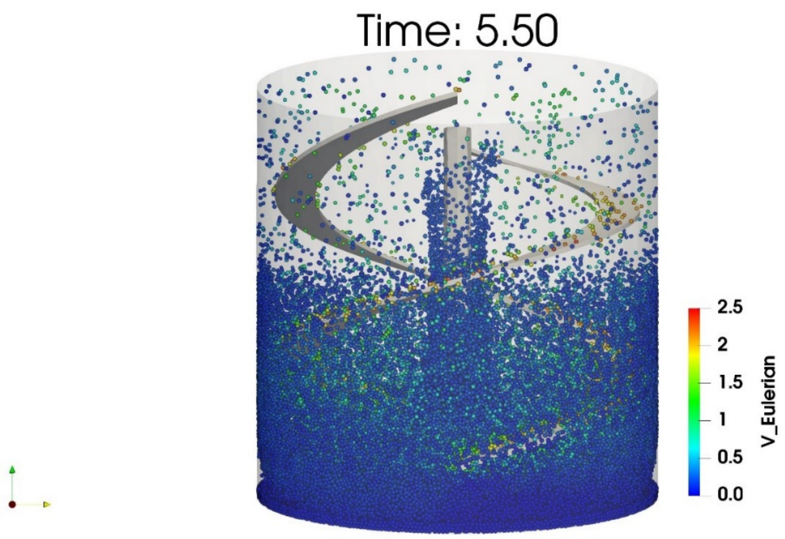

Figure 2 : Suspension of 3mm particles in a viscous fluid using a helical ribbon

14:30-15:00 - Dispersion of Fine Powders in Liquids

N. G. Özcan-Taşkın

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The formulation and hence manufacturing processes of several products- from pharmaceuticals to personal care, paints and inks to cosmetics, food to minerals and water treatment- require the incorporation of powders into liquids. In addition, the recent decades have seen the development of several novel, high performance products with nanoparticles in their formulation. Large scale manufacture of these to enable market introduction has often been a challenging task for process engineers and scientists.

Fine powders tend to form large clusters that float at the liquid surface due to apparent low density resulting from air entrapped within the agglomerates; poor wettability can also play a role(1). Following on from the incorporation of the powder into the liquid, deagglomeration is required to achieve as fine a dispersion as possible. This talk will briefly go through powder incorporation highlighting some recent advances and continue with findings relating to deagglomeration. A few brief comments will be added on the delamination of nanoclays. Outlines of a few case studies will also be presented.

Powder incorporation can be performed using a stirred tank which may also act as the feed tank for a more energy intensive device used to achieve deagglomeration. In addition to conventional impellers, proprietary design batch rotor-stators(2) or in-line rotor-stators(3) can also be used for the purpose. These allow the introduction of the powder into the regions of high liquid velocities and have been shown to be advantageous for formulations that have a powder concentration greater than a few % by weight.

Deagglomeration of fine powders can occur through different mechanisms: erosion of small fragments off the surface of agglomerates, rupture of large agglomerates that result in smaller agglomerates which are broken up further or through the shattering of an agglomerate into its finest fragments in a single event. The process can be monitored by particle size measurements and careful data analysis would indicate the dominant mechanism through of breakup(4,5), providing useful input in equipment choice/process design(6). Such measurements also indicate how fine a dispersion can be achieved. Whilst the mechanism of deagglomeration and dispersion fineness are primarily defined by the particular formulation, i.e. regardless of the choice of process equipment or operating conditions, the kinetics of deagglomeration depend on the flow field generated by the specific design and operation of an equipment. In addition to these, dispersion rheology, due to both the addition of powder and deagglomeration or delamination in the case of nanoclays can play an important role.

Knowledge from such studies has proven to be useful in process design and scale up, both of which are challenging tasks, especially considering the different devices available on the market(7,8) and the need to use different types of equipment from formulation to manufacturing scale. There has been significant progress made in the area with both experimental studies and numerical modelling. Further work continues and the author is keen to establish collaborations.

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15:00-15:20 – Particle characterization in formulation, pharmaceutical particulate material quality control

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The twin business imperatives of increasing competitiveness, and reducing risk are common to all manufacturers, if not more so in the pharmaceuticals. This translates into increasing supply chain diversity and product complexity, with a constant pressure on costs, while operating in a highly legislated, regulated environment.

Unit operations involving particulate operations are widespread in the processing of pharmaceuticals, even within the chemical sector; with up to 60% of processes involve particle processing. Therefore maintaining quality control through particle characterization of raw materials and intermediaries is a vital piece of delivering the business objectives.

Despite all the claims and all the hopes, in the last 20 years the capability to characterise particulate systems in-situ has not met with the original expectations. Hence the industry is still dependant on laboratory instrumentation.

As the industry has moved forward so have measurement techniques with improvements to existing methods and an increasing number of new methods aimed at 'Nano' material formulations These recent advances in particle characterization allow a much greater degree of understanding both distribution of particle characteristic and the individual properties such particle shape.

The various options available to make these measurements are discussed in relation to the information required.

15:20-15:40 – Breaking from the laboratory - On line and at line characterisation of particles

Dr Stephen Ward-Smith, MRSC
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Particle characterisation has been a long standing laboratory technique. It has been used at line and on line with varying degrees of success over the last 10 – 15 years. Recent instrument advances in such areas as high concentration DLS / DWS and image analysis, coupled with improved dilution / sampling regimes are increasing interest in this arena. Recent improvements in data and modelling are also important, as equipment which is in constant operation will generate more data than a laboratory instrument, so now the tools to interpret the data generated are also coming of age.

This talk wil survey what is current available and some of the do's and don'ts when it comes to investigating the purchase of on line equipment.

15:40-16:00: Combining Methods for Particle Size Dispersion Measurement in a Research and Development Environment

Dr Kerstin Jurkschat
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Although various methods are available to measure particle size distributions for a wide range of sizes and concentrations, most of them are best suited to relatively simple systems, i.e. one type of particle in a one or two component dispersion medium. However, real systems are often more complex containing different materials and shapes of particles and stretch the limits of commercially available instruments. In combination with imaging techniques and their analytical capabilities these instrument limits can be extended to gain a better understanding of the systems. To illustrate this, one example where the addition of TEM and EDX methods allowed the study of a complex, environmental sample will be presented.

16:10-16:40 – Sustainable mineral/ microfibrillated cellulose composite formulation additives: Properties and uses

David Skuse

FiberLean Technologies Limited, Par Moor Centre, Par Moor Road, Par, Cornwall, UK PL24 2SQ

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When pulp and minerals are co-processed in aqueous suspension, the mineral acts as a grinding aid allowing cost effective production of fibrils. Furthermore, this processing uses robust, industrial milling equipment. Mineral/ microfibrillated cellulose composite materials produced in this manner have been commercialised under the tradename, FiberLean® MFC. There are 12 000 dry metric tonnes of FiberLean® MFC fibril production capacity in operation, worldwide. Such composites can be prepared using a wide range of virgin and recycled pulp sources and a wide range of minerals, with choices dependent on the end formulation requirements.

The products can be used in a wide range of applications:

- As wet and dry strength additives to enhance the mechanical properties of web-based structures, such as paper, packaging, tiles and boards.
- To enable wet-end coating of optically effective, light weight coatings in graphic applications, in particular, as surfaces for ink-jet printing.
- As a component of grease, moisture and gas barrier systems.
- As the main constituent in products such as non-woven mats.
- As rheology modifiers, for example in paints and oil field formulations.
- As reinforcing additives in composites.

In this presentation we will describe the mineral/ microfibrillated cellulose products and demonstrate how the choice of minerals and fibres used in their preparation can affect the end-use properties.

16:40-17:10 – From particles to an ice-repellent coating

Emily Haas[1], Christina Puijk[1], Selina Ambrose[1], Angelo La Rosa[2], Geraldine Durand[2], Anna Wojdyla-Cieslak[3], Alan Taylor[3]

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Promethean Particles is a pioneer in the manufacture of high specification nanomaterials for a range of applications including pigments, printed electronics, energy capture and storage, functional nanoceramics and coatings.

The company has developed breakthrough innovations in nanoparticle technologies by designing safe and commercially viable liquid dispersions at its multi-material manufacturing plant. Located at Genesis Park, Nottingham, the site manufactures nanoparticles with tonnes-per-year capacities through a patented continuous flow process - making it the largest reactor system of its kind in the world.

Advancements in technology over the years has made nanoparticles accessible to a variety of industries. Operating in a wide range of sectors, the company tailors its process to customer specification by using both large and small-scale reactors to continuously meet demand and maintain quality.

As a partner in the national, Innovate UK-funded project ICELIP, Promethean Particles is involved in the development of super-hydrophobic nanomaterials for use in anti-icing coatings. Since the project contains partners from all parts of the value chain, it provides a great overview of how nanomaterials can be adapted and upscaled using Promethean's continuous flow methodology, to then be formulated downstream into an ice-repellent coating for the aerospace sector. The project primarily focuses on the aerospace sector for end-use, to reduce drag and associated costs while improving fuel efficiency, but the coating technology could benefit several other sectors including, but not limited to, architectural coatings, the oil and gas industry, and ground transportation.

Particle Dispersion in Liquid Formulation: Delegates

We are delighted to attract so many delegates to our online meeting (454).

Names and affiliations for those that agreed on list are below (363). Alphabetical by surname.

Name	Affiliation
Professor Steven Abbott	Steven Abbott TCNF Ltd
Mr Mostafa Abdine	FOSROC
Mr Akeem Abiodun	Intecil Products Limited
Professor Michael Adams	University of Birmingham
Dr David Adrian	Dow
Dr Attia Afzal	The University of Lahore
Dr Sabiyah Ahmed	Surface Measurement Systems
Other Rana Ahmed	Mawlana Bhashani Science and Technology University
Mrs Swetha Ainampudi	GlaxoSmithKline
Mr Emmanuel Ajayi	Formulation Science & Technology Interest Group
Mr Emad Alavi	Amirkabir University of Technology
Mr Robert Albright	FMC
Mr Bahjat Alhasso	University of Huddersfield
Dr Muhammad Ali	Department of Textile Engineering, NED University of Engineering & Technology, Karachi
Dr Md Ashraf Ali	Associate Professor, Mawlana Bhashani Science and Technology University
Dr Mohammed Alkattan	OxSonics Therapeutics
Dr Rohan Ambardekar	Almac Group
Dr Kris Angamuthu	GlaxoSmithKline
Mr Mohd Ajmal Ansari	Laid off worked for Baker Hughes
Mr Khairil Anshari	Universitas Gadjah Mada
Dr Justice Archer	University of Bristol
Mr Javier Arrieta Escobar	Université de Lorraine
Mr Yaser Asgari	SNF
Mr Akshai ASHOK KUMAR	UNIVERSITY OF LEEDS
Ms Rachel Atkinson	PRA
Dr Mélodie Auriol	L'Oréal
Mr Jake Austin	Malvern Panalytical
Dr Carlos Avila	Independent Consultant
Mr James Bacon	Loughborough University
Dr Payal Baheti	Aston University
Dr Anna Giulia Balducci	Chiesi
Mr Nelson Barrios	University of Carabobo
Professor Andrew Bayly	University of Leeds
Dr Shilpa Beesabathuni	Quaker Houghton
Mr Alexandre Beirao	HeiQ Materials - Portugal
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Mr Milan Bharadwa	Ahmedabad Institute of Technology
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Dr James Bowen	Open University
Mr Phil Bowles	Impact Fluid Solutions
Dr Mark Bradley	Chemring Countermeasures
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Mr David Brossault	Cambridge University
Dr David Brown	Verona Pharma
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Dr Alberto Cattazzo	University of Padova
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Mr John Paul Cerroti	Analytical Approaches
Mr Ander Cervellera Dominguez	AkzoNobel
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Ms Pinyu Chen	KU Leuven
Dr Min Chen	Troy Corporation
Dr Shih-Chi Chu	Givaudan
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Dr Clare Conboy	Printed Electronics Ltd
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Mr Juan Del Rosario	BYK México
Ms Lucie Delforce	Université de Lille
Miss Shreyasi Deshpande	Unilever
Mr Alastair Dodds	Lubrizol
Dr Ben Douglas	University of Leeds
Dr Jo Douglas-Harris	Venator
Dr Marijana Dragosavac	Loughborough University
Mr Harry Drysdale	Northumbria University
Mr Volodymyr Dudko	Department of Chemistry and Bavarian Polymer Institute, University of Bayreuth
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Mr Don Dunner	FMC Corporation
Mr Isaiah Ekeanyanwu	OSCAR PAINTS LLC
Mr Toni El Geitani Nehme	Polytechnique Montreal
Miss Sarah EL MASRI	ULCO
Mr Abdelrahman El-Diasty	Prime Rock Energy Capital
Ms Mayada Elgohary	Alexandria university
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Dr Anabelle Elton-Legrix	Imerys
Mrs Ayeh Emrani	Tehran University of Science
Mr Laurent Espitalier	Harkness screens
Dr Chris Evans	EvanMile Consulting / Peratech Holdco Ltd
Mr Paul Evans	Lonza
Mr Amirhossein Fadavi	Petroleum university of technology
Mr Muhammad Faisal	PT. Nano Matrix-Lab International
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Dr Laura Felisari	bp
Dr Kay Florence	Siltbuster
Dr Claudio Fonte	The University of Manchester
Dr Carlos Fonte	Johnson Matthey
Dr Robin Fraser	Vive Crop Protection
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Dr Andre Zamith Cardoso	Cardoso & Wied Engineering
Dr yixing (Philip) Zhao	Quaker Houghton Company
Mr Qing Zhou	Syngenta
Dr Xiaomin Zhou	University of Copenhagen

Upcoming Events for 2021 and beyond:

In line with guidance from the Royal Society of Chemistry we have postponed all face-to-face events. Looking forward to the future we plan to offer both face-to-face and online events.

2021 Events:

RSC-FST **2 March 2021 – Keeping it Green in Personal Care**

RSC- FST **12 March 2021 - Formative Formulation 2** - Early career event

RSC-PC **25 March 2021** – The Forge – Characterisation of Pharmaceutical Formulations

RSC-FST **5-9 September 2021 – Advances in Corrosion Protection by Organic Coatings (ACPOC7)**

Future events:

We would love to hear from you if you have an idea for a new RSC-FST meeting and/or if you would like to help with the organisation of an event.

Please visit our dedicated website to find out more about our upcoming events.

Relive our past events:

You can read reports from our past conferences and re-live past online events via YouTube links!

10 November 2020 – **Modelling and Simulation in Formulation**
Hear about how modelling and simulation are revolutionising formulation

3 November 2020 – **Formulation 4.1: Putting Digital in Formulation**
Hear about how we are bringing digital to all aspect of formulation and look at how Industry 4.0 is changing the formulation industry.

Please visit the RSC-FST website at: http://formulation.org.uk/
Please visit the RSC-PC website at: http://my.rsc.org/groups/home/118
Please contact the RSC-FST: secetary@formulation.org.uk

RSC INTEREST GROUP FORMULATION SCIENCE & TECHNOLOGY

RSC INTEREST GROUP PARTICLE CHARACTERISATION