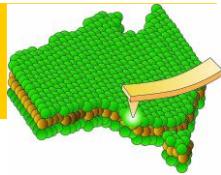


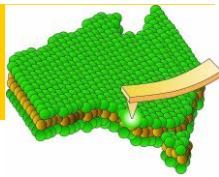
Program and Abstracts

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Preface



The inaugural Australian Colloid and Surface Chemistry Conference student conference was held in Melbourne in 1967, under the watchful eyes of our Australian colloid science founding-fathers, Bob Hunter and Tom Healy. The aim of the conference was to allow students to present their research in an informal environment, and to expose them to robust questioning and constructive criticism. The spirit of the first conference has been retained throughout each of the subsequent conferences. The numbers attending the conference have grown dramatically over the intervening years, and now as many as one hundred and twenty participants take part in each meeting, with representation from as many as fifteen Australian universities and half a dozen Australian industrial companies.

The Ian Wark Research Institute at the University of South Australia has the honour of hosting the 24th Australian Colloid and Surface Science Student Conference (24ACSSSC). This conference has 46 student talks, 28 poster presentations and 1 plenary lecture given by an overseas visitor. We would particularly like to welcome our overseas contingent, including 7 students and 3 conference guests. We are also delighted by the level of interest and support in the conference by our range of sponsors.

Welcome to the 24th ACSSSC. We hope you enjoy all the facilities available to you, particularly some of the fine South Australia products.

Gayle Morris

Nate Stevens

David Beattie

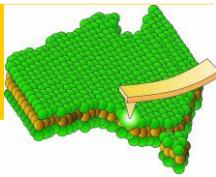
Kathy Prohaska

Gillian Kaggwa

Lucy Wang

Gary Hanly

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The 24th Australian Colloid and Surface Student Conference Organising Committee would like to extend their sincere thanks for the generous support that the following sponsors have provided:



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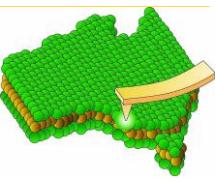


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Previous Conferences



Year	Location	Total No Attendees
1967	Melbourne	
1968	Sydney	
1970	Melbourne	14
1972	Sydney	
1973	Melbourne/Blackwood	30
1974	Sydney/Woy Woy	39
1976	Canberra	33
1977	Melbourne/Blackwood	34
1978	Yarrawood	
1980	Kioloa	50
1982	Mt. Eliza	75
1983	Yarrawood	51
1985	Roseworthy	49
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1988	Albury	107
1990	Camden	
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1993	Deakin Uni	144
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List of Participants



BRAGG INSTITUTE, ANSTO

Michael James
Jamie Schulz

AUSTRALIAN NATIONAL UNIVERSITY

Drew Evans

CHIANG MAI UNIVERSITY, THAILAND

Preecha Panya

CSIRO, MOLECULAR SCIENCE

Patrick Hartley
Sofia Kihlman Øiseth
Sineaid Lagan
Darrell Wells
Annabel Wood

EPA, VICTORIA

Gillian Sparkes

KASIKTAN INSTITUTE OF CHEMICAL PROCESS, TASHKEN

Nikta Bor

LATROBE UNIVERSITY

Mike Angove
Daniel Bennett
Tristan van Emmerick
Jacqui Heard
Jaslin Ikhsan
Bruce Johnson
Ali Shareef
John Wells

MONASH UNIVERSITY

Anke Wendelborn

SWINBURNE UNIVERSITY OF TECHNOLOGY

Gordon Bewsell
Russell Crawford
Daniel Eldridge
Teresa Harding
Philip Tu

UNIVERSITY OF BRISTOL, UK

Rob Atkin
Terence Cosgrove
Pierre Starck

UNIVERSITY OF GRAZ, AUSTRIA

Angelika Basch
Liliana De Campo

UNIVERSITY OF MELBOURNE

Alexandria Angelatos
Muthupandian Ashokkumar
Sasha Boskovic
Frank Caruso
Rachel Caruso
Thanh Tam Chau
Envi Ciawi
Tristan Croll
Dave Dunstan
Michelle Gee
Brendan Gladman
Sarah Glasson
Di Goodall
Will Goodall
Franz Grieser
Tom Healy
Lillian Lee

*Paul Luckham - visiting from Imperial College, UK

Scott McLean
Felix Meiser
Ineke Muir
Shobha Muthukumaran
Jessica Pacifico
Ying Pan
Shashi Parcha
Anna Plotka
Stuart Prescott
John Sader
Peter Scales
Colin Scholes
Ritu Singla
Anthony Stickland
Lindsay Studer
Chor Sing Tan
Elvira Tjipto
Shane Usher
David Verrelli
Vivek Vivekanand
Thanh Duc Vu
Richard Watson
Nee San Yap
Jasmina Zukan

UNIVERSITY OF NEW SOUTH WALES

Konika Khan
Peter Kovalsky

UNIVERSITY OF NEWCASTLE

George Franks
Paul Jailani
Aaron Olsen
Emelyn Smith
Erica Wanless
Peter Yates
Ying Zhou

UNIVERSITY OF OTAGO, NZ

Scott Dickie
David Warren

UNIVERSITY OF QUEENSLAND

Simon Corrie
Angus Johnston

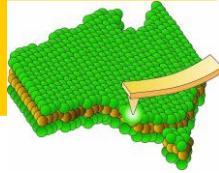
UNIVERSITY OF SYDNEY

Annabelle Blom
Bob Hunter
Kathryn Topp
Greg Warr

UNIVERSITY OF SOUTH AUSTRALIA

Jonas Addai-Mensah
Sameer Al-Bataineh
David Beattie
Kristen Bremmell
Jarred Clasohm
Jinming Duan
Ana Duarte
Yan Er
Saeed Farrokhpay
Carlos Gontijo
Gary Hanly
Roger Horn
Le Huynh
Gillian Kaggwa
A. Kanta
Nicola Lake
Huixin Li
Angus McFarlane
Melanie McGuire
Tatu Miettinen
Gayle Morris
Clive Prestidge
Craig Priest
Rada Pushkarova
John Ralston
Rossen Sedev
Spomenka Simovic
Nate Stevens
Su Nee Tan
Lucy Wang
Tony Zhu

Scientific Program



	2/2/04	3/2/04 Breakfast	4/2/04 Breakfast	5/2/04 Breakfast	6/2/04 Breakfast
07:00					
9:00		Saeed Farrokhpay	Teresa Harding	Ying Pan	Plenary Speaker – Paul Luckham
9:20		Nicola Lake	Angelika Basch	Aaron Olsen	
9:40		Sarah Glasson	Colin Scholes	Shobha Muthkumaran	
10:00		Daniel Eldridge	Annabelle Blom	Kathryn Topp	
10:20	Registration	Morning Tea	Morning Tea	Morning Tea	
10:50		Gillian Kaggwa	Su Nee Tan	Jacqui Heard	
11:10		Chor Sing Tan	Anthony Stickland	Felix Meiser	
11:30		Liliana de Campo	Emelyn Smith	Sameer Al-Bataineh	
11:50		Brendan Gladman	David Warren	Huixin Li	
12:10			Jaslin Ikhsan	Ali Shareef	
12:30	Lunch	Lunch	Lunch	Lunch	
2:00	Spomenka Simovic	Simon Corrie		Ritu Singla	
2:20	Tristan Croll	Yan Er		Pierre Starck	
2:40	Ineke Muir	Scott Dickie		Philip Tu	
3:00	Nate Stevens	Rada Pushkarova		Elvira Tjipto	
3:20	Afternoon Tea	Afternoon Tea	Staff-Student Beach Cricket	Afternoon Tea	
4:00	Craig Priest	Drew Evans		Poster Session	
4:20	Lucy Wang	A. Kanta			
4:40	Peter Yates	Scott McLean			
5:00	Angus Johnston	Will Goodall			
5:20	Free Time	Free Time	Free Time	Free Time	
6:00	Happy Hour	Happy Hour	Happy Hour	Happy Hour	
7:00	BBQ	Dinner	Dinner	Conference Dinner	
		Quiz Night	Talent Night		

Monday 2/2/04	1:50pm	WELCOME	Gayle Morris
Monday 2/2/04	2:00-3:20pm	ORAL # 1	Chair: Erica Wanless
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5:00pm	Novel Colloidal Substrates for High Throughput Biological Screening: Drug and Gene Balls Explained	23	
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Monday 2/2/04	7:00pm-late		BBQ

Tuesday 3/2/04	9:00-10:20am	MADDERNS ORAL # 3	Chair: David Beattie
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9:20am	Photo-Induced Reversible Wetting of Bio-Molecular Architecture	25	
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STAFF-STUDENT BEACH CRICKET

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TATACHILLA WINE TASTING

Thursday 5/2/04 7:00pm-late

EPA VICTORIA CONFERENCE DINNER

Friday 6/2/04 9:00-10:00am BHP BILLITON PLENARY Chair: Tom Healy

9:00am Poking Around and Stirring Things up: AFM and rheology of colloidal and biological systems.....

Paul Luckham

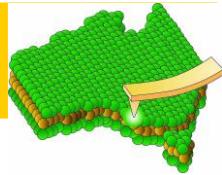
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Friday 6/2/04 10am

CLOSING REMARKS

Bob Hunter

Conference Awards



Healy-Hunter Award

The most outstanding oral presentation will be awarded the Healy-Hunter Award. This award is a prize of a \$1000 travel grant and is voted by the staff.

Best Poster Award

The most outstanding poster presentation will be awarded the Best Poster Award. This award is a prize of \$300 cash and is voted by the staff.

The Most Probing Question and the Most Memorable Moment Awards

As part of the tradition, there will be a presentation of specially designed trophies for both the Most Memorable Question and the Most Memorable Moment witnessed at the conference. To be eligible, the event is nominated and a winner decided by a carefully chosen panel of judges. The prize – embarrassment and notoriety!

Abstracts: Oral



Silica Nanoparticles at PDMS Droplet-Water Interfaces

Spomenka Simovic, Clive Prestidge, Daniel Fornasiero

Ian Wark Research Institute, University of South Australia, Mawson Lakes SA 5095

Spomenka.Simovic@postgrads.unisa.edu.au

The aim of this work is to investigate the behavior of silica nanoparticles (~ 50 nm) with different wettabilities at PDMS droplet ($1\text{-}2 \mu\text{m}$) - water interfaces. Nanoparticle adsorption isotherms have been quantified the thermodynamics of adsorption and the adsorbed layer microstructure visualized using freeze-fracture SEM. Droplet stability against coalescence upon screening the electrostatics was monitored using optical microscopy. The potential of nanoparticle layers to act as controlled release membranes was probed by *in vitro* dissolution test.

Hydrophilic silica adsorbs onto PDMS droplets up to a monolayer¹ whereas hydrophobic nanoparticles may form multilayers². Particle packing at the interface is governed by lateral particle-particle interactions. Surface coverages correspond to effective particle diameters for hydrophilic silica and closer packing for hydrophobic silica. Furthermore, interfacial particle saturation occurred at a salt concentration two orders of magnitude less than the critical coagulation concentration (ccc) for hydrophobic silica in water. This phenomenon was independent of droplet cross-linking and indicates that particle interaction through the water phase plays a decisive role in particle packing at the interface. SEM indicated: densely packed hydrophilic particle layers when the double layer thickness is a few nanometers¹, a rigid interfacial hydrophobic crust layer at salt concentrations corresponding to interfacial saturation and a multi-layered interfacial particle wall at salt concentrations $\geq \text{ccc}^2$.

Droplets coated by hydrophobised silica form stable flocculated networks in high salt solutions, whereas hydrophilic silica coated droplets give unstable networks due to the low attachment energy and less rigid interfaces. Regardless of the wettability, particles are not detached from the interface during coalescence. Limited coalescence generally occurred at sub-monolayer coverage with a droplet diameter arrested by interfacial saturation. When the coverage is between 70% and a monolayer, mesophase-like microstructures have been noted.

Droplet coating by nanoparticles dramatically influenced drug release profiles: bare droplets, as well as those coated with permeable particle layers (below 10^{-3} M NaCl) have half life for release less than 1 minute, and those coated with rigid particle layers (above 10^{-3} M NaCl) more than 18 h. The interfacial rate constant k_1 was calculated to be $4733 \text{ nm}^2 \text{ s}^{-1}$ which is significantly higher than for Pluronic® stabilised emulsions ($4.5\text{-}45 \text{ nm}^2 \text{ s}^{-1}$). These have potential as controlled release drug delivery systems.

1. Simovic, S; Prestidge C.A., Langmuir 19, 3785-3790 (2003)
2. Simovic S; Prestidge C.A., Langmuir, 19, 8364-8370 (2003)

Creation of a bio-functional surface on poly(lactic-*co*-glycolic acid) for use in soft tissue engineering

Tristan I. Croll, Andrea J. O'Connor, Geoff W. Stevens, Justin J. Cooper-White

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Current surgical transplant techniques generally have a number of drawbacks, such as donor site morbidity (autograft) and donor shortfall (allograft). Tissue engineering aims to address these problems, by persuading the body to re-grow the damaged, diseased or removed tissue. A commonly-used approach is to provide a porous, biodegradable polymeric scaffold in the shape of the desired tissue, which over time allows the infiltration of the desired cell type, and eventually degrades away leaving only native tissue.

Poly(lactic-*co*-glycolic acid) (PLGA) is a popular choice of polymer for this application, due to its ease of processing, good elastic properties, tuneable biodegradability and low toxicity. However, native PLGA has been shown to elicit a strong foreign-body response when implanted into mice, rats and pigs, leading to the formation of highly fibrous scar tissue. Surface modification aims to alleviate this problem, by post-processing the formed scaffolds to create a more bio-compatible interface, while leaving the bulk of the polymer untouched.

We have investigated two relatively simple techniques, base hydrolysis and aminolysis, to introduce functional end groups into the PLGA surface. These reactions have been followed using air/water contact angle measurement, x-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (ToF-SIMS). Crucially, we have shown that the reactions can be carried out throughout the bulk of polymeric scaffolds with overall dimensions in the order of centimetres and internal structures <2 microns thick, a test which many other surface modification techniques fail. Furthermore, XPS and ToF-SIMS was used to show that the newly-formed groups can be readily activated to covalently bind biological molecules, however at levels well below a complete monolayer.

In order to completely cover the PLGA surface, we have investigated a layer-by-layer approach starting from surfaces treated via the above methods, using two common biological polysaccharides. Preliminary results from this work, including cell adhesion and migration studies, will be presented.

Surface Interactions and Conformations of Adsorbed Proteins.

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The Surface Forces Apparatus¹ (SFA) uses interferometry to gain information regarding the conformation and surface interactions of polymeric species adsorbed onto a substrate. More recently, this method has also been applied to investigate the adsorption and surface interactions of proteins². Given the structural complexity of proteins in comparison to polymers and polyelectrolytes, the analysis of these systems is less straightforward. This is predominantly due to the secondary and tertiary structures of proteins, which enables them to adopt a variety of conformations at the solid/aqueous interface.

This study investigates the effect of solution conditions on protein adsorption at the solid/liquid interface using the technique of SFA. The data shows interesting phenomena with respect to the adsorbed structure and indicates that factors such as the concentration of protein and electrolyte, type of electrolyte, and adsorption history, greatly influence the equilibrium adsorption, and hence the measured surface interactions.

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The Wettability of Particles

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Advancing and receding contact angles of liquids on solid surfaces can be measured using a variety of different techniques such as the sessile drop, Wilhelmy plate or tilting plate. The “advancing” contact angles of packed particle beds can also be measured in systems that have been described by Washburn or Laplace-White equations where the rate of liquid rise or pressure required to stop capillary rise can be calculated into a meaningful contact angle.

In a flotation cell one of the important factors that contributes to the flotation rate constant described in the general flotation model is the contact angle. More specifically it is the sessile bubble contact angle or receding contact angle that is important. The techniques for advancing contact angles in particle beds do not lend themselves to receding contact angle measurements and therefore for high performance industries such as mining and oil recovery the measurement of the receding contact angle in a porous or particulate system would have great significance.

By modifying the Equilibrium Capillary Pressure Technique we can recede liquid from an ideal capillary and measure the receding contact angle. However in a particulate system it is impossible to observe how the liquid meniscus is behaving in real time thus making the measurement impossible. We have developed a method that measures the pressure decay when the liquid is rapidly receded from a saturated particle slurry and have found a relationship with the fraction of hydrophobic material in the slurry.

Contact Line Pinning on Heterogeneous Surfaces

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The wetting of heterogeneous surfaces is critical to the success of both industrial and natural processes. The efficiency of thin film preparation, cleaning, and separation process are highly dependent on the wettability of surface components. In many cases, the wettability of a chemically heterogeneous surface is predicted by the Cassie equation. However, for other applications (commonly industrial) Cassie's equation proves inadequate as a predictive tool.

By developing model micro- and nano-domain surfaces of contrasting wettability, the applicability of the Cassie equation to real surfaces has been tested. We show that random domain distributions and surfaces with gradual wetting transitions follow Cassie's law. Alternatively, isolated or ordered chemical features, namely "pinning sites", cause the measured contact angle to deviate from Cassie's prediction. It is this phenomenon that has become the focus of our attention.

To determine the origin and magnitude of the "pinning force", individual macroscopic domains were prepared on slides suitable for Wilhelmy Plate analysis. This method allowed direct measurement of pinning forces with excellent reproducibility and resolution. Reproducible pinning forces on micro-patterns of magnitude less than 2 μN have been resolved. Quantifying pinning forces directly on chemically heterogeneous surfaces is a significant step towards a predictive theory for non-Cassian wetting. We observe empirically the linear relationship between pinning force and contact line deformation, and show that "overstretching" is related uniquely to receding contact lines. Further characterization of the pinning force is expected to lead to an improved understanding, possibly including quantitative prediction, of contact angle hysteresis.

Investigation of Self-Assembled Monolayers on Mercury Using a Surface Forces Apparatus

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Studies of self-assembled monolayers (SAMs) of thiols on metals have attracted increasing interest in nanotechnology, biotechnology, colloid science and materials science. However, SAMs formed on solid metals tend to have defects such as pinholes and steps, caused by lattice mismatches between the thiol molecules and the crystalline structure of the solid (e.g. gold, silver, copper etc). Therefore in our work, a mercury drop was used as a substrate, to provide a defect-free and mobile surface. As a result, the SAM can form with high uniformity, smoothness and integrity.

A surface force apparatus¹ has been recently modified to allow investigations of interactions between mica and a mercury drop in the aqueous solutions to be studied. Applying an electrical potential to the mercury allows electrical double-layer forces between mercury and mica in aqueous solutions to be studied². Different thiols with different functional groups (-COOH and -OH) and different chain lengths (C₂ and C₁₀) were used to modify the mercury drop. From a comparison with measurements between a bare mercury surface and mica, it is evident that the -COOH SAM is negatively charged in aqueous solutions (at natural pH). Although the -OH SAM is uncharged, it modifies the structure of the double-layer at a charged mercury surface, and hence has a significant effect on the double-layer interaction with mica.

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Heterocoagulation of Colloids Using Nano-Particles

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Heterocoagulation is a widespread phenomenon. It is a common process in the pulp and paper industry, mining industries, and used extensively in the formation of coreshell particles and toner compounds. In the separation of particulate solids from liquids, polymers are usually used to create large aggregates, which can be more easily separated from the liquid than the original particles. This work focuses on an alternative to polymer flocculation, where small nano-sized particles are used as the flocculant instead of a polymer. Alumina particles (approx. 400 nm diam) are used as the colloid to be removed from the water. Silica nano-particles (size 6 nm to 300 nm diam) are used as the flocculant. The size and structure of aggregates and filtration behaviour are all investigated in this work. Low angle laser light scattering was used to investigate the size and fractal properties (structure) of the aggregates. The dose (mass of nano-particles) required for efficient flocculation increases with increasing nano-particle size, but the number of nano-particles required decreases with increasing nano-particle size. The growth of the aggregates (0.01% w/w Alumina) was monitored over time for all Silica particle sizes and it was found that the aggregate size peaked in the first 5 min of aggregation and then steadily decreased to smaller equilibrium size. A discussion of the effect of Silica particle size on the mass fractal dimension and aggregate size will be presented. The effects of overdosing, underdosing, shear in mixing and consolidation (filtration) behaviour will also be discussed.

Novel Colloidal Substrates for High Throughput Biological Screening: Drug and Gene Balls Explained

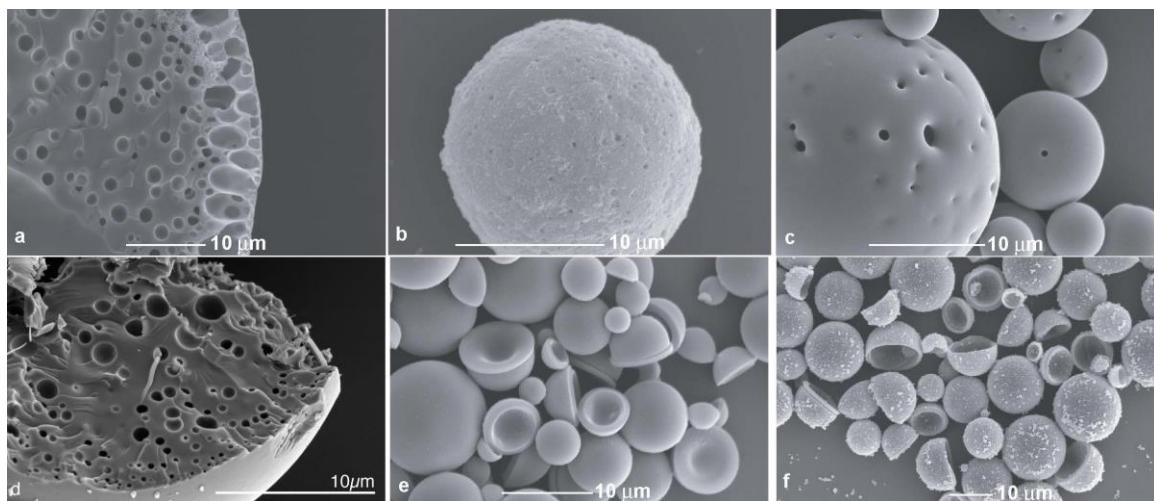
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The combination of high-throughput screening and combinatorial chemistry has the potential to be an extremely powerful tool for Genomics, Proteomics, Diagnostics and Drug Discovery. Large libraries of biomolecules, such as oligonucleotides and peptides, can be used to investigate cellular interactions such as proteins binding to immunoglobulins, the binding of regulatory proteins to DNA and RNA¹ as well as being of use in gene sequencing and Single Nucleotide Polymorphism (SNP) detection. The existing technology to encode combinatorial libraries has a number of limitations, so as an alternative we are using optically encoded colloidal particles as the supports for combinatorial chemistry². The supports can code for in excess of 10 million compounds and 'hits' can be detected and decoded at speeds up to 50 000 beads per second.

The basis of this procedure requires support particles which can have DNA and RNA coupled to the surface of the particle, as well as being able to hold an optical signature which does not significantly alter throughout the particle's lifetime. To reach this end we have developed a new class of porous functionalised ceramic, which can be encoded through combinations of fluorescent dyes which are covalently incorporated into the ceramic network to produce optically unique particles. We have also developed non-cleavable linker chemistry which allows DNA to be synthesised on the particles and keeps the DNA attached to the particles through all synthesis, deprotection and hybridisation steps. We will report on the controlled variation in particle morphology and optical diversity which can be achieved, as well as demonstrate how single nucleotide mismatches in DNA sequences can be discriminative using a coded DNA library.



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Dispersion Behaviour of Titania Pigment Particles in Aqueous Solution of Polyacrylic Acid and Polyacrylamide Based Dispersants

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Dispersion of titania pigment particles is very critical in industrial production, such as paints, paper and plastics. In water-based paints, for example, poor dispersion can cause low gloss and low opacity, as well as lower storage stability. Dispersion can be enhanced by the addition of dispersing agents such as organic or inorganic polymers. Therefore an improved understanding of the interactions between dispersing agents and pigments can provide more information in order to choose a suitable dispersant.

In this study, the interaction of polyacrylic acid and three polyacrylamide based dispersants with titania pigment particles is reported. Polyacrylamide reagents include a homopolymer, polyacrylamide substituted with hydroxyl functional groups and polyacrylamide substituted with carboxylate and hydroxyl functional groups. Thus a range of different functional groups (acrylamide, carboxylate, hydroxyl) has been analyzed. Adsorption isotherms of the dispersants onto titania pigment particles are discussed as a function of pH and electrolyte concentration. The effect of these dispersants on the pigment particles rheology, electrochemistry behaviour and particle size are reported at pH 6.0 and 9.5, either side of the pigment iep. Polyacrylic acid and anionic carboxylate substituted polyacrylamide have the greatest effect on the pigment particles rheology reducing the yield stress to very low values. The hydroxyl modified polyacrylamide has a negligible effect at pH 6.0, but a considerable effect at pH 9.5 while the effect of homo polymer is negligible.

Pigment dispersion is strongly influenced by type and number of the functional groups: Acrylamide shows no ability to disperse pigment particles but carboxylate is effective at both pH values. The dispersion property is enhanced in the presence of hydroxyl groups, with greatly enhanced when both hydroxyl and carboxylate are present.

Photo-Induced Reversible Wetting of Bio-Molecular Architecture

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Thymine (5-methyluracil) is a base of DNA which, when grafted to a surface as a self-assembled monolayer, displays interesting physical properties. The pKa alters from that in solution whilst the water contact angle reflects both the structure and charge at the interface. When irradiated with UV light of a defined wavelength, the molecular structure at the solid-water interface alters and a photodimer is formed. This dimerization process is accompanied by a change in surface wettability. Manipulation of the interfacial properties permits reversible wetting changes over a number of irradiation cycles to be recorded, depending upon the pH of the contacting liquid. The novel aspect of this system is that neither the liquid phase nor the solid phase needs to be altered in order to effect large changes in contact angle. Substitution of functionality at position C-5 with hydrophilic or hydrophobic moieties significantly alters the contact angle of the substrate as well as the pKa of the monolayer. Configurational changes that occur upon exposure to UV light are more pronounced and large contact angle variations are recorded. The surface molecular structures that cleave when exposed to UV light may also be photocleaved with sensitizers. The latter model the bio-chemical action of the enzyme photolyase. Introduction of sensitizers allow the reaction to proceed at controlled rates in visible light. The significance of this work is discussed with respect to molecular structure, surface analysis and possible applications.

The Role of the Interface in Solvent Extraction

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Solvent extraction is a useful tool in the hydrometallurgical industry for the separation and concentration of metal ions. The process generally involves the metal ion, dissolved in an aqueous phase, being transferred to an organic phase via complexation with an oil-soluble ligand. The aim of this project is to develop a better understanding of the mechanism of transfer of metal ions across a liquid-liquid interface in commercial solvent extraction processes. In this study, the kinetics of copper extraction by the active ingredient in Acorga P50 (5-nonyl salicylaldoxime) will be investigated using micelle systems to emulate the oil-water interface, as well as Attenuated Internal Total Reflectance (ATR) spectroscopy applied to free liquid interfaces. In addition to investigating the kinetics of extraction of this system, it is intended to investigate the role of additives on the kinetics of metal extraction. These additives either enter the solution from the ore or are added as flocculants during the process, and can have a significant effect on the performance. With the move to smaller, shorter residence time contactors, this has become increasingly important if these new contactors are to be used efficiently.

Adsorption of Aqueous Metal Ions onto TiO₂, Polystyrene Latex and Other Substrates

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Many studies have been performed in which aqueous heavy metals have been adsorbed onto a variety of different colloidal substrates in an attempt to model aqueous metal ion removal from industrial waste-water. One challenge in this modelling process is that it is very difficult to vary the individual properties of the adsorbent, such as its iso-electric point, surface area or dielectric constant, whilst keeping the other properties constant. There are substrates, however, where select properties can be varied whilst maintaining the overall chemistry of that substrate.

Current work is focusing on synthesising, and potentially controlling, the dielectric constant of, titanium dioxide (TiO₂). TiO₂ is an unusual oxide in that its dielectric constant, which can be varied by a change in crystal structure, includes values close to that of water. For this TiO₂ phase, there should be minimal difference to the solvation energy of a metal ion before and after adsorption. Adsorption would then, speculatively, be largely controlled by electrostatic forces and the commonly observed relationship between hydrolysis and adsorption would not be present. Rather, the adsorption pattern would be largely dependent solely on the charge of the ion. Trivalent ions would be expected to adsorb more readily than divalent cations and ions of a similar valency would be expected to exhibit similar adsorption.

Experimentation found that adsorption of aqueous metal ions onto TiO₂ (anatase crystal structure, dielectric constant 38.2) does not follow either pattern alone. The order of adsorption with respect to pH was found to be Pb²⁺ > Cr³⁺ > Cd²⁺ = Zn²⁺ > Ni²⁺ > Mg²⁺ > Ca²⁺. This closely matches the order observed for metal ion adsorption onto amphoteric polystyrene latex. Literature shows that this order of metal ion adsorption affinity is not uncommon, irrespective of the adsorbing substrate employed.

These observations warrant the future study of the adsorption of the same suite of metal ions onto a variety of substrates to determine just how common this metal ion adsorption affinity pattern is. Such a study will allow insight into the processes that dictate metal ion adsorption, as well as the differences in adsorption efficiency between various metal ions.

The Influence of Polymers on the Inhibition of Bubble-Particle Interactions

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The influence of polymers on bubble-particle interactions is of great significance in mineral flotation and other separation processes. The attachment of a bubble to a mineral particle, allowing mineral recovery to occur, involves the formation of a finite contact angle, θ , at the three-phase contact line (tpcl) or, more correctly, the small zone where the solid-liquid-gas phases merge. The contact angle provides important information regarding whether or not a bubble will adhere to a surface and the stability of the bubble-particle attachment. The study aims to determine the influence of polymers on bubble-particle interactions by relating the contact angle with the polymer type and structure, adsorbed amount and adsorbed layer thickness. Lastly, the contact angle will be linked to force-distance data where bubble-particle interactions are determined by colloid probe AFM measurements.

The investigated surfaces embrace both model and mineral systems, where modified silica surfaces model the mineral talc. The studied polymers are well-defined polyacrylamides and polysaccharides. Contact angle measurements on flat model surfaces have shown that the contact angle of hydrophilic silica remains zero in the presence of polymer. The contact angle of dehydroxylated silica decreases in the presence of the studied polymers. The contact angle of methylated silica is only decreased in the presence of a high molecular weight polysaccharide. The contact angle of talc decreases in the presence of polymer in the same trend found for dehydroxylated silica. The contact angle measurements have been related to the absorbed amount, as determined through adsorption isotherms. Dynamic Surface Tension (DST) measurements have also shown that the polymer does not adsorb at the liquid-vapour interface and that polymer adsorption is exclusively at the solid-liquid interface. The Tapping Mode Atomic Force Microscopy (TMAFM) has been used to image the adsorbed polymer on the studied surfaces both in air and solution. These images have been related to the adsorption isotherms, the contact angle measurements and layer thickness determination by zeta potential measurements.

The investigation of the influence of polymers on bubble-particle interactions will prove to be valuable to the mineral processing industry. The study will provide a predictive tool that can link a specific polymer with a contact angle change. This will be achieved by investigating the adsorbed amount, layer thickness and the force of interaction between a bubble and a particle. This information can then be used to predict a flotation response in mineral processing.

Motion of Protein in Smart Gel – From FRAP Study

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The technique of fluorescence recovery after photobleaching (FRAP) is introduced for the measurement of translational motion of solutes in various media, i.e. free solution, polymer solution, and condensed media. The diffusion coefficient of the solutes is calculated from the spatial Fourier analysis of the fluorescence recovery video images. The operation and theory of analysis of FRAP will be specifically explained. This method is used to measure the diffusion coefficient of various proteins in an associative polymer gel, poly(ethylene oxide)-*b*-poly(propylene oxide)-*b*-poly(ethylene oxide)-*g*-poly(acrylic acid) (Pluronic-PAA). Above the critical micellization temperature (CMT), the Pluronic-PAA polymers form micellar aggregates. The diffusion of rigid spheres (proteins), i.e. bovine serum albumin and hemoglobin was found to depend on the Pluronic-PAA gel structure. A transition in normalized diffusion coefficient of these proteins was observed at the CMT of Pluronic-PAA, due to the obstructed motion when the gel structure changes.

Monoglyceride-Water Systems: Bulk Phases and Dispersions

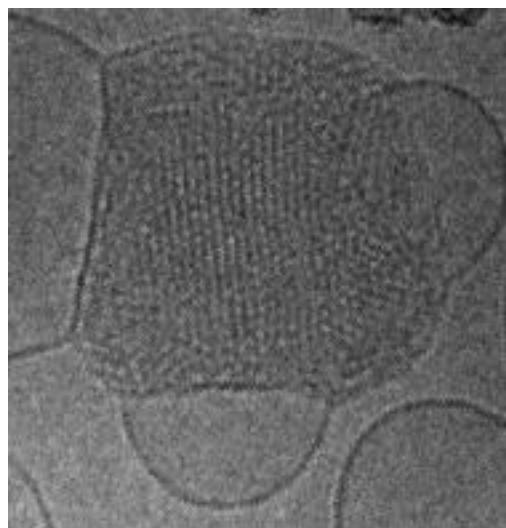
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Monoglyceride-water systems exhibit a large variety of nano-structured phases depending on water content and temperature¹. It is of special interest for potential applications that bicontinuous cubic liquid crystalline phases are formed within a certain range. These can be dispersed as nano-structured submicron particles in excess water stabilized by an additional emulsifier (so-called “cubosomes”², see Cryo-TEM image in the figure).

In order to understand the influence of various effects in these cubic phases, we made SAXS investigations to retrieve information on the structures occurring in the dense monolinolein-water system as function of composition and temperature in great detail. We found that especially samples in the cubic Ia3d phase at higher temperature have a strong tendency to align with respect to the sample holder and thereby create big monodomains, which rendered a successful application of our average SAXS experiments impossible (combination of slit-collimation and capillaries as sample holders). At the moment we are working out how we can modify sample preparation and SAXS-setup to prevent these problems.



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The Effect of Shear on Compressional Dewatering

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Gravity thickeners are a commonly encountered industrial process for concentrating flocculated suspensions prior to tailings disposal. Provided that the rheology of the suspension is known, it is possible to model the thickening process and therefore predict the dewatering performance. It has been found that when compared to measurements from a pilot scale thickener, the model predictions are very good. At full scale however the model under predicts the performance by up to an order of magnitude. One possible explanation for this discrepancy is the neglect of shear forces in the mathematical model. The aim of this work is to determine what role, if at all, shear plays in dewatering. In the presentation I will discuss a novel technique for isolating shear as a failure mechanism in a quantifiable way, and also show what effect varying shear has on the suspension rheology.

Encoded Colloidal Libraries for Reproducible Gene Expression Analysis

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One of the conclusions from the recently completed Human Genome Project is the requirement of new technologies for the sequencing and analysis of entire genomes in an efficient and high-throughput manner. Array-based technologies developed over the past 10 years have contributed a great deal to the field but still fall short of requirements due to major problems with data reproducibility and limited throughput ($\sim 10^5$ oligos/slide). Recent literature suggests that replicate array experiments are susceptible to unacceptably high random error and ~15% statistical outliers. The variability between laboratories is a major obstacle to setting up public expression databases and even intra-laboratory results are doubtful⁽¹⁾. Colloidal DNA libraries hold great promise due to the three-dimensional liquid phase kinetics and higher throughput (theoretically $\sim 10^{10}$)⁽²⁾. Such methods are expected to provide rapid developments in medicine including gene expression analysis, related diagnostic techniques and advanced therapeutics based on personalised medicine.

This work involves the development of microbead-based DNA libraries for use in biological sequencing and genetic analyses. Gene expression is the first of the applications targeted as small bead-based libraries significantly increase the reproducibility and hybridisation signal strength compared with current 2D microarrays. Single nucleotide discrimination is currently possible using a colour encoded library of oligonucleotides directed to a single fluorescent target molecule. With the process of on-bead library synthesis having been established, the size and diversity of these libraries is constantly being scaled up for more complex genetic analysis. The results of repeated gene expression analyses will be presented, showing the reproducibility, superior hybridisation kinetics and signal to noise ratios. Ongoing optimisation of library synthesis and diversity will also be displayed to indicate the applicability of colloidal DNA libraries.

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Interfacial Adsorption and Drug Release from Liposomes and the Influence of PEG

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Liposome instability in blood circulation and difficulties in the efficient and stable encapsulation of drugs have limited the number of liposomal pharmaceutical products. Poly (ethylene) glycol (PEG) is widely used to impart steric stabilization of liposomes⁽¹⁾ and Stealth® liposomes offer many advantages over conventional liposomes. The aim of the current investigation is to develop a more comprehensive understanding of the role of PEG in controlling the interfacial interactions of liposomes and drug release kinetics.

Conventional liposomes containing lecithin and cholesterol, and Stealth® liposomes containing dimyristoyl phosphatidyl ethanolamines-PEG 5000 were investigated. A model drug, guanosine was loaded by a reverse evaporation-freeze thawing method. Interfacial interactions between liposomes and solid surfaces were studied by Fourier transform infrared (FTIR) spectroscopy in attenuated total reflectance mode⁽²⁾. Increasing the molar ratio of PEG in the liposomes significantly reduced both the rate and kinetics of liposome adsorption. Further investigations of the influence of solution and surface properties on liposome adsorption kinetics have given insight into the adsorption mechanisms. Guanosine release from Stealth® liposomes was determined using a dialysis-bag method. The influences of dialysis conditions, such as medium temperature, pH and salt concentration, as well as stirring speed on guanosine release rate from liposomes have also been investigated⁽³⁾. PEG incorporation in the liposome formulation also reduced the kinetics of guanosine release.

Findings from this study give insight into the actions of Stealth® liposomes in the body and may form the basis for improved liposome formulations.

Key words: liposome, adsorption, drug release, PEG, guanosine

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Dispersion of TiO₂ Paint Pigments

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Titanium dioxide (TiO₂) is the most important white pigment used in the coatings industry. However due to recent advances in the paint industry there is increasing pressure to produce more stable pigment dispersions. While there is a wide range of dispersant options available these have predominantly been arrived at via an empirical approach rather than through an understanding of the surface chemistry of the pigment. A reappraisal of traditional pigment dispersion methods is currently required and there is a need for dispersants, which attach more permanently to pigmented surfaces in a variety of paint preparations. Identification of the most suitable dispersant groups requires information about the surface chemistry of the pigment and on the chemical nature and strength of any dispersant-pigment interaction. Infrared spectroscopy using attenuated total reflection (ATR) allows for evaluation of both the nature and strength of such interactions.

A commercially available boehmite AlO(OH) sample has been selected as a model for the coating present on the majority of commercial pigments. Thin films (~0.5 μm) of the boehmite for *in situ* IR experiments are deposited onto a ZnSe ATR prism by drying an aqueous dispersion. The behaviour of these films with respect to variation in hydration and pH was investigated and the conditions under which they become unstable established. It was found that carbonate exists bound in a monodentate fashion to the surface of freshly prepared films. A procedure for the removal of adsorbed carbonate has been developed that yields an essentially clean surface for further experimentation. The strength of interaction between a range of ligands and the boehmite surface has been measured by the Langmuir adsorption isotherm. Molecules containing carboxylate groups bind well to the surface while phosphate and phosphonate groups provide an even stronger attachment.

SFA Force Measurements at a Gas-Liquid Interface

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The potential at a gas-liquid interface is very important for a number of practical issues, such as flotation and bubble coalescence, and for scientific aims: to gain a better understanding of the gas-liquid interface structure. From previous research, the data about surface charge of bubbles are very often contradictory because of side effects of the electrokinetic methods used, including surfactant contamination and bubble deformation affecting the analysis of results. Taking these facts into account, it is important to compare the potential on the surface of the bubbles using different methods that are not based on electrokinetics.

Recent developments of the surface force apparatus (SFA) allow fluid surfaces to be investigated. This alternative method, which we describe here, allows direct measurement of the double-layer and Van-der-Waals forces acting across a liquid film between a gas bubble and a mica surface. This modified SFA method was developed by Connor to research forces between a mercury drop and mica.^{1,2} The aqueous film thickness between the drop and the solid surface is determined using the same optical technique (FECO) as that employed in the surface force apparatus (SFA),³ in a situation where the top of the drop is flattened by the double-layer repulsion. It has been demonstrated¹ that the mercury drop can be replaced by a bubble in this experimental arrangement, and reasonable resolution (< 1 nm) can still be obtained in the film thickness measurement. The thickness depends on double-layer and van der Waals repulsion between bubble and mica, and acting pressure. The latter can be determined from measurement of the internal pressure of the bubble, (using a differential pressure gauge or Young-Laplace calculation).

Three ranges of film thickness have been observed, which can be explained from DLVO theory and hydrophobicity. When the potential on the air surface was larger than the mid-plane potential corresponding to film pressure, a large film thickness (up to 200 nm) is attributed to electrical double layer and van der Waals repulsion. In this case the potential on the gas-liquid interface can be calculated, since the mica potential is known from standard SFA or streaming potential measurements. In the case when potential on the air surface is less than the mid-plane potential, a small film thickness (7 – 15 nm) results due to van-der-Waals repulsion only. In some cases, zero film thickness was measured, which is thought to be due to the mica surface becoming hydrophobic.

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Nanorheology: An Atomic Force Microscopy Technique to Dynamically Study Properties of Materials of Small Volumes

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The atomic force microscope (AFM) has previously been applied to the measurement of surface forces (including adhesion and friction) and to the static investigation of material properties, such as hardness. The static measurements are too insensitive to study the properties of very small volume materials. By the use of a dynamic technique, one can measure material properties with sensitivities several orders of magnitude greater than their static counterparts. Here we describe a modification to the AFM that allows dynamic and static measurements to be made simultaneously. Typically this technique has been used to study the dynamic behaviour of highly confined liquids. The application of this technique has now been extended to study properties of solids of very small volume (μm^3). Examples of this technique for both liquids and solids will be illustrated by some recent experimental results.

Photo and Thermally Induced Wettability Changes on Silica and Titania

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The transition from hydrophobic to hydrophilic TiO_2 surfaces after exposure to UV radiation has been reported recently. This observation is important because it provides a simple method for wettability control (e.g. the antifogging and self-cleaning properties of titania are of significant practical interest). However the mechanism underlying this observation has not been correctly elucidated. In order to understand the surface phenomena responsible for these changes on (initially molecularly smooth) titania and other metal oxides a detailed comparison of the relevant surface chemistry with silica has been made.

Changes in surface concentration of silanol (SiOH) and titanol (TiOH) groups on low-surface-area silica and titania due to heat treatment and UV irradiation have been investigated using Static Time of Flight Secondary Ion Mass Spectroscopy, streaming potential and contact angle measurements. Changes in surface roughness and crystallinity were assessed by atomic force microscopy and Raman spectroscopy, respectively.

The MOH^+/M^+ ($\text{M} = \text{Si}, \text{Ti}$) intensity ratios were used to characterize the influence of UV irradiation and dehydroxylation (at 1050°C) on silica and titania. A decrease in $\text{SiOH}^+/\text{Si}^+$ intensity ratio was observed on dehydroxylated silica, which is in accordance with the silanol-siloxane conversion. A decrease in $\text{TiOH}^+/\text{Ti}^+$ intensity ratio was observed on titania after dehydroxylation and is ascribed to titanol-titanoxane conversion. In both cases these ToF-SIMS data were correlated with *in situ* ATR-FTIR data. Although the zeta potential decreased on both materials after dehydroxylation, interpreted through changes in electrical double layer characteristics, different wettability behavior resulted. Silica became hydrophobic (contact angle of 42°) whereas titania was completely wetted by water. Wettability changes for titania were ascribed to the decomposition of organic contaminants whereas for silica there is an essential alteration in water-solid interactions.

The Effect of Time and Compression History on the Adsorbed Behaviour of a pH-dependent Polymeric Micelle

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Poly(2-vinylpyridine)-poly(ethylene oxide) (P2VP-PEO) shows potential as a possible drug delivery system for anti-tumour drugs since it forms pH dependent polymeric micelles. When P2VP-PEO is initially adsorbed above the pK_a of the P2VP block, P2VP-PEO is thought to adsorb from solution as micelles that exist as either partially collapsed- or hemi-micelles at the mica surface. Below the pK_a of P2VP, the P2VP-PEO adsorbs as unimers, likely in a compact layer showing little looping and tailing into solution. Hence to better understand the adsorption behaviour of this polymer we have studied the interaction forces between layers of P2VP-PEO adsorbed onto mica as a function of time and compression history. This has been accomplished using the Surface Forces Apparatus (SFA) when the solution pH is either above or below the pK_a of the P2VP block.

The Characterisation of Gold in Complex Ores using QemSCAN and PIXE

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The comprehensive characterisation of ores in the gold mining industry is becoming increasingly important as the need to process complex ores becomes more prevalent. An in depth understanding of the associations and distribution of gold is required to identify the most appropriate processing route for any given gold ore and will help with lowering the cut-off grade for an operation to the absolute minimum.

A number of complimentary chemical, microscopy and microbeam techniques have been used to show the distribution and associations of gold in a variety of complex ores. QemSCAN mineral mapping was used as a precursor to more detailed elemental mapping using Proton Induced X-ray Emission (PIXE) to show the association of gold within minerals. This information was then compared with the results of diagnostic leaching of each ore to show the bulk distribution of gold between different minerals. These methods are complimentary and were used to develop an overall picture of how gold was associated and distributed throughout the ore minerals.

Emulsion Phase Matching (EPM) Technique for Predicting Optimal Emulsifiers

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In recent times cosmetic emulsions have increased in complexity with performance claims of the product being added to product labelling, as well as special mentions of beneficial ingredients such as vitamins, herbal extracts or essential oils. The effect of this is to increase the amount of ingredients commonly found in commercial creams and lotions and thus increase their complexity. In addition, the importance of high skin compatibility for raw materials has limited the choice of emulsifier(s) that can be selected.

The formation of a ‘stable’ emulsion remains more of an art than a science. Despite the very many commercial emulsion products available and the considerable time and effort spent on research over the last fifty years, there is still no definitive technique available that can pinpoint an emulsifier or emulsifier combination that will form a guaranteed ‘commercially stable’ emulsion. Trial and error remains an integral part of emulsion formulation. The popular Hydrophile-Lipophile Balance (HLB) technique can be used to reduce the number of trials and is well known to the emulsification industry. However, the technique has limitations, both from the complexity of modern formulations, and the increased usage of new, raw materials used in cosmetic products which do not have a required HLB yet determined..

A fresh approach to emulsifier selection is desirable and especially an approach that can take into account all the ingredients included in the emulsion. This work proposes a new concept called the Emulsion Phase Matching or EPM technique. The name arises from the fact that, in this technique, each phase of the emulsion is considered independently of the other, although with all auxiliary ingredients included. A quantitative measure of the ‘difference’ between the two phases to be emulsified (in this case interfacial tension) is ‘matched’ to the difference between the hydrophobic and hydrophilic moieties of the emulsifier to be used. A good match is aimed to correlate to a ‘stable’ emulsion.

Although the work completed only touches the surface of a full validation of the proposed method, it does show encouraging signs and raises some interesting questions regarding emulsion formation, resulting stability and the mechanisms involved with each process. This is an area where further work is justified in the quest to remove the trial and error approach to emulsifier selection.

Substrate Induced Coagulation (SIC) of Carbon Black in Non-Aqueous Media: A Method to Manufacture Highly Conductive Cathode Materials for Li-Ion Batteries

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The electrical conductivity of cathodes for Li-ion batteries is usually obtained by adding a known amount of carbon black to the active material. In the optimum case, the carbon black should cover the whole surface of the particles as uniformly as possible. Conventional fabrication of carbon black electrodes entails the mechanical mixing of carbon black and the active material. The percentage of carbon black can be significantly decreased by coating the particles by the Substrate Induced Coagulation (SIC) process. The conductivity of electrodes and the amount of active material remains high because of an improved percolation pattern. This results in a more uniform distribution of current and potential.

SIC is a relatively new and widely applicable method for coating different surfaces (e.g. oxides, glass, metals) with fine particulate material. The process has been well established in aqueous solutions and provides a self-assembled rinse-proof coating of small particles that is free of binder.

The SIC process involves conditioning a substrate surface with a “monolayer” of polyelectrolyte. In a second step, the substrate is dipped into a meta-stable dispersion of particles (e.g. carbon black or metal oxides) containing a surfactant as a stabilizer (in water). An electrolyte is added to the dispersion in order to attain the desired stability. The polyelectrolyte or polymer on the surface destabilizes the dispersion and induces coagulation.

A major drawback, so far, has been that materials sensitive to hydrolysis could not be coated by the SIC process. The acceptable limit for trace water in non-aqueous batteries is 20 ppm and so the SIC process will only find practical application for coatings of active-materials used in Li-ion batteries (such as LiNiO₂ or LiCoO₂) if it can be performed in aprotic, non-aqueous solvents.

We report here a non-aqueous SIC carbon coating process that causes the conductivity of LiCoO₂ to increase substantially, whilst maintaining a high percentage of active battery material.

Photoinduced Energy Transfer Study on Bovine Serum Albumin Upon Adsorption to Hydrophilic Silica

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Serum Albumins are blood proteins that are of interest since they rapidly adsorb to synthetic biocompatible materials. Much interest has been shown in understanding their conformational state upon adsorption, due to the belief that surface proteins instigate further adhesion of biological material. Here under evanescent wave conditions, photoinduced energy transfer within Bovine Serum Albumin (BSA) is investigated to probe the extent of denaturation on a hydrophilic silica surface.

Time-Resolved Evanescent Wave Induced Fluorescence Spectroscopy (TREWIFS) provides insight into conformational change upon protein adsorption through fluorescence lifetime behaviour of chromophore species within the protein undergoing energy transfer. Since energy transfer efficiency is strongly influenced by inter-chromophore separation and orientation, changing transfer efficiency between chromophores will indicate the extent of rearrangement upon adsorption. Evanescent wave depth profiling provides the advantage of resolving transfer efficiency as a function of distance from the silica surface.

Direct Visualisation of Mesh Phases at Solid/Solution Interfaces by AFM

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Using soft-contact AFM imaging, the adsorbed layer structure of single chained dodecyltrimethylammonium bromide (DTAB) and double chained didodecyldimethylammonium bromide (DDAB) mixed solutions at varying compositions has been studied. DTAB forms rods on mica and globules on quartz whereas DDAB exists as bilayer on both substrates. On mica, the observed morphologies as composition was changed from DDAB-rich mixtures to DTAB-rich mixtures changed from bilayer to rods but also exhibited an isotropic, periodically textured structure in a narrow intermediate composition range. On quartz, the transition sequence followed bilayer → periodic isotropic texture → rods → globules with increasing DTAB content. Again, the periodic isotropic texture was observed only over a very narrow composition range.

We interpret the observed transitions by the formation of a mesh phase on the surface. This phase is consistent with the average curvature progression from bilayer to rods, and consists of branched rods. Studies performed on solution structures of fluorinated surfactants¹ and long-chained polyoxyethylene surfactants² have elucidated the balance between headgroup curvature forces and alkyl chain flexibility as responsible for formation of intermediate structures in bulk. It is believed that the resulting balance of forces between structures of high curvature preferred by DTAB and the reduced alkyl chain flexibility of DDAB is being satisfied by the formation of a mesh phase at the solid/solution interface.

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The Relationship Between Gibbs Elasticity and Dynamic Foam Behaviour of Polypropylene Glycol

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Foam is very important in various industrial processes such as plastic and ink recycling, mineral processing and enhanced oil recovery, however, little is known about factors that influences foam behaviour. Static interfacial property cannot fully account for foam behaviour, which is reflected in the dynamic nature of foam. The focus of this work is to study factor that influences dynamic foam behaviour.

The foamability of the aqueous solutions of low molecular weight polypropylene glycol (PPG), molecular weight of 400 and 725 g/mol, was found to reach a maximum at intermediate concentration. Foamability is an indirect interpretation of the drainage rate of liquid between bubbles in the foam. Surface tension gradients present on a bubble surface can slow down the thin film drainage rate and impart stability to foam. The Gibbs elasticity defines the surface tension gradient present on a surface that is initially in equilibrium with the bulk solution.

We have found a parallel relationship between Gibbs elasticity, measured by the oscillating bubble technique, and foamability after correcting for the non-equilibrium interfacial behaviour in foam using a model. It can thus be concluded that the ability of frothers such as popropylene glycol to create a surface tension gradient influences the dynamic foam behaviour. It is thus possible to predict foam behaviour based on a knowledge of surfactant properties.

Non-Traditional Constant Pressure Filtration Behaviour

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Pressure filtration is a common industrial process used for solid/liquid separation and as a laboratory technique to measure the compressional rheological properties of suspensions. Traditional modelling of constant pressure filtration behaviour of particulate systems using Darcian approaches stipulate quadratic behaviour of time with filtrate volume. This is a consequence of long cake formation and in relative terms, short cake compression times. However, this is not necessarily a fundamental attribute of pressure filtration, but rather a result of the assumptions made of the compressibility and permeability of the material. This work solves diffusion-type constant pressure filtration governing equations using a finite element technique to demonstrate that filtration, under certain circumstances, is expected to show negligible cake formation times and long compression times. Materials that exhibit such behaviour have often been classified as non-traditional and methodologies have been developed to interpret such trends. This work illustrates that such behaviour is in fact covered by extant filtration models and do not require any extra interpretation of the forces involved.

The Adsorption of pH Responsive Block Copolymers to Silica: The Effect of Aggregation

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The adsorption of a diblock copolymer [poly - 2-(dimethylamino)ethyl methacrylate-*b*-poly-2-(diethylamino)ethyl methacrylate (DMA₉₃-DEA₂₄ Mn ~ 20 000 g.mol⁻¹)] at the solid/liquid interface has been investigated. Both blocks of the copolymer are weakly basic (pKa ~7.0-7.5 (*NB: these values are for the homopolymer*)) and charge/discharge as a function of pH. Aggregation into micelles arises because at low pH both blocks of the copolymer are highly charged and the copolymer exists in solution as unimers, whilst above pH 8 the DEA block is sufficiently discharged to become 'hydrophobic' and drive the formation of micelles. This transition between unimers and micelles is reversible and occurs over a narrow pH range as observed using fluorescence spectroscopy.

The adsorption behavior of this copolymer at the silica-solution interface has been the focus of this work where a comparison is drawn between flat silicon wafers and 1000 nm diameter silica particles. Given the pH sensitivity of the diblock copolymer in solution, it was decided to monitor adsorption at both low and high pH values to investigate the effect of copolymer aggregation on the adsorption characteristics. Adsorption isotherms onto silica particles were determined using a standard solution depletion methodology and optical reflectometry (OR) was used to examine the adsorption onto oxidised silicon wafers; where the adsorption kinetics were also investigated.

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In Situ Studies of Photocatalysis On TiO₂ Thin Films

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In order to improve the efficiency of photocatalysts a better understanding of the influences of UV radiation on the surface structure of TiO₂ and the nature of the adsorbed species is required. The importance of oxygen and water are well known [1,2] and it seems probable that reactions proceed through such intermediates as superoxide and hydroperoxy species [3]. In spite of this, little work has focussed on the mechanisms of the interfacial processes.

We have used attenuated total internal reflection (ATR) infrared spectroscopy to monitor in situ adsorption and photocatalysis experiments. Micron thick particle films of the TiO₂ were deposited on an inert diamond-faced ZnSe ATR prism surface and irradiated in near UV light whilst in contact with gaseous or aqueous media. Infrared spectra in the 4000-600 cm⁻¹ region of both adsorbates and the TiO₂ thin films, as well as difference spectra after irradiation with near UV “black light” were obtained.

The effect of adsorbates on the surface of TiO₂ has been detected by the loss of absorbance due to surface phonon modes at ~830 and ~750 cm⁻¹. The irradiation of evacuated TiO₂ films rapidly gives rise to a broad absorption below 1500 cm⁻¹, previously attributed to surface peroxides but more likely due to mobile surface electrons [4].

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Adsorption of Aminopyridines to Clay Minerals

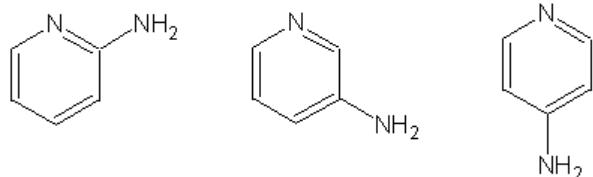
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Sorption of pesticides to soil particles may have a significant effect on their availability and their mobility within the environment. Because the structure of several common pesticides enables them to form complexes with heavy metals, sorbed pesticide molecules could also affect the availability and mobility of heavy metals.

As part of a wider study on pesticides we have studied the sorption of 2-, 3- and 4-aminopyridine



to three montmorillonite samples, and the co-sorption of aminopyridine and Zn(II). The clay minerals as supplied were in the K⁺ form; one of them was modified by exchanging the surface K⁺ with Ca²⁺. Sorption was measured over a range of pH and of concentration, and acid-base titrations conducted on suspensions. Changes in interlayer spacing, which may result from intercalation of adsorbates between the crystal layers, were monitored by X-ray diffraction. Changes in the protonation of sorbed aminopyridine were monitored by attenuated total reflectance infrared spectroscopic measurements on clay slurries. Finally surface complexation models of the sorption equilibria were developed.

The results showed that the three aminopyridine systems behave differently. Some of the experimental data, and their interpretation, will be presented.

Interfacial Phenomena in Micelle and Liquid-Liquid Extraction System: II. Interfacial Property of Extractant LIX 84 and Copper/LIX 84 Complex

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Solvent extraction is an important separation processes in the hydrometallurgical industry. It is used to purify a range of metals including copper, nickel, cobalt in Australia. This project aims at gaining in understanding at the role of the interface in extraction processes. In particular the study of the kinetics of copper ion extraction by LIX84 (2-hydroxy-5-nonylacetophenone oxime) is being undertaken. The techniques used to measure the kinetics are, stopped flow and attenuated total internal reflection spectroscopy (ATR). The first is based on the kinetics at a non-ionic micelle interface and the second at a free aqueous-organic interface.

In this presentation the interfacial activities of extractant LIX 84 (HL) and complex CuL_2 are presented. The interfacial tension data in an *n*-heptane/aqueous system are used to calculate surface excess by different isotherms such as the Gibbs, Szyzkowski, Temkin and Polynomial and used to predict the mechanism of copper extraction. It is generally accepted that reaction between copper and hydrophobic hydroxyoximes having a long alkyl proceeds at the interface. Thus the interfacial hydroxyoxime concentration is an important parameter, which influences the rate of copper extraction.

Kinetic mechanisms are derived from interfacial tension isotherms and compared with reaction data obtained from experiment over a range of conditions.

Shear Rate Dependence of Polymeric Flocculant Efficiency

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Effective solid-liquid separation by polymer flocculation requires efficient inter-dispersion of particles and flocculant. This is usually achieved by the introduction of fluid shear through mechanical mixing of the suspension. Whilst enabling the desired effect of inter-dispersion, fluid shear also affects the nature of the flocculation process and of the flocs produced. This work investigates the effect of shear forces on flocculant efficiency, specifically for the electrostatic patch flocculation mechanism. Optimum flocculant dosage is used as an indicator of flocculant efficiency. This is determined by static light scattering measurements of the rate of floc growth, rapid floc growth being indicative of effective flocculation. Alumina suspensions were flocculated with several poly(acrylic acids) of differing molecular weights. Results showed a dependence of optimum flocculant dosage on shear rate that increased with polymer size. Existing kinetic models for flocculation are used to explain these observations. A novel method for distinguishing between electrostatic patch and polymer bridging mechanisms is also introduced. Determination of the critical flocculation concentration for a given particle-polymer system, again by static light scattering, allows definitive determination of mechanism.

Enhancing Permeability During Cross Flow Membrane Filtration Using Ultrasound

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Whey protein concentrate (WPC) is a highly nutritional protein supplement produced from the ultrafiltration of dairy whey. Membrane fouling, which results in loss of productivity, is one of the most important operational concerns of this membrane process. In the present study, whey ultrafiltration is performed in cross flow mode using a single sheet membrane unit immersed totally within an ultrasonic bath. The effect of trans-membrane pressure, feed velocity, feed concentration and ultrasonic energy upon the steady state permeate flux is examined. The results show that the flux rate increases when ultrasound is applied. These results suggest that ultrasonically induced cavitation is effective in loosening and detaching the cake deposited on the surface of the membrane.

Experimental results are interpreted using a combined pore blockage and cake filtration model. The data is in good agreement with model calculations, with the parameter values providing important insights into the mechanisms governing whey fouling. This basic approach provides a framework that can be used to analyze whey fouling under different conditions.

Oligomeric Surfactants Through Polymerisation

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Oligomeric (sometimes known as 'gemini') surfactants consist of several ordinary surfactant units with short chains linking the hydrophilic heads, and display interesting and unusual self-assembly and rheological properties. Since their synthesis can be a lengthy and difficult process, we are investigating polymerisation techniques as a way of producing a range of oligomeric surfactants.

We are currently optimising the RAFT (reversible addition-fragmentation chain transfer) process for the polymerisation of a methacrylate-based surfactant monomer. RAFT agents (e.g. cumyl dithiobenzoate) are a special class of chain transfer agent, and when used in free radical polymerisations, provide both low polydispersity and control over the degree of polymerisation.

This technique allows specific low-molecular-weight surfactant oligomers (dimers, trimers etc.) to be produced. Analysis of the reactions is performed in real time using near-infrared spectroscopy, and the reaction products are identified and characterised using a variety of methods.

Foaming in Wastewater Treatment Plants

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One of the most common ways to treat sewage is through activated sludge wastewater treatment plants. This system uses microorganisms to break down organic components present in the influent and to remove other components such as nitrogen and phosphorous. One of the major drawbacks with this treatment method is that stable foam may be produced during the breakdown process. This causes many problems for the plant operators as it reduces the plant efficiency, leaves organic matter in the effluent and can lead to air-borne pathogens.

The questions we are trying to answer are: What is the role of the microorganism in foam formation and stability? Do they create the foam by producing a biosurfactant, and/or do they stabilize the foam once it is formed?

We have isolated and identified the dominant microorganisms from the five wastewater treatment plants located in the Melbourne area. During their growth cycle the bacteria produce a surfactant which causes a decrease in surface tension of the growth medium, and the production of stable foam on aeration.

Different spectroscopic techniques, ATR-FTIR, XPS and TOF-SIMS, are being used to investigate the bacteria, with particular emphasis on cell surface characteristics. From this work we aim to modify the bacteria, perhaps by adsorption, and thereby prevent foaming incidents. Results of bacterial growth trials and some spectroscopic data will be presented.

Fluorescent Rare-Earth-Doped Nanoparticles as Biolabels,

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Fluorescent rare-earth-doped lanthanum phosphate nanoparticles with a narrow size distribution of 5-6 nm have recently been synthesized by Riwozki *et al.*¹ 2001. The fluorescence is due to transitions between f- and the d-electron states, as well as their local symmetry in their guest crystal. The emission can be tuned by varying the dopant ions (Ce, Tb, Eu etc.). These properties make the nanoparticles attractive biolabels, which would overcome certain disadvantages of other biolabels, such as bleaching associated with organic dyes.

The first goal was to modify the surface of the nanoparticles to enhance the colloidal stability in water and to functionalize the surface for the attachment of biomolecules. By using the bifunctional molecule aminohexanoic acid the particles could be well dispersed and used for subsequent attachment of biomolecules.

To demonstrate the bioconjugation avidin was chosen, a tetrameric protein that has four symmetric biotin-binding pockets with a very high affinity to biotin ($K_D \sim 10^{15} M^{-1}$), which makes the avidin-biotin system a widely used system. Using the zero length crosslinker ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (EDC), avidin was covalently coupled to the surface of the nanoparticles. The particles and the particle-avidin bioconjugates were characterised by various methods, such as absorbance, zeta potential, dynamic light scattering and TEM. The bioaffinity of the bioconjugate was verified in binding assays with the dye biotin-fluorescein and the antibody biotin-anti-mouse-IgG.

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Static time-of-flight Secondary Ion Mass Spectrometry (ToF-SIMS) and X-ray Photo Electron Spectroscopy (XPS) of Surface-Immobilized Antibacterial Furanone Coatings

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The colonization by bacteria of biomedical devices presents a serious concern for human implant surgery. This study explores how bacterial colonization of materials can be prevented by the appropriate design and fabrication of antibacterial coatings, with a major focus on surface-immobilized furanone molecules. Brominated furanones are produced naturally by *Delisea* algae species and those compounds as well as synthetic analogous have been shown to be effective inhibitors of the bacterial process known as “quorum sensing”, which is an essential step in the process of biofilm formation. If quorum sensing can be inhibited of bacteria sitting on the surface of implants, one may be able to eliminate implant-centred infections. Substantial reductions in bacterial colonization have been achieved in a recent study [1], but not to the high degree required for clinical applications. Therefore, in this study, we are investigating how the method of surface immobilization of various furanone compounds affects the antibacterial effectiveness, via the structure of coatings, the mode of immobilization, and the orientation and presentation of the active moiety.

Furanone coatings were prepared following the previously used azide protocol [1]. First, a surface of fluorinated ethylene propylene copolymer (FEP) substrate was modified by n-heptylamine plasma polymerisation (HApp) in a custom-built reactor to create a thin film coating of amino groups. A further interfacial bonding layer was applied by immersing the FEP-HApp sample in an aqueous solution of poly(acrylic acid) (PAAC) at PH 4 for 16 h. Onto this carboxylated surface, a layer of 4-azidoaniline (AZA) was covalently attached by immersing the FEP-HApp-PAAC sample in an aqueous solution of 4-azidoaniline hydrochloride buffered to PH 8.8 under dark conditions for 16 h. Onto the azide surface a furanone compound was then immobilized by reaction of furanone compound with azide groups under UV illumination in a dark room. The surface modification steps were assessed by XPS and ToF-SIMS.

Each step of the immobilization sequence for the furanone were evaluated by XPS and ToF-SIMS. XPS results obtained at each step of the immobilization sequence for the furanone shown in Figure 1 are shown in table 1. XPS analysis of the HA layer showed complete attenuation of the F peak after plasma modification and the N 1s peak position of 399.3 eV is indicative of amino groups. The high resolution C1s spectrum demonstrated three components with binding energies associated with hydrocarbon (285eV), C-N (286eV), little contribution for C-OH/C-O-C (286.5eV) and N-C=O (288.1) functionalities. Positive ToF-SIMS analysis of the HA layer gave prominent peaks assignable to N-containing fragments. Grafting of PAAC leads to a decrease in the N content and an increase of the O content as expected for a PAAC layer with a thickness of less than 10 nm in the dry state as it is observed from the XPS results. High resolution of C1s region showed new component for COOH (288.9 eV) and an increase in the N-C=O (288.1eV) contribution. Fragments assignable to amid linkage bond (CNO), and CHO₂ were observed. Grafting of AZA leads to a decrease in the O content and an increase in the N content. After immobilization of furanone compounds new peak assigned to Br appeared in the XPS survey spectra and peaks for Br, N- and O-containing fragments were detected in the ToF-SIMS spectra. The most intense peaks in the Tof-SIMS spectra were for hydrocarbon fragments.

Table 1: XPS results of furanone multilayer build-up, atomic concentration in %

Sample	C	N	O	F	Br
FEP	33.4	0	0	66.7	0
FEP-HApp	89.3	9.5	1.3	0	0
FEP-HApp-PAAC	74.5	4.9	20.9	0	0
FEP-HApp-PAAC-AZA	76.2	9.6	11.6	0	0
FEP-HApp-PAAC-AZA-furanone (figure 1)	77.1	8.9	12.5	0	1.65

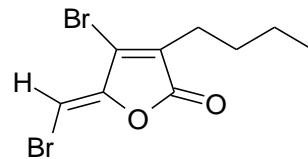


Figure 1:4-bromo-3-butyl-5-bromomethylene-2(5H)-furanone

Conclusion XPS and ToF-SIMS data show successful surface modifications and furanone immobilization. Detailed analysis of the C 1s and N 1s XPS signals shows that the intended reaction scheme does not proceed as cleanly as one might have hoped.

B. Muir *et al.*, Proc. 6th World Biomat. Congr., Hawaii, May 2000, p. 596

Particle Size Distribution Revealed Mechanisms of Al(OH)₃ from Caustic Aluminate Solutions

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The growth kinetics of colloidal Al(III)-containing particles formed in optically-clear, supersaturated caustic aluminate solutions as a precursor to Al(OH)₃ crystals, has been studied using dynamic light scattering (DLS) and X-ray powder diffraction (XRD). Three different solutions with constant gibbsite relative supersaturation (σ) of 7.55 ([NaOH] = 1.50 – 4.00 M, [Al(III)] = 0.97 – 3.28 M) were analysed (22 °C) to determine the particle growth behaviour and the crystalline phase of Al(OH)₃ crystals.

The non-negative least square (NNLS) method has been successfully employed within the multi-angular dynamic light scattering (MDLS) analysis program as the inversion method for calculating the particle sizes distribution from dynamic light scattering data [1]. The particle size distribution of the growing nuclei showed that the particles initially detected in a 1.5 M NaOH, 0.97 M Al solution (200–290 nm) were larger than those in a 4.0 M NaOH, 3.28 M Al solution (95–140 nm). X-ray diffraction patterns show that the crystalline phase of Al(OH)₃ precipitated from the less concentrated caustic solutions were predominantly bayerite (α -Al(OH)₃), whilst for the concentrated solution the phase is predominately gibbsite (γ -Al(OH)₃). The solution with the intermediate NaOH concentration (e.g. 2.00 M NaOH) showed an dimorphic phase mixture.

The particle size distributions in a size range of 30 to 1000 nm were calculated for these three solutions in three forms: scattered intensity, particle volume and particle number weighted. It appears that the particle growth mechanism involves both the addition of molecular units and agglomeration and/or Ostrald ripening of colloidal-size Al(III)-containing particles for both the dilute and concentrated caustic aluminate solutions.. The presence of multi modal particle size distributions in the more concentrated caustic aluminate solutions suggested the possible existence of secondary nucleation. This is in contrast to the unimodal distribution observed for the less concentrated solutions.

[1] Bryant, G., Abeynayake, C., Thomas, J. C., "Improved Particle Size Distribution Measurements Using Multiangle Dynamic Light Scattering. 2. Refinements and Applications" Langmuir, 6224-6228, Vol.12, No.26 (1996)

Endocrine Disrupting Chemicals (EDCs) in the Environment

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The presence of compounds in the environment with estrogenic properties known as endocrine disrupting chemicals or EDCs, has become a major concern over the past few years. The emergence of a number of such chemicals of industrial and pharmaceutical origin that influence the endocrine system in both humans and the wildlife has lead to extensive research on the development of analytical methods to determine EDCs in the environment. Some of the most prominent estrogens include natural female sex hormones 17β -estradiol (E2) and two of its metabolites estrone (E1) and estriol (E3); and the synthetic hormone 17α -ethynodiol (EE2).

The amount of work done on monitoring of EDCs in Australian aquatic environment is still very limited. We have been working on devising analytical methods to determine a selection of the most potential EDCs using GC-MS, which is often used to determine low concentrations (ng/L) of such pollutants in the environment.

While much of the current studies focus on identification of EDCs, sorption studies on EDCs may be invaluable in predicting how these environmental contaminants get transported into the food chain. We have done some preliminary work on adsorption of selected EDCs onto different soil minerals to gain some understanding of the behaviour of estrogenic compounds in the environment. Our initial choice of EDCs were Bisphenol A (an industrial EDC reported in high levels in many parts of the world), ethinodiol (the synthetic female sex hormone found in prescribed contraceptive pills) and estrone(a natural hormone). Some of the substrates include goethite, kaolinite and montmorillonite.

Sonochemical Decomposition of Benzoic Acid

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Ultrasonic irradiation has received considerable interest as an advanced oxidation process because it leads to the degradation of chemical pollutants in water. Sonication of aqueous solution causes cavitation in liquids, which results in the formation of **H** and **OH** radicals. **OH** radicals are known to oxidize chemical pollutants present in wastewater[1].

The aim of this study was to investigate sonolytic degradation of benzoic acid, a common pollutant in wastewater, at an ultrasonic frequency of 355 kHz. The initial rate of sonolytic degradation of benzoic acid followed first order kinetics. The initial rate of degradation increased linearly with increase in initial concentration of benzoic acid. The degradation rate constant was found to be pH dependent. The degradation of benzoic acid was favored at lower pH. At lower pH this solute can be degraded both inside the bubble by thermal cleavage and at the bubble/water interface by the reaction of **OH** radicals. At higher pH this solute can react with **OH** radicals only in bulk solution. The intermediates formed were analyzed with analytical techniques such as high performance liquid chromatography, mass-spectrometry and total organic carbon analysis. During the sonolytic degradation of benzoic acid monohydroxy substituted intermediates were observed. Further **OH** radical attack on the monohydroxy intermediates led to the formation of dihydroxy derivatives. Continuous hydroxylation of the intermediates led to ring opening followed by complete mineralization.

[1] M. Ashokkumar and F. Grieser, Reviews in Chemical Engineering, **15**, 41-83, 1999.

Aggregation of Latex Particle Mixtures

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The main objective of this project has been to explore routes for the controlled flocculation of mixtures of polymer latex particles. Two types of particles have been prepared and characterised. The first were functionalised with carboxylic acid groups (type A), and the second with oligomeric ethylene oxide (EO) chains (type B). For both types, two latex sizes were prepared, one typically $< 70\text{nm}$ and the other $> 300\text{nm}$.

Flocculation results after mixing type A and type B particles, at low pH values (such that the carboxylic acid groups are largely in the protonated form). Under these conditions there is a H-bond interaction between the $-\text{OH}$ of the carboxylic acid groups and the ether oxygen of the EO groups, which leads to heteroaggregation of the A and B type particles. Mixtures of different size ratios of the A and B particles have been investigated.

For the hetero-aggregating mixtures (i.e. type A + type B particles) rheological measurements, namely shear stress - shear rate measurements (leading to Bingham yield stress values) and creep recovery experiments have been carried out, also leading to yield stress values. These have been investigated as a function of pH, ionic strength, number concentration ratio, temperature, time and order of mixing.

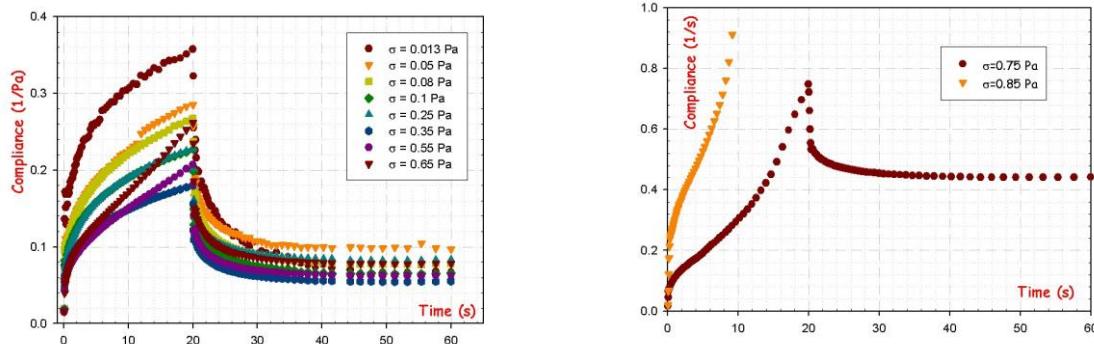
In a creep experiment, an instantaneous stress is applied to the sample and the change in the compliance (i.e. the ratio strain over stress) is observed over time. When the stress is released, some recovery may be observed as the material attempts to return to its original shape.

Creep Recovery Experiment:

Mix between Big PAA Particles (400 nm) and Big PEG Particles (300 nm)

$\text{Fn}=0.5$, $\phi_{\text{total}}=0.33$

0.08M KCl



In the above figures, we can see that the material is behaving viscoelastically. At low stress values (left-hand figure) the material recovers after removing the stress. However, increasing the stress to $\sigma = 0.75 \text{ Pa}$ and beyond (right-hand figure), the material does not fully recover (i.e. some irreversible flow has occurred). The yield stress is therefore $\sim 0.75 \text{ Pa}$.

Catalytic Conversion of Low-Density Polyethylene into Liquid Hydrocarbons

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Plastics have found many applications in the modern society. This is due to their high level of convenience of application and their relatively low price compared to conventional materials such as steel. With the advance of research and technology, these applications and their productions are on a rise and subsequently disposal of these plastics waste is also increasing steadily, causing major environmental concerns. As an alternative for disposal, recycling has been suggested via various methods, and chemical recycling is considered the best alternative.

The most promising alternative to plastics waste recycling up to date is catalytic recycling. Unlike thermal recycling, using catalysts as an aid requires less energy and forms valuable hydrocarbons in the gasoline range, eliminating the requirement for further processing. Extensive works have been published using acid catalysts, mainly amorphous $\text{SiO}_2\text{-Al}_2\text{O}_3$, and a number of different zeolites. Such catalysts have been used because of their known ability for carbon-carbon bond cleavage and their acidic properties. In this paper, we report on the catalytic cracking of low-density polyethylene (LDPE) over three types of layered double hydroxides (LDH) base catalysts: MgAl, NiAl, and ZnAl. LDH constitutes a class of layered compounds, resembling the naturally occurring hydrotalcite and are generally considered complementary to the clays in that they contain positively charged layers and anions in the interlamellar space. These hydrotalcite compounds are generally described by the empirical formula $[\text{M}^{2+}_{1-x}\text{M}^{3+}_x(\text{OH})_2][\text{A}^{m-}_{x/m}\text{.nH}_2\text{O}]$, abbreviated here after as $[\text{M}^{2+} - \text{M}^{3+} - \text{A}]$, where x may vary from 0.17 to 0.33, depending on the particular combination of di- and trivalent elements M^{2+} and M^{3+} . A represents the m -valent anion necessary to compensate the positive charge.

The catalytic cracking of low-density polyethylene (LDPE) was carried out in a semi-batch operation at 350°C and 400°C using solid basic MgAl, NiAl, and ZnAl Layered Double Hydroxides (LDH), and compared with thermal degradation and catalytic degradation using HY-Zeolite. The polymer to catalyst ratio used in this study was 100:1. Thermal degradation of LDPE at 350°C was negligible (< 1% conversion). The order of LDPE cracking was as follows: NiAl > HY > ZnAl > MgAl > Thermal. However, HY produced lighter products, mainly attributed to its high acidity. All reactions produced low amounts of gaseous product (< 5 % wt), which is beneficial because hydrocarbon liquid products are considered more valuable. Catalytic degradation of LDPE using LDHs showed lower coking and deactivation compared to HY. Rapid deactivation of HY was observed due to its high occurrence of hydrogen-transfer secondary reaction. Overall, NiAl and ZnAl-LDHs showed potential catalytic properties for the degradation of polyethylene.

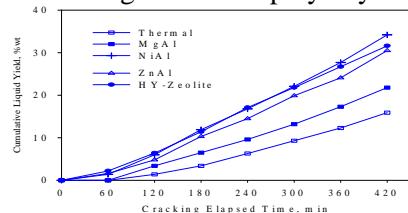


Fig 1. Rate of liquid product collected from LDPE thermal and catalytic cracking at 400°C.

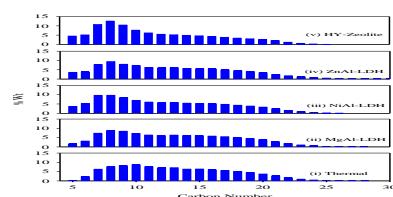


Fig 2. Liquid Product distribution from thermal and catalytic cracking of LDPE at 400°C.

Growth of Multilayer Films: Effect of pH on Assembly of Weak Polyelectrolytes

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The buildup of multilayers by the layer-by-layer (LbL) technique involves the alternating adsorption of oppositely charged polyelectrolytes. With each adsorption step, the surface charge is reversed and this allows adsorption of the next layer of polyelectrolyte. This is a versatile method that allows multilayer construction with controllable thickness, composition and chemical functionalities. The simplicity of this process has attracted significant interest. Functional multilayer films and coated colloids have been fabricated to incorporate various materials such as dyes, proteins and inorganic and metallic nanoparticles.

While the LbL method is applicable to colloids, colloids coated with weak polyelectrolyte multilayers tend to be less stable than those coated with strong polyelectrolytes. In this work, a copolymer of styrene and maleic anhydride, PSSMA (4-styrenesulfonic acid-co-maleic acid) is assembled into thin films and characterized by using QCM (Quartz Crystal Microbalance), UV-Vis spectroscopy and AFM. PSSMA is particularly interesting because it has styrene-sulfonate side groups that are strongly charged and maleic anhydride side groups that can be charged or uncharged, depending on pH conditions. Consequently, the styrene-sulfonate group may provide stability to the multilayer films while the carboxylic acid groups can be used to alter multilayer properties by adjusting the extent of ionization.

PSSMA is assembled into thin films with polymers of varying charge densities, such as PDADMAC (poly(diallyldimethylammonium chloride)), which is permanently charged, and PAH (poly(allylamine hydrochloride)), which has a pH dependent ionization degree. The growth of multilayer films is explained in terms of electrostatic interactions and also secondary interactions, such as hydrogen bonding and hydrophobic interactions. These factors together affect film thickness, ionic crosslink density, chain conformation and internal and surface composition.

Abstracts: Plenary Lecture



Poking Around and Stirring Things up: AFM and rheology of colloidal and biological systems

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Particles dispersed in liquids are common place in every day consumer products such as foods (e.g. tomato ketchup), paints, and cleaning fluids, as well as in more specialised fields such as agrochemicals and pharmaceuticals. In these products a certain property of the fluid is required, for example in non-drip paint we wish the paint to be non-drip when on the brush, but to flow easily on application. It is the forces between the microscopic constituents of materials, which to a large extent determine the macroscopic properties. For example it is the differences in bonding between the carbon atoms which determines the different physical properties of carbon and graphite. The same is true in colloidal systems. In colloidal systems there are three common types of interactions between particles, van der Waals forces, electrical double layer forces and steric forces. In this talk, examples as to how these forces can be measured, modified and even manipulated will be given and how these interactions affect the bulk properties of colloidal systems, such as the rheology will be demonstrated.

Finally, applications of this type of technology to measuring interactions in biological systems, such as recognition interactions and cell adhesion, which at first glance seems very different, will also be described.

Abstracts: Posters



Optically Addressable Microcapsules for Controlled Drug Delivery and Release

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Drug carriers have the potential not only to enhance the therapeutic index of a drug, but also to enable drug delivery to be tailored to individual patients. Nano- and microcapsules are especially well suited to this application because of their unique physicochemical properties.

This work focused upon two particular aspects of the formulation of capsular drug carriers. Firstly, the modification (using lipids) and functionalisation (using antibodies) of the capsule surface to achieve biocompatibility and biospecificity, respectively. Secondly, the capsule loading and release.

By means of various microscopic techniques and core dissolution experiments, it was found that a more homogeneous lipid coating is obtained when the lipid bilayer is assembled via the adsorption of lipid molecules from a saturated solution, rather than via the adsorption and spreading of preformed lipid vesicles. In addition, it was found that mouse immunoglobulin G (IgG) can be coupled to the lipid bilayer, but to confirm this coupling using fluorescently labeled anti-mouse IgG, it is necessary to first wash with bovine albumin serum solution as the secondary antibody's binding is non-specific. Using fluorescein isothiocyanate (FITC) labelled dextran as the model drug, it was observed that the loading of multilayer poly(sodium styrenesulfonate)/poly(allylamine hydrochloride) microcapsules occurs to roughly the same extent at pH 3 and pH 5 and that a large proportion of the capsules are damaged in the process. Finally, the use of fluorescence spectroscopy demonstrated that the coating of FITC-dextran loaded capsules with colloidal gold for the purposes of laser-induced release significantly impacts the fluorescent properties of the system.

Effect of wildfire in the Victorian high country on soil and water quality

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Charcoal and ash residues collected in the Mt. Beauty region after the 2003 alpine bushfires in NE Victoria were characterized by scanning electron microscopy, diffuse reflectance infrared spectroscopy (DRIFT), X-Ray diffraction, potentiometric titration and surface area analysis. The leaching of humic substances from these residues into water over a period of two weeks was measured at pH 4.5, 7.5 and 8.5, with leachates characterized by DRIFT spectroscopy. The charcoal was a highly porous and amorphous material with a high surface area ($403 \pm 2 \text{ m}^2 \text{ g}^{-1}$). It was predominately organic with high aromaticity. The ash was a heterogeneous mixture of smaller particles that had a lower surface area ($15.6 \pm 0.2 \text{ m}^2 \text{ g}^{-1}$) even though it consisted of smaller particles. The ash was more oxidised and acidic and contained a significant mineral component and was approximately 150 times more soluble in water compared to charcoal, with the aqueous concentration of humic material increasing as pH increased. The nature of the leached material changed over time, with leachates becoming less polar as the leaching time increased. The leaching results suggest that bushfire residues, particularly the ash component, will be a significant source of soluble organic material in fire affected soils and water.

Aqueous Metal Adsorption onto Biological Substrates: The Effect of Surface Chemistry

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Heavy metals released into the environment through industrial activities tend to persist indefinitely, accumulating through the food chain. This can ultimately lead to serious environmental and public health problems ranging from mild hypertension to early termination of pregnancy, genetic malformation and/or death of adults. One of the main causes of heavy metal pollution is the release of heavy metal-containing industrial effluent into waterways. This is often as a direct result of inefficiencies associated with existing heavy metal removal processes. Consequently, the efficient and effective removal of heavy metals has become a significant problem for industry. Over the past few years, adsorption of metals onto substrates of biological origin (biosorption) has been widely investigated due to their potential as a renewable source of a substrate which is highly efficient and probably effective.

In this study, three fungal substrates (*Mucor rouxii*, *Rhizopus stolonifer* and *Pycnoporus cinnabarinus*) were used to adsorb aqueous metals from solution as a function of pH. It was found for Zn(II), for example, that the amount of adsorption at a given pH was much greater for the fungus *Mucor rouxii*. Two structural components of fungi (chitin and chitosan) were also investigated for their adsorptive properties.

Spectroscopic techniques are being used to characterise the surface composition and chemistry, and the adsorptive properties are being related to these properties. Specifically, focus has been on the difference between *Mucor rouxii* and the other fungi in terms of their carboxyl and nitrogen nature and content.

New Evidence for DLVO Theory based in Binary Particle Interactions.

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Since its inception in the 1940's DVLO theory has allowed the interaction of colloidal particles in aqueous solutions to be quantitatively analysed. A major drawback with the theory is that, while it has been demonstrated to be appropriate for binary applications, the theory is unable to cope with systems containing more than two particles. For this reason, the research undertaken in this study concerns a system that, in almost all cases, contains only two particles: the male scrotum. While some correction must be made for the interaction of the testes and the vas deferens, we have demonstrated for that for the most part, the testes behave in a manner consistent with that suggested by classical DLVO theory regardless of the testicle size, shape or critical sperm volume (CSV). The principle methods of investigation include the response of the testes to different levels of system perturbation: in most instances, the subject was asked to cough and the time to equilibrium is monitored. However, in some cases, more extreme methods of excitation were required. These include response to direct current applied via electrode stimulation, motivation with high power optical tweezers, and in the worst cases the novel "pull and twist" technique recently developed at this institution. Practical results will be analysed in conjunction with established DLVO theory.

Microemulsion Templated Nanoporous Silica Adsorbents for Separations in the Pharmaceutical Industry

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In 1992 researchers at the Mobil Research and Development Corporation succeeded in developing mesoporous molecular sieves. For the first time inorganic materials were available that had narrow pore size distributions and regular well defined pore systems in the mesopore range. The materials were synthesised by combining a silica source with a surfactant in an aqueous environment and a liquid crystal templating mechanism was proposed for their formation. These materials offer promise for biomolecule separation, extending the molecular sieving range of zeotype materials into the realm of macromolecules or as high capacity separation support materials making use of their uniform pore size, high surface areas and pore volumes. Their discovery has also led to further developments in new and larger pore sized materials based on similar synthesis concepts, one of the more remarkable of these is the microemulsion templated nanoporous silica foams. These materials consist of spherical voids interconnected via windows, forming a highly porous 3-D network.

We plan to use these microemulsion templated materials as affinity chromatography supports for the separation of antibodies using Protein A from *Staphylococcus aureus* as the affinity ligand. Protein A affinity chromatography is the preferred method for separation and purification of monoclonal antibodies from clarified cell cultures and with the increasing demand for monoclonal antibodies resulting in higher concentrations in feedstocks, a need has arisen for higher capacity supports.

In this presentation we outline the structural characteristics and synthesis parameters of these materials and provide some preliminary results on a post synthesis vapour phase functionalisation to covalently attach 3-aminopropyltrimethylsilane moieties. The amino functionality is important as it is the first step to the coupling of Protein A, the affinity ligand.

AFM at Deformable Surfaces in the Presence of DDAPS

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Emulsion systems are of great interest in many areas such as: food technology, petroleum science, solvent extraction, chemical manufacturing, cosmetics, *etc.* Instability of emulsion systems leads to droplets coalescence and separation. The desired stability of colloidal systems may vary from industry to industry. In some industries such as solvent extraction, oil recovery and membrane separations, fast coalescence is highly preferred to increase the efficiency of the industrial processes. In other industry such as food industry and cosmetics, highly stable emulsion systems are required to prolong the product's life and quality. Understanding the interfacial forces is crucial in controlling the stability in these colloidal systems. Direct measurement of the interfacial forces between a rigid surface and single deformable surface, using Atomic Force Microscope (AFM) has become routine.

The interfacial forces between the oil-water interface and a silica sphere were investigated in the present of zwitterionic surfactant, dodecyldimethylammoniopropane sulfonate (DDAPS). Both electrostatic repulsion and van der Waals attraction were observed for all concentrations. The reducing of surface tension caused an increase in deformation to the oil-water interface and led to the decrease in the force curve slopes. However, step like jump-in behaviour in the force curves cannot be explained by DLVO theory. Interactions between two hard surfaces and two soft surfaces in the present of DDAPS will be investigated to clarify the formation of aggregates in the solution or monolayer swelling on the silica sphere.

Sonochemistry of Phenol and Substituted Phenols in Aqueous Solutions

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When ultrasound of sufficient intensity passes through a liquid medium, such as water, it initiates the phenomenon of cavitation. This involves the formation, growth and implosion of micro gas bubbles in a liquid. The violent collapse of these bubbles results in the near adiabatic heating of the gas and vapour inside the bubbles leading to very high, localised, temperatures and pressures. Due to the extreme temperatures reached on collapse, these events are normally referred to as “hot spots”. In aqueous solutions, homolysis of water molecules (reaction 1) takes place within these hot spots to produce highly reactive H· and OH· radicals and other secondary oxidants.



A number of investigations have been reported on the use of these ultrasound generated radicals to initiate a range of chemical reactions [1].

Orica Adhesives & Resins manufactures formaldehyde-based resins, which are produced by the reaction between phenol and formaldehyde in aqueous solutions. When the reaction is completed, the solution is left with unreacted phenol and formaldehyde. The aim of this study was to make use of ultrasound generated radicals to convert unreacted phenol into useful or less toxic chemicals. Sonochemical experiments, carried out at 20 kHz and 358 kHz, suggested that OH· radical attack on phenol led to the formation of dihydroxy and trihydroxy substituted intermediates. Sonochemistry of these intermediates were also investigated in order to identify the reaction pathways involved in the sonochemical degradation of phenol.

[1] M. Ashokkumar and F. Grieser, Reviews in Chemical Engineering, **15**, 41-83, 1999.

Fluid Flow in Thin Films

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An understanding of the flow properties of simple liquids in a confined space, in particular the boundary condition at the solid-liquid interface, is important to the development of many micro-scale devices. The conditions under which a simple liquid can exhibit ‘boundary slip’ (a nonzero flow velocity) at a solid surface have been a matter of debate for many years. A technique for investigating the flow of confined liquids is proposed, which is based on measuring the rate of flow through a channel of sub-micron dimensions. Parallel cylinders of cleaved mica will form the channel walls. Measurement of the gap width can be done with high accuracy (0.2nm) using the FECO (fringes of equal chromatic order) technique often employed in SFA (surface force apparatus) experiments. The anticipated capabilities of the technique will be presented, along with some of the foreseen developmental challenges.

Sphalerite and Silica Aggregation Studies: UV-VIS Technique Development

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Particles interactions and their subsequent aggregation or dispersion state is an important aspect in mineral separation. This is due to the adsorption (attachment) of very fine particles onto mineral surfaces, which can affect mineral flotation amongst other industrial processes. The interactions between minerals can be studied according to the DLVO theory, which considers only electrostatic and van der Waals interactions [Toikka et al., 1997]. However, there are a large number of systems that will not fit this theory in the understanding of particle stability [Sjollema and Busscher, 1990]. For those, a modified DLVO theory can be applied.

Suspension stability or dispersibility can be studied by several methods that include optical microscopy, scattering methods, electronic counting technique and rheology, among others. The first three techniques involve, in some way, the counting of the number of particles present in a suspension and the last technique involves the correlation between viscosity and shear rate [Laskowski and Ralston, 1992]. The present work is aimed at the development of the UV-VIS technique. This method can be used as a tool for quantifying the amount of colloidal silica particles that can be adhered to a sphalerite surface.

Zeta potential measurements were used to predict the pH at which aggregation could be greatest. Based on this data, it was possible to define that at approximately pH 6.5, silica and sphalerite can achieve maximum aggregation. This prediction was confirmed using UV-VIS to quantify particle aggregation and it was found that approximately 43% of silica could be attached to the sphalerite surface by changing the pH of the suspension. However, this methodology still needs further development due to sphalerite dissolution products that can interfere in the absorbance measurement. As an example, the addition of acid prior to the measurement is a point of consideration for the present technique.

As a preliminary conclusion, silica attachment to sphalerite surface can be explained by DLVO theory. At a pH lower than the sphalerite iep (pH 6.5), the aggregation would be less intense. However, maximum of aggregation would be reached at a pH close to iep, due to the absence of electrostatic repulsive forces.

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Fate of Selected Organophosphate Pesticides in the Natural Aquatic Environment

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Organophosphate pesticides are used extensively world wide as fungicides, insecticides, herbicides and animal pesticides. This group of pesticides has important advantages over the now banned organochlorine class of pesticides such as DDT, which include an increased decomposition rate and a decreased tendency to accumulate in the biological food chain. Two widely used pesticides are chlorpyrifos and malathion, both of which undergo abiotic (hydrolysis and photolysis) and biotic (microbial) degradation in the environment. For both of these pesticides hydrolysis has been reported as being the major degradation route in both the soil and water environments. The reported half-lives (i.e. the time required for half of a specified amount of pesticide to decompose) for these compounds in the aquatic environment is typically between 2 and 10 weeks.

Sorption of these compounds and their metabolites onto soil and sediment components can play a significant role in their persistence, toxicity and transport of residues in the environment. Therefore, sorptive interactions will determine if residues and their metabolites are available for plant uptake and leaching to surface and ground waters. Thus it is critical to understand the sorption mechanisms between the pesticides, their metabolites, soil and sediment components and organic matter.

The broad aim of this project is to not only investigate how chlorpyrifos, malathion and their major metabolites interact with soil and sediment components, but to also study if these pesticides, and in particular their metabolites are present in waterways near application sites and at what concentrations. This work is therefore significant as regulatory bodies and natural resource managers become increasingly aware and concerned about the levels of pesticide metabolites present and their possible detrimental effects on non-target organisms.

Coarse Particle Flotation

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The recovery of the particles in a mineral flotation process is strongly influenced by both the particle size distribution and contact angle. Coarse particles are often not recovered during the flotation process. The most important factors governing coarse particles response to flotation are detachment and surface heterogeneity (and consequentially the contact angle). The kinetic energy of a particle is proportional to the cube of its diameter. Inertial forces have a dominant role for coarse particle flotation.

Each particle size fraction requires a minimum contact angle in order to float. Above these cut-off values in contact angle flotation is possible. This threshold for flotation was detected for average particle size varying from 10 to 120 μm . For intermediate and coarse particles (60 to 120 μm respectively), flotation recovery increases strongly at contact angles higher than the cut-off. Using this approach we can identify a flotation domain. The cut-off values mentioned above were obtained using a modified Hallimond tube with minimum turbulence (mostly laminar flow).

The aims of this project are:

- 1 - To determine the effect of particle size on the flotation rate constant and recovery of coarse particles (in the size range of 50-250 μm).
- 2 – To determine the effect of particle size and surface heterogeneity (which influences contact angle) on particle detachment under turbulent conditions.
- 3 – The development of methods to modify the energy dissipation to improve coarse particle flotation, without compromising fine particle collision efficiency.

As model particles quartz methylated to different extents will be used, as well as silicon carbide oxidized to different extents. In the present project we intend to extend the study of the contact angle cut-off values for coarse particles under turbulent conditions, such as found in the Rushton turbine cell, which is similar to the industrial mechanically agitated cells.

Rupture of a Thin Liquid Film

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Thin liquid films occupy a unique place in the realm of surface colloid science and are common throughout many industrial processes and applications. They model the stability of molecular interactions and are special in respect to their small volume systems, with altered thermodynamics and kinetic properties with respect to the bulk. The stability of a thin liquid film is essential in many processes, including paints, cosmetics, electronics and mineral flotation.

The stability properties of these fluid films depend on the nature of the film phase and the character of each of the two neighbouring bulk phases. The ability to influence the film stability and film rupture can in part rely on the forces perpetuated from the solid/liquid interface, an interface that is rarely void of heterogeneities. Whether these heterogeneities are chemical, morphological or a mixture of both these, their impact can be studied by carefully structuring of both these factors.

This project will be approached in four phases, with each phase providing further clarification of the ultimate goal. Phase one will involve a carefully characterisation of a range of select metal oxide interfaces. The second phase will involve flotation tests to further test observations from the previous phase. The third phase will examine surface forces defined under an array of conditions by the use of atomic force microscopy with the fourth phase involving the rupturing of a thin aqueous film in a controlled manner studied with a thin film balance. With the overall aim of the project to provide a comprehension synopsis of the forces of stability for asymmetric thin liquid films and to provide understanding of the rupture mechanisms for unstable films with focus on the impact of heterogeneities of the interface.

Rheological Behavior of Nano-Sized Alumina Particles in Monovalent Electrolyte Solutions

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The forming of nanostructured materials from nano-sized advanced ceramic particles have received significant interest in the recent years due to the favorable material properties that are suitable for various emerging technological applications such as nanotechnology, ceramic bone grafting, catalyst supports and ceramic membrane filters. Among the known forming technique, the well established colloidal processing approach has been successfully applied to form the nanostructured products with improved mechanical properties, strength reliability and homogeneous microstructure. This route involves the manipulation and control of interparticle forces when the nano-particles are dispersed in suitable liquid as a suspension. Therefore, it is of paramount importance to have a sound fundamental understanding of how one may alter the rheological behavior of the nano-particle suspension by manipulating and controlling the surface forces between nano-particles in liquids to fabricate a more reliable high performance advanced ceramic components inexpensively.

Surface forces interactions between particles are much more pronounced than inertial and gravitational forces for particles in the nano-scale region. This is due to greater influence of surface forces since the particle size and range of the forces are nearly the same. Thus, the effect of surface forces such as the electric double layer, van der Waals and ion specific forces on the control of rheological behavior becomes significantly important as the size of the particles decreases.

The aim of our research is to investigate the specific effects of different monovalent electrolytes (following the Hofmeister monovalent cations sequence: Li⁺, Na⁺, K⁺ and Cs⁺) on the rheological behavior of alumina nano-particle suspension. The effect of ion specific forces will be investigated over a wide range of salt concentration; from 0.05 to 0.5 M.

Two different commercially available nano-sized γ alumina powders with an average particle size of 11 and 44 nm are used in this study. The shear stress and shear viscosity as a function of shear rate were determined at three pH values (4.5, 9 and 12) which corresponds to positive, uncharged and negatively charged surfaces. The dynamic oscillatory shear measurement (amplitude sweep method) was used to probe the viscoelastic behavior of the suspension without the influence of hydrodynamic effects at the similar pH values. Shear yield stress and ζ potential of the alumina suspension was measured using the vane and electroacoustic method respectively as a function of pH ranging from pH 4 to 12.

Simulation of Sedimentation by Discrete Element Method

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The aggregation of particles forming larger structures plays a very important role in many industrial processes. Floc microstructure affects the formation of sediments, filter cakes and other particulate collectives and has a large impact on the design and operation of solid – liquid separation equipments. However, **there is no fundamental understanding of how changes in floc properties** such as the size, fractal dimension and degree of looping in floc structure affect the operating condition of downstream processes.

In order to clarify the dynamic behaviour of flocculated particles, experimental work is not enough because in most cases fractal aggregates represent very fragile mechanical structures, which can be easily disrupted as a result of external forces, resulting from measurements - it is extraordinarily difficult to measure the movement of individual particles without disturbing the system. However, the unified way to describe properly the suspended matter has not been yet established since the macroscopic behavior of flocculated substance depends on the mesoscopic mechanics of individual particles in a complicated manner.

The project is approached with computer simulations to examine in detail how the structure and properties of the sediment bed (or cake) develop over time as a function of both the properties of the flocs that form the sediment and the applied consolidation pressure. Properly validated simulation will allow the measurement of properties that are not accessible in real physical systems.

Novel Particle Characterisation Techniques

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Focussed beam reflectance measurement is a technique that shows some promise as a technique for the online characterisation of floc size and structure. A scanning laser is focused into process fluid and the reflected intensity is measured as a function of time. This light intensity signal is normally processed to return chord length measurements which contain combined information on particle size and shape.

This study shows that the unprocessed signal can be expected to encode information about the texture of the particle being scanned in addition to size and shape. For the purpose of characterising floc structure, the power law exponent can be recovered from the Fourier transform of the unprocessed signal and this in turn is related to the mass fractal dimension of flocs.

The technique is demonstrated with computer generated flocs and with flocculated polystyrene latex of a primary particle size of $5\text{ }\mu\text{m}$ in electrolyte solution. A mass fractal dimension of $D_f=2.5$ was determined by the volume obscuration method for the latex flocs.

The mass fractal dimension results obtained through simulation studies of scanning of computer generated flocs demonstrate that a correlation exists between the power law exponent and the mass fractal dimension. Loose aggregate structures of mass fractal dimension $D_f=1.54$ returned a power law exponent of -1.77 whilst a compact aggregate structure of mass fractal dimension of $D_f=2.39$ returned a power law exponent of -1.92.

Results obtained through experimental laser scanning of polystyrene latex flocs of mass fractal dimension of $D_f=2.5$ returned a power law exponent of -2.00.

Effect of Cationic Polyelectrolytes on the Forces of Interaction within a Sand Filter

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Oocysts of *Cryptosporidium parvum* have been associated with several waterborne outbreaks of gastro-enteric disease. Currently, one of the main methods for pathogen removal from a water supply is provided by sand-bed filtration. There have been very few studies investigating the forces involved in the interaction between sand particles and oocysts, and what factors have the greatest influence on these interactions.

This poster examines the effects of cationic polyelectrolytes on the forces of interaction between oocysts and silica surfaces, used as representatives of filter media particles. Specifically, Atomic Force Microscopy (AFM) was used to examine how the presence of a commonly used cationic polyelectrolyte, LT35 (whose characteristics include high molecular weight, and high cation density), affected both the attachment of oocysts to the silica surface and subsequent detachment forces. The effect of variations in dosing schedule have been investigated. Results suggest that the most effective retention of the oocysts occurs when a threshold force is applied between the oocyst and a silica surface pre-treated with cationic polyelectrolyte. The data obtained highlight the role played by both steric and electrostatic type interactions in modulating the repulsion and/or attraction between the oocysts and the model sand particle used as a silica colloid probe.

This study contributes to a broader project focused on assessing the effects of common physico-chemical treatment practices on pathogen inactivation / removal from raw water samples. The results of this study may be applied to the development of operational strategies for maximising pathogen inactivation / removal during conventional drinking water treatment.

Optimising Flocculation And Dewatering Behaviour Of Clay Dispersions

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In the present work the effect of shear on the dewatering of smectite dispersions at pH 7.5 has been investigated using high molecular weight anionic ($\approx 4.9 \times 10^6$ Da) and non-ionic ($\approx 4.5 \times 10^6$ Da) polyacrylamide (PAM) flocculants. “Controlled dispersion” of Na-exchanged smectite particles was facilitated by the use of hydrolysable metal ion (Ca [II]), leading to rapid cation exchange with the Na originally present, suppression of swelling and reduction of electrokinetic zeta potential.

Variation in shear during flocculation agitation rate (100 – 500 rpm) and agitation time (15 s – 60 s) influenced settling rates significantly. The settling rate increased to a maximum with increasing agitation rate up to 200-300 rpm before decreasing upon further increase in agitation rate up to 500 rpm. Peak settling rates were greater for 15 s than 60 s agitation times and increased with increased polymer dosage. Lower agitation rates were required to produce optimum settling rates for longer agitation times.

Flocculation with anionic PAM produced large space filling flocs exhibiting higher yield stress and lower consolidation than with non-ionic PAM. Increasing polymer dosage from 250 to 500 g polymer/t solid did not cause an increase in pulp shear yield stress, however consolidation decreased from ≈ 23 to 20 wt% solid for both polymers used. Applying shear to pre-sedimented pulp decreased yield stress and improved pulp consolidation significantly, with a maximum ≈ 28 wt% solid. These trends appear to be related to polymer functional group influences on solution conformation and subsequent orthokinetic, non-equilibrium bridging flocculation. A clear relationship appears to exist between polymer structure-dependent chain flexibility/conformation, pulp yield stress and consolidation behaviour.

Dewatering of Mixed Silica and Iron Oxide Suspensions

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Australian mineral processing plants generate approximately 40 million tonnes of waste tailings per year. Several billion tonnes of mineral waste dispersions are dumped in impoundment dams every year, still containing millions of tonnes of water. The current methods available for the dewatering of silica and iron oxide suspensions fall short of acceptable in terms of water recovery for recycling. Water conservation has become a growing concern over the last decade, making the discovery of a more efficient dewatering technique a high priority.

Although improved thickener technology has shown increases in the effectiveness of fast sedimentation in recent years, current dewatering methods still only allow the industry to produce a final solid content of approximately 25 to 60 wt%. A more acceptable and desirable result would be to increase the solid loading to at least 75 to 85 wt%. To achieve this, investigations must be conducted into the rationale behind the interfacial chemistry, particle interactions, and what it is that makes the particle suspensions retain so much water.

This project focuses on the behaviour and interactions of silica and iron oxide suspensions with polymeric flocculants (PEO and PAM). We will use the information gathered by electrokinetic zeta potential, dynamic light scattering, adsorption isotherms, rheology and direct interaction force measurements in the atomic force microscope (AFM) to gain further insight into the particle interactions in the hope to achieve more efficient dewatering. Mixed silica and iron oxide systems will be investigated. The rheology, direct interaction force and settling tests data will be correlated as a function of dispersion pH.

Fine Particle Flotation

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Fine particle flotation is becoming more and more significant all the time, for the demand for minerals is increasing and the extent of available ore is decreasing. The recovery of mineral particles by flotation is most successful in the size range from 10-100 μm in particle diameter. The recovery rate for fine particles drops steeply due to their lower momentum in collisions with bubbles so great amounts of valuable minerals are lost in tailings.

The majority of research has dealt with larger size particles, which either neglect the fine particle range, or assume the trend associated with larger particles remains applicable for fine particles. The effect of the electrostatic, van der Waals and hydrophobic interactions is more significant when the particle size is small because particles are almost “weightless”. Furthermore, diffusion starts to play more and more important role when the particle size decreases.

This study investigates the effect of small bubbles (0.1-0.6 mm) on the flotation rate of fine particles ($0.1\text{-}10 \mu\text{m}$). The effects on bubble surface mobility, particle charge and hydrophobicity on the bubble-particle collection efficiency are being carried out. Experiments with single bubbles and bubble swarms and in Rushton turbine cell with single and mixed minerals will provide data for model development to calculate the flotation rate constant for fine particle flotation. The main goal is to increase the flotation rate of fine particles without adversely affecting the flotation of intermediate and coarse particle.

Zeta Potential and Rheology of a Ceramic Glaze: the Effect of Ionic Surfactant Adsorption

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The stability and rheology of a ceramic glaze slip is important to control the various stages of the ceramic glazing process. Slurry stability is vital since the settling of particles results in poor performance and products. Control of the rheological behaviour of the slip is particularly important during the application processes such as spraying, dipping and waterfall techniques. The addition of a surfactant as a deflocculant can be used to stabilize such ceramic suspensions.

The aim of this project is to study the stabilization of mixtures of four types of particles of varied surface chemistry (feldspar, limestone, quartz and kaolin) using surfactants as dispersants. The effect of both a cationic and an anionic surfactant (cetylpyridinium chloride (CPC) and sodium dodecylbenzenesulfonate (SDBS)) on the zeta potential and rheology of the ceramic glaze suspensions have been studied as a function of pH and ionic strength. Adsorption isotherms were measured at room temperature (25°) using the depletion method. The zeta potential measurements were performed on equilibrated 10 wt% solids suspensions with a Colloidal Dynamics, AcoustoSizer II. Rheology measurements were performed on equilibrated 60 wt % (48 vol%) suspensions with a Bohlin CVO 50 Rheometer with concentric cylinder geometry.

Additional stability can result from either electrical double layer repulsion or steric repulsion depending on the surfactant type, concentration, ionic strength and pH. Underdosing may result in poor stability due to hydrophobic attraction between particles partially covered with adsorbed surfactant, while overdosing may result in poor stability due to depletion flocculation caused by micelles in the solution. We will show that the situation is particularly complicated for glazes containing limestone (calcium carbonate) and anionic surfactant at moderate to low pH where calcium carbonate solubility increases dramatically. The anionic surfactant and calcium ions can form complexes that are poorly soluble and strongly adsorb to the surface of the particles. The resulting coating of precipitated complex produces a significant repulsion and stable suspensions. Zeta potential measurements and adsorption isotherms are used to interpret the rheological behavior.

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Chain-length Dependence in Living Polymerisations

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Recent progress has been made in the application of living polymerisation techniques (such as reversible addition-fragmentation chain transfer, RAFT) to emulsion polymerisation by developing a good mechanistic understanding of the systems. The free-radical polymerisation of hydrophobic monomers in emulsions is the method of choice for implementing most free-radical polymerisations on an industrial scale, particularly in producing surface coatings. Resulting products from classical emulsion polymerisations typically have quite wide distributions of molecular weights, and even relatively simple architectures such as A–B blocks are impossible to synthesise. RAFT polymerisation techniques have been the focus of a great deal of recent work as they allow unprecedented control over the molecular architecture for polymers made by free-radical polymerisation. RAFT/emulsion polymerisations have considerable technical potential: to “tailor-make” material properties, to eliminate added surfactant from surface coatings, and so on. However, considerable difficulties have been experienced in using RAFT in emulsion polymerisation systems.

A Monte Carlo model is used to show that chain-length dependent termination plays an important role in free radical polymerisation systems containing RAFT agents. As dormant chains are activated through the reversible transfer reaction, the chain-length of the active species changes. By changing the length of the propagating radical, the RAFT agent changes the behaviour of the entire system, through the chain-length dependent termination reaction. The amount of polymer that may be produced before two radicals terminate is studied as a function of system parameters such as the transfer constant of the RAFT agent, the concentration of the RAFT agent and the length of the dormant chains by modelling RAFT-mediated polymerisation. It is found that high transfer constant RAFT agents and short dormant chains exhibit significantly shorter radical lifetimes, and hence have a slower overall rate of polymerisation (seen as a lower average number of radicals per particle) than systems without RAFT agent. Conversely, long dormant chains may lead to an extension of radical lifetimes and an increase in the number of radicals per particle. These effects are due to the change in the chain length of the polymeric radical due to transfer to dormant species, such that significant amounts of short-short termination are seen at low conversions and long-long termination is required at high conversions. This work also suggests a number of experimental techniques, including the use of oligomeric adducts to the RAFT agent, that may offer solutions to the known problems of RAFT-mediated polymerisations in bulk, solution and particularly emulsion polymerisation.

The Effect of Water Treatment Processes upon Sludge Production and Sludge Dewaterability

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Water treatment results in formation of a sludge by-product whose significance is growing as a result of more stringent drinking water quality requirements and rising sludge disposal costs.

An important contribution can be made by specifying operating conditions that will minimise the amount of sludge produced and/or maximise the ‘quality’ of the sludge produced (subject to drinking water quality requirements).

For this a better understanding of the formation and behaviour of floc particles and sludge is essential. Influences include the nature of the raw water, the coagulation and flocculation operating conditions (coagulant type, dose, temperature, etc.), and other (mechanical) processing of the floc particles. The effects will be correlated with sludge character to produce a model that may be used to predict the dewaterability of a sludge produced under given conditions, and hence optimise the process.

The Formation of Excited State Metal Atoms during Acoustic Cavitation: An Effort to Find the Mechanism

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Line emission from excited state metal atoms (e.g., Na^+) can be observed in sonoluminescence (SL) spectra when aqueous solutions containing metal salts, such as NaCl , are sonicated [1-3]. The key reaction steps involved for such emissions to occur are the reduction of the metal ions followed by the excitation of the neutral atoms to higher energy levels. The extreme thermal conditions reached by the (acoustic) cavitation bubbles are more than sufficient for the second process to occur. However, an acceptable mechanism for the first process, namely, the formation of the neutral atoms from the metal ions is yet to be established.

With a view to understanding the metal ion reduction process, I have investigated the influence of various experimental parameters, such as, counter ions, dissolved gas, the presence of volatile solutes, ultrasound frequency, etc. on the intensity of emission from Na^* . An identical intensity of emission from Na^* was observed from aqueous solutions containing 1 M sodium ions with chloride or bromide or iodide as counter ions. However, the emission intensity was affected by counter ions, such as, alkyl sulfonates, alkyl carboxylates. The presence of such surface active counter ions enhanced the Na^* emission intensity. Based on these and other experimental observations, a plausible mechanism for the reduction of metal ions within the cavitation bubbles will be discussed.

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Spectroscopy of Polymer Solutions (Complex Fluids) in Flow

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Complex fluids are composed of polymers, particles or mixtures of both. Complex fluids are integral to life and also everyday activities. Polymers are an essential part of the food we eat and the functions of life. Polymer dynamics have a significant impact upon the properties exhibited by complex fluids. e.g. viscosity.

The aim of this work is to investigate the polymer dynamics, alignment and other effects of the application of shear upon polymers in solution using fluorescence polarisation spectroscopy.

The polymer dynamics of fluorescently-labelled polystyrene solutions in a range of concentration regimes at varying shear rates are to be investigated. The shear is applied in a Couette cell allowing observation of the system in both a cross flow and receding flow regime.

Aggregation And Sedimentation – Shear Experiments With Natural Samples

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The capability of suspended solids in riverine systems to transport associated nutrients, heavy metals and organic pollutants is widely recognized¹. The extent and rate of sedimentation itself is also very important to the ecosystem, as siltation can lead to dramatic alteration of habitat, and suffocation of fish eggs and bottom dwelling organisms². In response, the Victorian EPA program, “Freshwater Sedimentation And Suspended Particulate Program” aims to define a threshold value for total suspended solids.

Sedimentation rates are greatly influenced by aggregation mechanisms like coagulation and bridging flocculation³, which are in turn mainly controlled by the flow regime, solution chemistry and particle concentration^{4,5}. In this project, a Couette flow system⁶, where shear forces and duration can be varied, will be used for investigating these factors. The shear cell is equipped with a sampling port to transfer the aggregates into a settling column, where they can be monitored using a camera with magnifying lenses. This is interfaced to a computer with image analysis software, so that direct measurements of size, shape and settling velocity of aggregates can be undertaken.

The experiments involve variation of salt, organic matter and sediment concentrations and are initially being carried out on three freshwater samples from sites around Melbourne. These sites have different concentrations of suspended solids, humic substances and organic macromolecules. In addition the solution, primary particles and resulting aggregates will be characterized by usual water quality parameters as well as multi-angle light scattering.

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Investigation on the Force Interaction Between a Single Particle and Single Bubble Using the Atomic Force Microscope (AFM)

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Froth flotation is a well-established method used for the separation of valuable minerals from pulps. Nonetheless, the conventional flotation technique is not successful in recovering coarse particles such as those often found in the coal and diamond industry. Several decades ago, researchers began to develop a new separation technique called separation in froth (SIF). In this technique, the pulp is fed directly onto the froth bed generated on the surface of a liquid medium. Unlike conventional flotation where the separation takes place in the pulp-froth interphase, in SIF the mineral particles are in direct contact with the froth. Previous work has demonstrated that the SIF method provides an effective way for separating coarse particles up to a few millimetres in size. In SIF, the surfactant properties and the mineral characteristics (e.g. hydrophobicity, shape and roughness) are the key factors in the interaction of particles and froth. This greatly influences the stability of froth and hence plays an essential role in determining the performance of particles on the froth bed.

A fundamental knowledge of the interaction mechanism between different particles and the froth is necessary in order to develop a flotation model which could be implemented on a mineral processing plant. Different chemical models and theories, such as double layer and van der Waals, as well as hydrodynamic and electrostatic mechanisms have been proposed to describe the SIF technique. However, no single mechanism has been identified and accepted universally to explain the majority of results reported in the literature.

With the advancement in atomic force microscopy (AFM), it is now possible to measure the interaction forces between the particle and bubble directly at the nanometer resolution level. In this work, AFM was used to investigate the force interaction between a single particle and a single bubble. The attraction force of the extending and retracting hydrophobic probe approaching a single bubble was measured and studied at different surfactant concentrations and pH. Glass beads and real minerals, such as pyrite, galena and coal, were used in this investigation to study the effect of surface properties on particle-bubble interactions. These measurements are of great significance, because so far only few attempts have been made to measure the forces between natural minerals and bubbles directly. Furthermore, the hydrophobicity of these materials was modified and the contact angles ranging from 30° to 110° were determined using the sessile drop method. The AFM results will be related to direct measurements of the froth stability and this will provide a fundamental understanding of SIF technique in mineral processing.

Floc Properties and Rheology of Silica Flocculated with PolyDADMAC

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Flocculation of fine particles, using polymeric flocculants, is important in many industrial processes such as wastewater purification and sewage treatment. The flocculant chemistry, solid concentration and method of mixing the solution and suspension control the floc properties such as size and structure. In this study, the effect of floc properties on the sedimentation behavior and the rheological behavior of the sediment bed is being investigated.

The flocculation of dilute colloidal silica particles of 90nm was investigated using small –angle static light scattering. Ranges of fully charged homopolymers of diallyldimethylammounium are used as flocculants, chloride with molecular weight ranging from 10,000 to 1,000,000. Floc structures were found to be mass fractal in nature for polymer -induced flocculation system. Both the fractal dimensions and size of the silica flocs were significantly dependent on polymer chemistry, polymer dosage and the concentration of the initial particles.

Measurements of adsorption isotherms and electrophoretic mobility of the particles will be made at varying concentrations of a range of PolyDADMAC. Results from measurements of adsorption isotherms and electrophoretic mobility will be used to develop a further understanding of the flocculation mechanism, as well as the configuration of adsorbed polymers on the particles surface.

The yield stresses of particulate suspensions control their flow and consolidation behavior. The volume fraction of the sediment will be increased by centrifugal compression of the particle network and the compressive yield strength measured. The shear yield strength of the particle networks will be determined as a function of volume fraction with the vane method. The relationship between floc properties such as size and structure and the strength of the particle network will be presented. A better fundamental understanding of the relationship between floc properties and sediment bed rheology will result in more efficient and profitable minerals processing operations.

Low Temperature Deposition TiO₂ Thin Film by Reactive RF Magnetron Sputtering

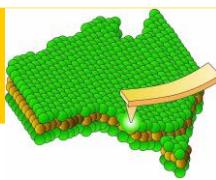
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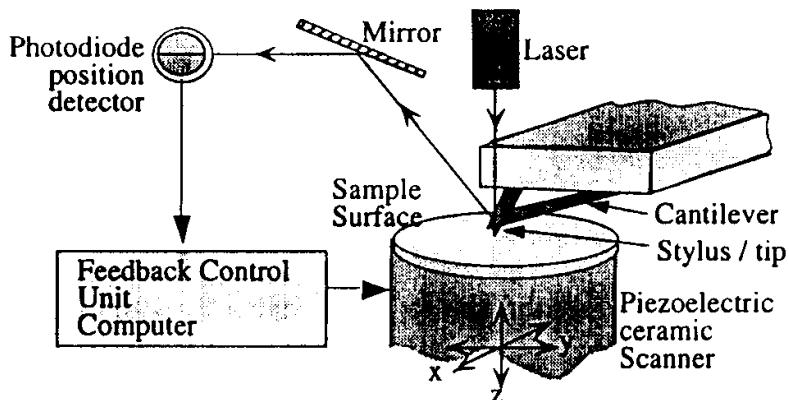
Thin films are generally thought of as the product of vapor atoms or molecules condensing and establishing a permanent residence on a substrate. It was suggested that the charged clusters nucleated in the gas phase and became the main growth unit for a thin film instead of atomic and molecular growth. The aim of this study was to determine whether or not TiO₂ thin film deposition by RF reactive sputtering occurred via this mechanism. TiO₂ thin film was deposited on substrates e.g. glass, silicon wafer etc under RF power of 200 W, 300W and 400W and target to substrate distance of 25mm, 50mm and 200 mm. Crystallinity of TiO₂ thin film was identified with XRD on silicon wafer substrate. Optical properties of TiO₂ thin film were measured using UV-Visible spectroscopy. Results of SEM and TEM were also analysed on the deposited thin film.

Experimental Techniques



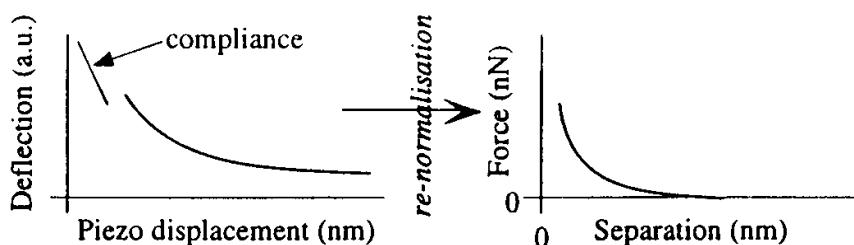
(Atomic) Force Microscopy

The Force Microscope is one of a family of Scanned Probe Microscopes derived from the Scanning Tunnelling Microscope. They all share three attributes: a mechanism for translating a surface in three dimensions typically with a precision on the atomic scale, a probe which is sensitive to a particular surface phenomenon which decays as a function of distance normal to a surface, and a feedback mechanism which can couple the previous two elements. In the force microscope the translating mechanism is a piezoelectric ceramic, and the probe is comprised of a sharp tip affixed to the free end of a cantilever. In its simplest operation as the sample is raster scanned under the tip which traces the contours of the surface causing the cantilever to be deflected. Thus the magnitude of the cantilever deflection is recorded as a function of sample position. Using feedback the piezo can be made to react to maintain the cantilever at a present deflection, and hence force. In this mode of operation the data collected is presented graphically as a topograph. Cantilever deflection is commonly determined by the displacement of a laser beam reflected off the free end of the cantilever (light-lever) technique.



Schematic of a light-lever force microscope

This scheme will operate in any non-viscous, optically transparent fluid. The tip may be replaced with other materials of different chemistry and geometry. Apart from providing information about surface topography and heterogeneity the scanning function can be disabled and the instrument be controlled such that only cantilever deflection versus normal displacement of the piezo is recorded. Curves of force versus relative surface separation can be obtained by normalising this deflection curve by the slope of the linear region found when the two surfaces are in ‘contact’ (compliance). Thus the interaction between two surfaces can be measured on colloidal length scales, {force $O(pN - \mu N)$, distance $O(\text{\AA} - \mu\text{m})$ }. Tip composition and geometry may be varied.

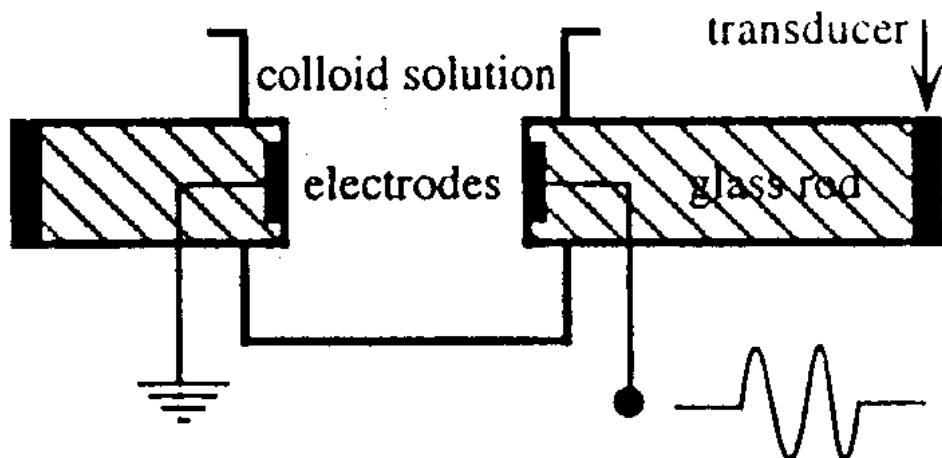


Suggested reading:

DiNardo, J., Nanoscale Characterisation of Surfaces and Interfaces, VCH, Weinheim, (1994).

Electrokinetic Sonic Amplitude Effect “Acoustosizer”

The application of an AC voltage across a colloidal dispersion causes the particles to vibrate at a velocity dependent upon their size, zeta potential and the applied frequency. At the high frequencies employed in the “AcoustoSizer”, typically around MHz, the particles emit acoustic waves in response to the alternating voltage. By pulsing the voltage signal the acoustic response, or ESA of the particles can be recorded as a function of frequency. The ESA signal is a linear function of the applied voltage and hence a Fourier transform of the recorded acoustic response is required.



Schematic of the "AcoustoSizer" cell.

From the ESA spectrum the dynamic mobility is obtained from which the size and charge of the particles can be determined. The larger the difference between the densities of the fluid electrolyte and the colloid, or the larger the volume fraction the greater the ESA signal.

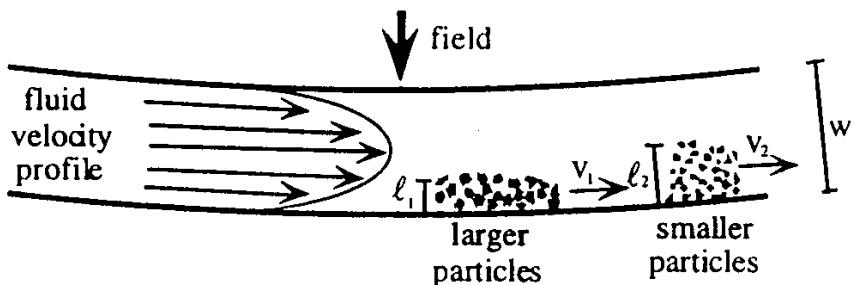
The dynamic mobility is a complex quantity equal in magnitude to the ratio of the particle velocity divided by the applied field. It is complex because there is a phase lag between driving frequency and the response of the particles to that field. As the applied field tends to zero the mobility becomes a real quantity, equal to the electrophoretic mobility defined for a static (DC) applied field. Field strengths are in the order of 100 Vm^{-1} , and at MHz frequencies typical particle displacements are in the order of 10^{-13} m . At low frequencies the inertia of the particle is not sufficient to cause a large enough phase lag to connect dynamic mobility with particle size, and only zeta potential is accessible. However, as the frequency increases the particle motion lags more and more behind the field and both size and zeta potential become available. At higher frequencies the ESA effect is too small to give a measurable signal. It is observed for many colloids systems that the equations governing the dynamic mobility simplify such that the size is determined solely by the argument and the zeta potential from the magnitude of this complex quantity. Unlike traditional electrokinetic measurements this technique is well suited to concentrated dispersions.

Suggested reading:

O'Brien, R.W., Cannon, D.W. and Rowlands, W.N., Electroacoustic determination of particle size and zeta potential, *J. Colloid Interface Sci.*, 173, 406 (1995).

Field-Flow Fractionation

Field-flow fractionation is a set of high-resolution liquid chromatography-like elution methods used for sizing and separating various samples including humic substances, clays, bacteria, viruses and polymers. There are several FFF sub-techniques (eg. sedimentation, flow, thermal and SPLITT) which are specific for certain samples. The mechanism for separation involves only physical interactions. Particles are injected onto the channel without the field turned on (i.e. centrifugal, crossflow, thermal, gravitational). The particles are evenly distributed across the channel. Then a field is applied which compresses the solute zone into a narrow layer against one wall. When the liquid flow is initiated, the solute zone is carried downstream at a rate depending on the layer thickness, ℓ which can be related to the particle size.

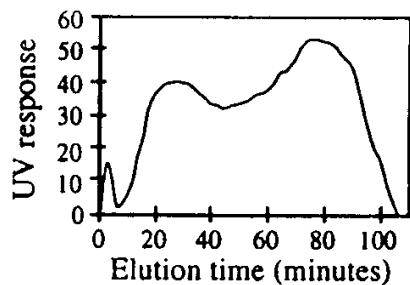


A schematic diagram of a sedimentation FFF channel.

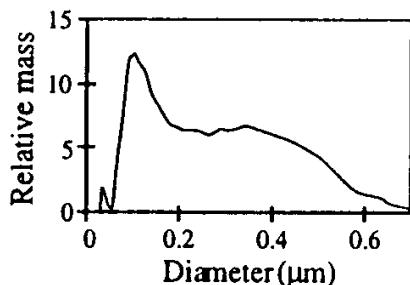
The data obtained is called a fractogram. The elution time can be converted to particle diameter using the equation;

$$d = \sqrt[3]{\frac{6kT}{\pi\omega^2 rw\Delta\rho\lambda}}$$

where, k is Boltzmann constants, T is the absolute temperature, w is the channel width, $\Delta\rho$ is the density difference between the particle and the carrier, ω is the field strength, λ is the retention parameter obtained directly from the measured retention time. Hence a particle size distribution can be calculated.



Typical Fractogram



Particle Size Distribution

Suggested reading:

Giddings, J.C., Field-flow fractionation: analysis of macromolecular, colloidal and particulate materials, Science, 260, 1456 (1993).

Summary provided by Jason van Berkel

Flotation

Flotation or froth flotation is a simple and inexpensive process used for the separation of particles (less than 500 microns) based on hydrophobicity differences of their surfaces. Only hydrophobic particles attach to gas bubbles (less than 2 mm) rising through the mineral pulp. At the top of the flotation cell, these gas bubbles break up allowing the floated particles to be recovered (Hallimond flotation tube). In froth flotation, the floated particles are trapped in the froth at the top of the flotation cell before being recovered; the froth constitutes an additional separation process.

To increase the mineral separation, a variety of reagents are added to the mineral pulp. Collectors are added to increase the surface hydrophobicity of valuable mineral particles and therefore increase their attachment to gas bubbles (only a few minerals are naturally hydrophobic, for example, talc, sulfur and molybdenite). Collectors are surfactants which consist of a head group which attaches to specific sites on the mineral surface and a hydrocarbon chain or tail which imparts hydrophobicity to the mineral surface. On the other hand, depressants such as inorganic ions or polymers are added to make the mineral surface more hydrophilic and, therefore, to depress the flotation of these minerals.

Flotation has also been used in the treatment of waste water, coal, clays, corn, proteins, dyes, fats, rubber, dyes, glass, plastics, etc.

Suggested reading

Matis, K.A. and Zouboulis, A.I, An Overview of the Process. Flotation Science and Engineering (Matis, K.A., Ed.), Marcel Dekker, New York, pp.1-43 (1995).
Fuerstenau, M.C., Miller, J.D. and Kuhn, M.C. in Chemistry of Flotation, AIME, New York (1985).

Summary provided by Daniel Fornasiero

Light Scattering

Dynamic Light Scattering (DLS), also known as Photon Correlation Spectroscopy (PCS) uses the time dependence of the intensity of the scattered light to determine the translational diffusion coefficient of small particles. If a laser beam illuminates a suspension, and the scattered light is collected over a small solid angle, then the light will exhibit intensity fluctuations on the order of microseconds to milliseconds time scale. This is because the Brownian motion of the scatterers causes the phase of the light scattered from each particle to change with time, hence the total scattered intensity also fluctuates with time.

From the intensity fluctuations it is possible to determine the electric field auto-correlation function

$$|g^{(1)}(\tau)| = \exp(-\Gamma\tau)$$

Here $\Gamma=K^2 D_T$ is the decay constant, D_T is the diffusion coefficient and K is the magnitude of the scattering vector. For spheres, the Stokes-Einstein relationship applies and the diffusion coefficient can be related to the hydrodynamic radius by

$$D_T = \frac{k_B T}{6\pi\eta r}$$

Here k_B is the Boltzmann constant, T is the temperature, and η is the viscosity of the suspending liquid. This relationship is the basis for particle sizing by DLS.

Classical light scattering involves measurement of the total scattered intensity of light as a function of angle, concentration, or both. This is commonly summarised in a Zimm plot, which is described by the equation

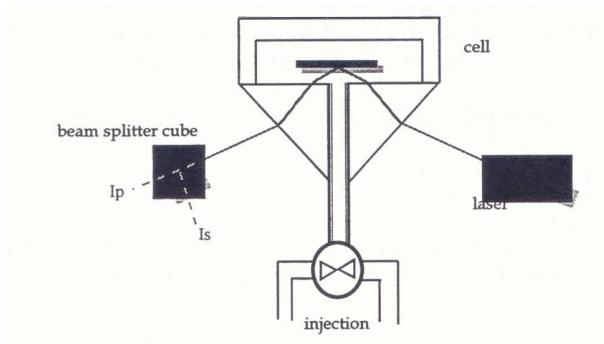
$$\frac{Hc}{R(\theta, c)} = \frac{1}{M_w} \left[1 + \frac{r_g^2 K^2}{3} \right] + 2A_2 c$$

Here c is the concentration, $R(\theta, c)$ is the excess Rayleigh ratio, K is the magnitude of the scattering vector, and H is an optical constant. Scattered light intensities are measured at several angles for each solution concentration and the pure solvent. It is then possible to determine the molecular weight, M_w , the radius of gyration, r_g , and the second virial coefficient, A_2 , for the species under investigation.

Summary provided by Roland Keir

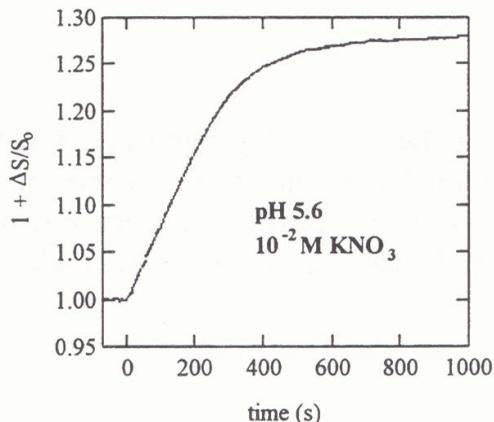
Reflectometry

This technique can be used to determine the kinetics of adsorption of polymers and nanoparticles at the solid-liquid interface. It is a simpler variant of ellipsometry and is readily combined with a stagnant point flow cell to measure adsorption under hydrodynamically well defined conditions.



Schematic of reflectometry in stagnant point flow

The surface of interest, usually a thin film (20-200nm tick) on a silicon wafer, is irradiated with a linearly polarised monochromatic and collimated beam (a He-Ne laser is often used). The refractive index difference between the film and wafer changes the polarisation of the incident beam. Passage of the reflected beam through a polarising beamsplitter cube allows detection of the intensity of the parallel and perpendicular polarised components. Adsorption of molecules or particles at the surface will change the refractive index immediately adjacent to the film and consequently, the ratio of the parallel/perpendicular components (S). It is the measured change in this ratio ($\Delta S/S_0$) with time that provides the desired kinetic information



A typical reflectometer trace obtained for 90nm silica particles adsorbing on an aminosilane treated silicon wafer

Adsorption processes, involving polymer and particles, that occur on timescales of a minute or longer are readily monitored. To convert the raw optical data into adsorbed amount (calibration) an optical model, which treats the system as a series of flat, parallel layers of uniform refractive index is used. The method is not sensitive to the confirmation of the adsorbed layers, unlike ellipsometry. However, its simplicity lends itself to kinetic studies in a range of liquids and particularly to sequential adsorption processes.

Suggested Reading:

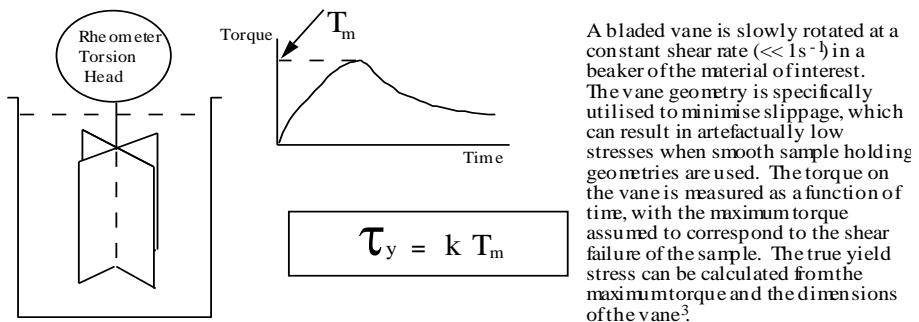
Dijt, J.C. Chohen Stuart, M.A., Hofman, J.E. and Fleer, G.J., Kinetics of polymer adsorption in stagnation point flow, *Colloids and Surfaces*, **51**, 141 (1990)

Summary provided by Rob Hayes

Concentric Cylinder Rheology

Rheology is the study of material flow under application of deformation. In practice, rheology is concerned with the flow, transport and handling of complex fluids such as dispersions, emulsions, foams, polymer melts and polymer solutions. Many industrial processes, including minerals processing, waste disposal, food and pharmaceutical formulation and polymer processing are highly rheologically sensitive. Rheological properties are commonly measured using concentric cylinder rheometers, which may operate in either constant stress or constant rate modes. Constant stress devices induce a stress in the sample by way of a torque induction motor and precisely measure its deformation, often with an optical encoder. Constant rate devices shear the sample at predefined shear rates and measure the torque, hence stress, by way of a transducer. Both types of instruments can be used in static or dynamic modes to measure flow or viscoelasticity. The commercially available constant stress devices generally have the advantage of a wider range of measurable shear rates (10^{-6} to 10^4 s^{-1}) and dynamic stresses or strains. Cone and plate is the preferred sample holding geometry, since homogeneous shear rates are experienced throughout the sample. However, suspensions are often analysed in cup and bob or plate and plate geometries for ease of handling and to avoid problems associated with particles in the gap between cone and plate.

In colloid and surface chemistry we are generally concerned with correlating rheological parameters with the colloidal micro-structure¹. The yield value^{2,3}, which is defined as the maximum stress achievable in a sample prior to flow, is commonly used to quantify the structure in a colloidal network and can be thought of as the elastic limit of a system under shear. Yield stresses are obtainable from the application of a model to a flow curve, e.g. Bingham (extrapolated) or Casson. The agreement of these values with the true yield value τ_y is questionable and may be dependent on the range of shear rates analysed. The Vane technique is therefore the favoured method for yield value determination in highly concentrated dispersions.



Schematic of the Vane technique for yield value determination

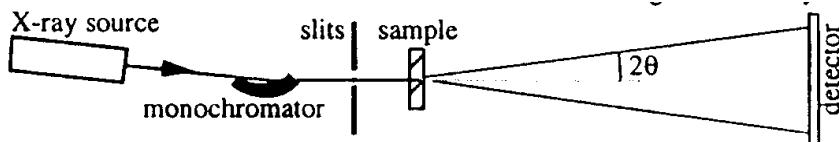
The viscoelastic characteristics of colloidal systems are also characterised by concentric cylinder rheometry, through measurement of the response to application of a sinusoidal stress or strain. G' , the storage or elastic modulus and G'' , the loss or viscous modulus, are determined from the stress-strain inter-relationship and the phase angle (ϕ) between them: $G''/G' = \tan\phi$. For non-destructive oscillatory measurements a material should be deformed within its linear viscoelastic region, typically $<0.1\%$ strain for aggregated suspensions. A material's viscoelastic response to frequency is generally characterised and this can then be related to colloidal interactions within the system¹.

1. Tadros, Th.F., Adv. Coll. Interface. Sci. **68**, 97 (1997)
2. Cheng, D. C-H, Rheol. Acta. **25**, 542 (1986)
3. Nguyen, Q. D., and Boger, D.V., J. Rheol. **27**, 321 (1985)

Summary provided by Clive Prestidge

Small Angle X-Ray Scattering

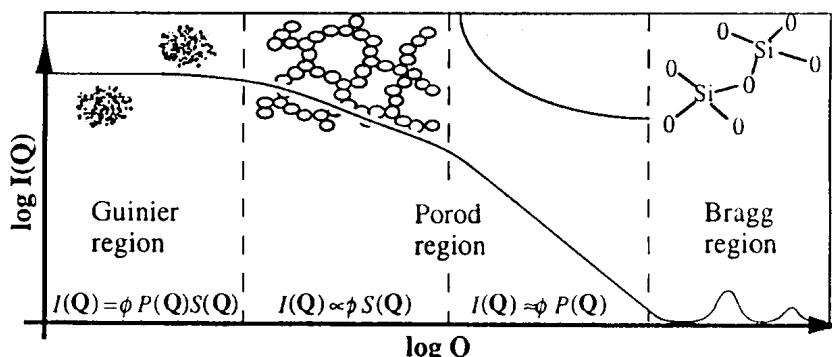
Small angle X-ray scattering allows the physical characteristics - dimensions, surface roughness and spatial arrangement - of particles ($\phi 10 - 1000\text{\AA}$) in solution to be studied. In practice a collimated beam of monochromatic X-rays is passed through the sample and the intensity of scattering measured as a function of scattering angle 2θ (usually $<2^\circ$).



The scattering of those X-rays arises from their interaction with the electronic distribution of the scattering material, the extent of scattering is dependent on the scattering length density, ρ_x , of the material given by;

$$\rho_x = \frac{n\rho N_A b_x}{MW}$$

where n is the number of electrons in the molecular unit of the scatterer, ρ is the physical density of the scatterer, N_A is the Avogadro's number, b_x is the scattering length per electron and MW is the molecular weight of the scatterer. The intensity of the scattering signal is proportional to the square of the contrast, (p_1-p_2) , where p_1 and p_2 are the scattering length densities of the particles and the surrounding medium respectively. The intensity of small angle scattering, $I(Q)$, is given by the general equation; $I(Q) = \phi P(Q)S(Q)$; ϕ is the number density of particles in solution and Q is momentum transfer, $Q = (4\pi/\lambda)\sin\theta$. The form factor $P(Q)$ which contains the $(p_1-p_2)^2$ term, reflects the distribution of scattering material in the scattering particle and the structure factor. $S(Q)$ is related to the spatial distribution of the scattering particles in the solvent.



At a large scattering angle, information on the atomic scale is obtained with Bragg diffraction. On the low- Q side of the Bragg region information regarding the surface texture of the primary scattering particles is obtained. At slightly lower Q again information regarding the physical density of scattering material within the aggregate is accessible. In the lowest Q -region depicted here, the Guinier region, the overall dimension and shape of the aggregates can be determined.

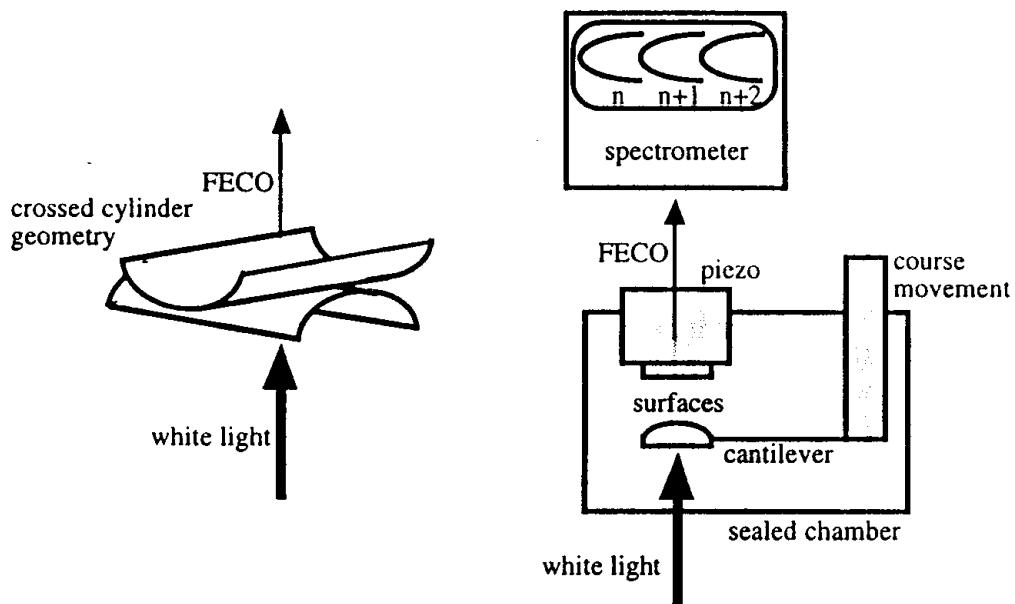
Suggested reading:

Glatter, O. and Kratky, O. (eds), Small angle X-ray scattering, Academic Press, Sydney (1982).

Summary provided by Julieanne Dougherty

Surface Force Apparatus

The Surface Force Apparatus has been the main-stay technique in the measurement of surface force for close to 30 years. Typically it is comprised of two partially silvered, thin ($O(\mu\text{m})$) mica crystals, one surface supported on a piezoelectric ceramic for fine movement control, and the other on a cantilever. When white light is passed through the two opposing silvered mica surfaces the optical cavity formed transmits light of discrete wavelengths (FECO, Fringes of Equal Chromatic Order). These wavelengths are known for a given separation of the mica surfaces and refractive index of the intervening medium. By analysing the transmitted light in a spectrometer the surface separation may be determined directly (cf. force microscopy).



Schematic of SFA with detail of partially silvered mica surfaces (left).

In the absence of any interactions between the surfaces the separation between the two surfaces reduces at the same rate as the piezo is displaced. However, when a force acts between the two surfaces, the cantilever deflects and the separation changes by a sum of the piezo displacement and the spring deflection. As the surface separation is directly measured the deflection of the spring is taken as the difference between the measured separation and the separation expected when no force is present for a given piezo displacement. Hooke's law then gives the spring restoring force, which is equal and opposite to the measured surface force. {force $O(0.1\mu\text{N}\cdot\text{mN})$, distance $O(\text{\AA}\cdot\mu\text{m})$ }.

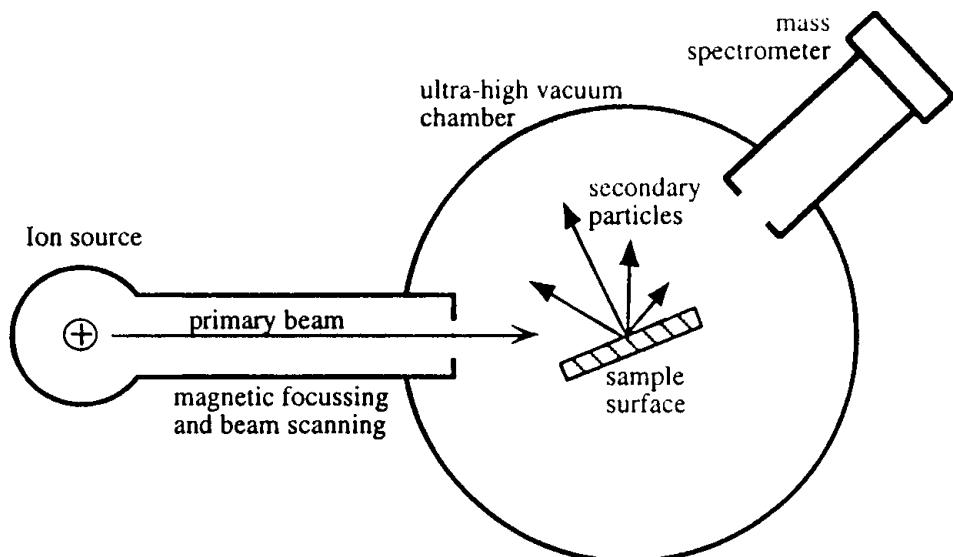
The crossed cylinder geometry approximates well as a sphere, with a radius equal to the geometric mean curvature of the two cylinders, interacting with a plane. Surfaces are restricted to those that can easily form transparent, thin smooth films, e.g. silica, sapphire, mica. Alternatively the interferometer may be formed between one reflective and one partially transmitting surface, e.g. mercury and mica.

Suggested reading:

Israelachvili, J.N., Intermolecular and Surface Forces, 2nd Edition, Academic Press, San Diego (1992).

Secondary Ion Mass Spectroscopy

SIMS is defined as the bombardment of a sample surface with a primary ion beam followed by mass spectrometry of the emitted secondary ions.



A (very) simplified schematic of a SIMS setup.

Primary beam species useful in SIMS include Cs^+ , O^{2+} , O_2 , Ar^+ , and Ga^+ at energies between 1 and 30 keV. Primary ions are implanted and mix with sample atoms to depths of 1 to 10 nm. The bombarding primary ion beam produces monatomic and polyatomic particles of sample material and resputtered primary ions, along with electrons and photons. The secondary particles carry negative, positive, and neutral charges and they have kinetic energies that range from zero to several hundred eV. The SIMS primary ion beam can be focussed to less than 1 micron in diameter. Scanning where the primary ion beam strikes the sample surface provides for microanalysis, and the measurement of the lateral distribution of elements on a microscopic scale.

During SIMS analysis, the sample surface is slowly sputtered away. Continuous analysis while sputtering produces information as a function of depth, called a depth profile. Sputter rates in typical SIMS experiments vary between 0.5 and 5 nm/s. Sputter rates depend on primary beam intensity, sample material, and crystal orientation.

When the sputtering rate is extremely slow, the entire analysis can be performed while consuming less than a tenth of an atomic monolayer. This slow sputtering mode is called static SIMS in contrast to dynamic SIMS used for depth profiles.

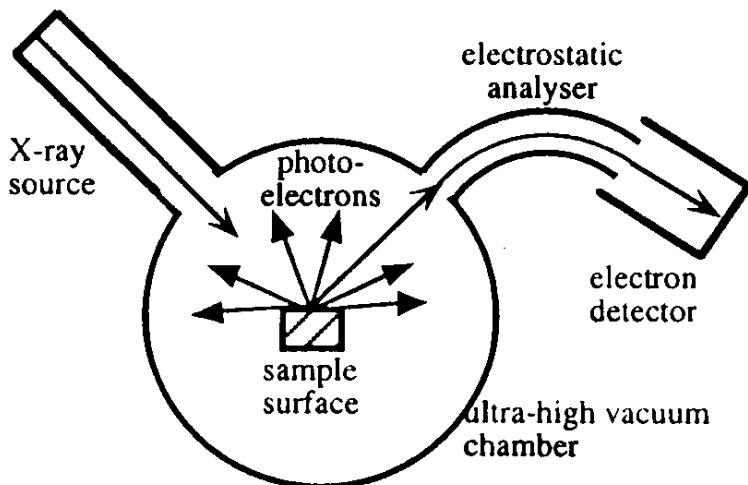
Mass spectroscopy not only provides isotopic discrimination, but both negative and positive species can also be analysed.

Suggested reading:

Benninghoven, A., Rüdenauer, F.G. and Werner, H.W., Secondary Ion Mass Spectrometry; Basic Concepts, Instrumental Aspects, Applications and Trends. Wiley, New York, (1987).

X-Ray Photoelectron Spectroscopy

XPS, also called ESCA (Electron Spectroscopy for Chemical Analysis), exploits the photoelectric effect to obtain information about the chemical composition and structure of a surface. The technique uses soft X-rays to eject electrons from core levels within the atoms. The energies of these core levels (binding energies) are characteristic of a particular element and its chemical environment.



Schematic of XPS setup

The kinetic energy of the emitted photoelectron, KE, is determined during the experiment, $h\nu$ is the energy of the incident photon, hence the binding energy, BE, for the emitted photoelectron can be calculated.

$$KE = h\nu - BE$$

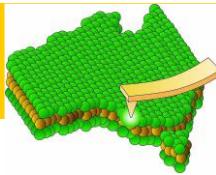
The energy of the photoelectrons emitted from an atom is also determined to some extent by the charge residing on the atom as a result of chemical bonding. Thus, a relationship between electronegativity and binding energy has been shown for many elemental systems, and a theoretical understanding of "chemical shifts" has become well-developed. From a practical point of view, it is usually possible to distinguish elemental oxidation states and sometimes other structural effects.

The kinetic energies of the photoelectrons leaving the sample are so low that, in practice, only those from the top 1-5 nm of the surface escape and arrive at the spectrometer, making it an extremely surface sensitive technique. Most elements (except hydrogen and helium) have practical detection limits of 0.02-0.2% a/a. This corresponds to 1-10% of a monolayer. Energy spectra are reported as binding energy against intensity. With the use of sensitivity factors, peak intensities can provide quantitative elemental surface compositions.

Suggested reading:

Brundle, C.R. and Baker, A.D. (Ed.), Electron Spectroscopy: Theory, Techniques, and Applications, Vols. 1 and 2, Academic Press, London (1977).

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