

PROGRAM & ABSTRACTS

23rd Australian Colloid &
Surface Science Student
Conference

Lake Hume Resort
Albury
30 Sept - 4 Oct
2002

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Preface

The Australian Colloid and Surface Chemistry Student Conference had their rather inauspicious beginnings some 35 years ago in the form of informal meetings between Tom Healy and Bob Hunter's research groups. Those early meetings, rumour has it, were lively, with full and frank discussions. Research presentations were delivered using chalk and blackboards - also the occasional refreshing beverage to help the discussions along.

As multi-media technology developed so too did the standard of the presentations, and it is now common that presentations are delivered using power point. Perhaps in another 35 years, presentations using 3D holographs and surround- sound will be considered normal.

About 20 years ago, in the Preface to the 10th ACSSSC Tom reflected on what would happen to Science in the future. He commented on the relevance of Colloid Science in a number of areas and importantly noted that without basic research, applied science would work in a vacuum. Much of what he wrote in that Preface is true today, basic science underpins innovation and it is there to support the development of new technology, such as the current "hot" areas of Nanotechnology and Biotechnology. It is vital that the education of new generations of students' remains strong in fundamental science and that there is always the basis there to provide the foundation for future developments in emerging areas. The ACSSSC plays an important role in this education.

Tradition has it that student speakers are not grouped in any particular theme, and this practise remains in place this year. However, there has been one amendment to this tradition. Any pregnant woman who may give birth during the conference will be the first speaker on the list (they may need to leave urgently)!

Welcome to the 23rd ACSSSC. May you all enjoy your time here, and remember your experiences fondly in the years to come.

Franz Grieser

Scott McLean

Kerry Hambridge

Muthupandian Ashokkumar

Peter Scales

CONFERENCE DELEGATES

ANSTO

Jamie Schulz

CSIRO

Division of Molecular Science

Patrick Hartley

Ewen Silvester

LaTrobe University

Bendigo

Michael Angove

Jacqueline Heard

Jaslin Ikhsan

Bruce Johnson

*Madeleine Ramstedt

- Visiting from
Umea University,
Sweden

Ali Shareef

RMIT

Terry O'Bree

Swinburne University of Technology

Choon Jek Ang

Gordon Bewsell

Russell Crawford

Daniel Eldridge

Kristi Hanson

Ian Harding

Ranjith Jayasekara

Lidia Konkol

Francois Malherbe

Marylou Molphy

Peter Sanciolo

Philip Tu

University of Newcastle

Rob Atkin

Amanda Durand

Simon Ferguson

George Franks

Jason Kilpatrick

University of Newcastle

Aaron Olsen

Emelyn Smith

Erica Wanless

Peter Yates

University of South Australia

Sameer Al-Bataineh

David Beattie

Linh Chiem

Jarred Clasohm

Patricia Curtis

Jinming Duan

Yan Er

Rick Fabretto

Saeed Farrokhpay

Daniel Fornasiero

Val Gilchrist

Graeme Gillies

Hans Griesser

*Stephan Herminghaus

- Visiting from
University of Ulm,
Germany

Roger Horn

Gillian Kaggwa

Alan Kanta

Heather Moore

Gayle Morris

Patience Mpofo

Craig Priest

Rada Pushkarov

Anthony Quinn

John Ralston

Nicola Richards

Rossen Sedev

Spomenka Simovic

David Snoswell

Nate Stevens

Anna Tarasova

Su Nee Tan

Lucy Wang

Jingwu Yang

University of Melbourne

Ainul Azzah Abd. Aziz
 Yang Cao
 Derek Chan
 Tristan Croll
 Ray Dagastine
 Dave Dunstan
 Joel van Embden
 Michelle Gee
 Sarah Glasson
 Di Goodall
 Will Goodall
 Chris Green
 Franz Grieser
 Peter Harbour
 Tom Healy
 Elisabeth Hill
 Joeska Husny
 Judy Lee
 Hadi Lioe
 Jason Looker
 Alexander Lubansky
 Scott McLean
 Paul Mulvaney
 Muthupandian Ashokkumar
 Kathy Northcott
 Truong Son Nguyen
 Andrea O'Connor
 Ying Pan
 Isabel Pastoriza-Santos
 Jorge Perez-Juste
 Julian Reichl
 John Sader
 Peter Scales
 Colin Scholes
 Fiona Scholes
 Ritu Singla
 Geoff Stevens
 Anthony Strickland
 Chor Sing Tan
 Debra Turner
 Tim Wade

University of Otago

New Zealand
 Jocelyn Collie
 Steven Dickinson
 Grace Liu

University of Sydney

Annabelle Blom
 Paul FitzGerald
 Bob Hunter
 Kathryn Topp

Monash University

Ian McKinnon

Yates Technical Services P/L

David Yates

Past Conferences

Sydney	1967
Melbourne	1969
Sydney	1972
Blackwood	1973
Pretty Beach	1974
ANU	1976
Blackwood	1977
Yarrowood	1978
Kioloa	1980
Mt Eliza	1982
Yarrowood	1982
Roseworthy	1985
Kioloa	1987
Albury	1988
Camden	1990
Roseworthy	1993
Deakin Uni.	1993
Fairy Meadow	1995
Murramarang	1996
Hahndorf	1998
Morpeth	1999
Bendigo	2001

	Monday	Tuesday	Wednesday	Thursday	Friday
AM					
7:00		Breakfast	Breakfast	Breakfast	Breakfast
9:00	On site registration				Departure
9:15			Session #6	Session #8	
9:30		Introduction to Plenary speaker by Tom Healy Plenary speaker - Ray Dagaatine			
10:30		Morning tea	Morning Tea	Morning tea	
11:00		Session #3	Session #7	Session #9	
12:30	Lunch				
12:45		Lunch	Lunch	Lunch	
1:00					
1:30					
1:50	Welcome F.Grieser				
2:00	Session #1	Session #4	Games afternoon	Poster Session	
3:15	Afternoon tea	Afternoon tea			
4:00	Session #2	Session #5		Introduction to Plenary Speaker by Bob Hunter Plenary Speaker - Fiona Scholes	
5:30	Free time	Free time		Free time	
6:00	Drinks at the fire	Drinks at the fire	Drinks at the fire	Drinks at the fire	
7:00	Dinner	Dinner/Trivia night	Dinner/Entertainment	Conference Dinner Guest Speaker Awards Entertainment	

Program Day 1, Monday 30th

9:00	<i>Registrations</i>
12:30	<i>Lunch</i>
1:50	<i>Welcome by Franz Grieser</i>
	Session #1 Chair - M. Ashokkumar
2:00	Ainul Azzah Abdul Aziz
2:25	Ying Pan
2:50	Anthony Quinn
3:15	<i>Afternoon tea</i>
	Session #2 Chair - Patrick Hartley
3:45	Aaron Olson
4:10	Lidia Konkol
4:35	Steven Dickinson
5:00	Kristi Hanson
5:30	<i>Free time</i>
6:00	<i>Happy Hour</i>
7:00	<i>Dinner</i>

Program Day 2, Tuesday 1st

7:00	<i>Breakfast</i>
9:30	<i>Introduction to Plenary speaker by Tom Healy</i>
	Plenary Speaker - Ray Dagastine
10:30	<i>Morning tea</i>
	Session #3 Chair - Erica Wanless
11:00	Su Nee Tan
11:25	Amanda Durand
11:50	Choon Ang
12:15	David Snowswell
12:45	<i>Lunch</i>
	Session #4 Chair - Russell Crawford
2:00	Craig Priest
2:25	Jocelyn Collie
2:50	Jaslin Ikhsan
3:15	<i>Afternoon tea</i>
	Session #5 Chair - Ian Harding
4:00	Kathy Northcott
4:25	Paul FitzGerald
4:50	Madeleine Ramstedt
5:15	<i>Free time</i>
6:00	<i>Happy Hour</i>
7:00	<i>Dinner/Trivia night</i>

Program Day 3, Wednesday 2nd

7:00	<i>Breakfast</i>
	Session #6 Chair - George Franks
9:15	Peter Yates
9:40	Patience Mpofu
10:05	Scott McLean
10:30	<i>Morning tea</i>
	Session #7 Chair - Gayle Morris
11:00	Rick Fabretto
11:25	Grace Liu
11:50	Rob Atkin
12:15	Lucy Wang
12:45	<i>Lunch</i>
2:00	<i>Games Afternoon</i>
	<i>Coordinator Julian Reichl</i>
5:15	<i>Free time</i>
6:00	<i>Happy Hour</i>
7:00	<i>Dinner/Entertainment</i>

Program Day 4, Thursday 3rd

7:00	<i>Breakfast</i>
	Session #8 Chair - Michelle Gee
9:15	Spomenka Simovic
9:40	Jason Kilpatrick
10:05	Yang Cao
10:30	<i>Morning tea</i>
	Session #9 Chair - Jamie Schulz
11:00	Jingwu Yang
11:25	Saeed Farrokhpay
11:50	Graeme Gillies
12:15	Nicola Richards
12:45	<i>Lunch</i>
2:00	Poster Session
3:15	<i>Afternoon tea</i>
4:00	Introduction to Plenary speaker by Bob Hunter
	Plenary Speaker - Fiona Scholes
5:15	<i>Free time</i>
6:00	<i>Happy Hour</i>
7:00	<i>Conference Dinner</i> <i>Guest Speaker</i> <i>Stephan Herminghaus</i> <i>Awards</i> <i>Entertainment</i>

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CONFERENCE AWARDS

Healy-Hunter Award

The most outstanding oral presentation will be awarded the Healy-Hunter Award. This award is a prize of a \$1,000 travel grant and is voted by the staff.

Group Scientific Best Poster Award

The most outstanding poster presentation in the Group Scientific Poster Session (Thursday afternoon) will be awarded the Group Scientific Best Poster Award. This award is a prize of \$300 cash (staff voted).

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!

Rheological behaviour of concentrated colloidal suspensions

Ainul Azzah Abdul Aziz and Ass. Prof. Peter J. Scales

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Due to high surface area to density ratio, colloidal suspensions have enormous applications either at domestic or industrial levels. Products from foodstuffs to personal cares, from ceramic productions to handling of water/waste water treatment sludges, all dealt with colloidal suspensions. Understanding the characteristics of the suspension either at microscopic or macroscopic levels is then important in controlling the bulk properties of the material such as flow behaviour.

In general, colloidal suspension is defined as micro-solid particles suspended in liquid medium. The flow behaviour of the suspension is largely controlled by the sum of interaction forces between particle and fluid, and particle and particle. Experimentally, the flow behaviour is easily measured using a conventional rheometer or specially designed apparatus. The rheological properties determined including shear viscosity, shear and compressive yield stresses, breaking strain, etcetera. However, dealing with concentrated suspensions, these measurements are more prone to rheological problems such as (slip). This effect will be discussed briefly.

After ensuring good data were collected, the rheological properties are compared for a model system of alumina AKP-30. Good correlations were found by using different type of measurements to measure similar properties. The relationship between all the rheological properties will be discussed.

Extraction Kinetics of copper with LIX84 in micelle

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Solvent extraction is an important separation process used in the hydrometallurgical industry. In Australia it is used to purify a range of metals including copper, nickel and cobalt. Micellar and microemulsion systems are widely used to study the metal extraction process on a fundamental level. The hydrophobic core of micelles are able to act as solubilization sites for hydrophobic extractants and in this respect the micelle pseudophase can act in a similar way to the organic phase that exists in a classical biphasic extraction system. From a research point of view, micellar systems are very attractive because of their microheterogeneous structure and small size which guarantees transparency to light. This latter property has a number of advantages especially for studying the kinetics and mechanistic aspects of extraction using optical methods. Micellar systems are also of interest from an environmental point of view, as it may be possible to achieve metal ion extraction in media that are up to 99% aqueous.

Stopped flow methods are well suited for studying the kinetics of reactions in homogeneous solution. The project that is described in this talk is the reaction that occurs between the oil soluble extractant LIX 84 and the metal ion, Cu^{2+} , in a non-ionic micellar solution. From the kinetic data obtained and using mathematical modelling to simulate the kinetics observed it was possible to extract the kinetic parameters for the mechanism that was proposed to account for the extraction process. A two step reaction mechanism between Copper(II) and LIX 84 was shown to explain the kinetic processes that were experimentally observed in the micellar system.

Electrowetting: Mechanisms for saturation

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Wetting interactions are common in a myriad of day to day activities. Appropriate surface modification can temporarily, or even permanently modify a surface's wettability leading to interesting applications. There are numerous methods available to achieve this change in surface response, for example, chemical techniques. However external fields offer exciting alternatives including, thermal stimulation, photoinduction, magnetic or electrical fields to alter wettability. The charge at a metal/electrolyte interface may be altered via an externally applied electric potential. An insulating material may be introduced between an electrode and an electrolyte, enabling the initial contact angle to be altered, depending on the dielectric material used, and a wider potential range to be utilised. Electrowetting is the term used to describe this forced wetting process via an external electric potential, and is both an avenue of strong research activity as well as of significant industrial interest in areas including the micromanipulation of aqueous electrolyte solutions and the development of optical or x-ray filters. Electrowetting theory is only applicable over a limited potential range however, for the forced wetting saturates prior to complete wetting. We propose a theoretical model and experiments, which, together, offer an insight into the phenomenon.

Effect of Shear Rate on Flocculation Efficiency of Polymeric Flocculants

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Polymeric flocculants are widely used in solid-liquid separation processes. Shear mixing plays a vital role in these processes; satisfactory flocculation requires efficient particle and polymer mixing. This work investigates the influence of shear mixing on optimum flocculant dosage. Colloidal alumina was flocculated using poly(acrylic acids) of greatly differing molecular weights under shear mixing applied by a 6-blade Rushton turbine. An investigation was conducted on the effect of shear rate on the flocculation efficiency at a given dosage for the different polymers. The flocculation efficiency was determined from turbidity measurements as well as visual observations.

The effect of shear rate on flocculation efficiency was found to be dependant upon the molecular weight of the polymer added. The correlation between molecular weight and the change in flocculation efficiency with shear rate has been linked to the difference in flocculation mechanisms of the polymers. A theory based on the effect of shear on the conformation of adsorbed polymer chains and the subsequent effect on flocculation mechanism has been proposed to explain these observations. Strength measurements of individual flocs, obtained using a four-roll mill apparatus, are provided in support of this theory.

Contaminants in Recycled PET Plastics

L. M. Konkol, R. F. Cross, I. Harding and E. Kosior.

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Contamination of soft-drink grade PET could result from a variety of sources, including consumer misuse, the original contents of the PET bottle and the recycling process. Chemicals may adsorb onto the surface and/or penetrate into the walls of PET bottles and consequently pose a potential, chronic health risk once the containers are recycled and used for food purposes.

The primary purpose of this study was to determine how curbside collected, washed and dried (henceforth known as postconsumer) PET differs from virgin PET and PET, which has undergone a full recycling process with respect to contamination. Contaminants were extracted from freeze-ground washed/dried flake (particle size = <300 μm) by soxhlet extraction using dichloromethane, a PET-swelling solvent. The extract, which was analysed by GC/MS, contained thirty semivolatile substances foreign to virgin PET. Limonene, cineole, naphthalene and its methyl/ethyl derivatives, lauric acid, 1,2,3,5-tetramethyl benzene, 5-methyl-2-(1-methyl ethyl) cyclohexanone, and 4-propyl anisole are examples of contaminants identified in postconsumer PET.

Selected ion monitoring (SIM) GC/MS was used to quantitatively determine the contaminant level of semi-volatiles in post-consumer PET and for most compounds, the value was below the US FDA food contact required threshold of 215 ppb. Limonene, benzophenone, dodecanoic acid and Benzoic Acid, 2-Hydroxy Methyl Ester are examples of compounds exceeding this limit. Recycling ensured that the levels of these contaminants were within the regulated amount.

Experimental conditions were optimised with respect to extraction time for post-consumer PET flake ground to 0-300 μm . The extraction kinetics was shown to be similar for all compounds, with an optimum extraction level of 24h. A common extraction trend suggested that there was one extraction mechanism prevalent for all compounds. Further analysis by sonication indicated that extraction was completed after just 3h, as the particles were now dispersed and in better contact with the solvent.

The extraction kinetics for two larger particle size ranges was also investigated. (300 μm –425 μm ; 425 μm –700 μm) Due to the less dense particle packing, extraction was completed after just 3h. Compared to the smallest particle size, the level of contamination in the larger particle sized flake was significantly lower. Total dissolution experiments of each particle size confirmed that these differences resulted from an inhomogeneous grinding procedure and not from the diffusion-controlled mechanism. It is thought that the grinding procedure selectively grinds the softer part of flake, containing more contaminant, to a smaller particle size.

Synthetic biomineralisation: the effects of short chain alcohols on calcium carbonate growth

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Biomineralisation is the formation of inorganic materials within a biological organism. The organism manipulates the growth of the inorganic materials, allowing production of a diverse range of shapes and structures. These materials have controllable and well-defined physico-chemical properties. Control being exerted through a variety of means e.g. specific molecular interactions, environmental matrix control, physical characteristics of the growing medium, and/or initial concentrations of reactants. The level of control achieved by organisms is currently beyond our synthetic capabilities.

Our aim is to understand at a fundamental level the interaction between the growing inorganic crystal and the reaction medium. It is envisioned that this will enable us to generate materials whose physico-chemical properties mimic those of biominerals.

To this end a simple alcohol series has been used to probe the influence of the alcohol moiety on calcium carbonate growth in a controlled environment. Nucleation and growth is in general inhibited in the presence of short chain alcohols. In solutions with a high alcohol content, a morphological form known as a hopper crystal begins to dominate. This morphology is characterised by deep face-centred pits in the rhombohedral calcite. Two mechanisms have been suggested to explain this growth phenomenon. The first mechanism involves growth spiralling outwards from a screw dislocation (growth under thermodynamic control) whilst the second mechanism requires limiting diffusion within the growth medium (kinetic control). We believe that hopper formation is due to the later, with growth being controlled by the viscosity of the solution.

The behaviour of laccase in the presence of inorganic and organic adsorbents

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Laccase, a phenol oxidase produced by many wood-rotting fungi, has received considerable research attention due to its potential use in a variety of biotechnological applications. Laccase is excreted into the solid compost growth matrix of the common white button mushroom, *Agaricus bisporus*, and has been extracted and purified from commercial spent mushroom substrate (SMS) in large quantities. It has been previously reported that a substantial amount of laccase remains bound to solid compost particles following the extraction process. Further, the presence of compost particles in reaction mixtures has been found to both enhance and stabilize enzyme activity. In order to determine how sorbent surface properties influence enzyme structure and activity, purified *A. bisporus* laccase was immobilized through adsorption on a variety of inorganic and organic colloidal sorbents, namely titania, hydrophilic silica, montmorillonite, polyethylene terephthalate (PET), cellulose, chitin, and a complex compost mixture. Enzyme-sorbent interactions were found to be pH dependent on most substrates, with maximum adsorption occurring below and just above the enzyme's pI. However, adsorption onto montmorillonite was strong over a wide pH range. Enzyme specific activity and stability were altered in the adsorbed state, suggesting that the protein undergoes conformational change during sorption.

Influence of low molecular weight nonionic surfactants on foam behaviour

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Foams and their properties are very important in various industrial processes such as plastic and ink recycling, mineral processing and enhanced oil recovery. There are many investigations of the influence of surfactants on foam behaviour. However, little is known about the role of low molecular weight nonionic surfactants. The focus of this work is to study the influence of low molecular weight polypropylene glycols on foamability.

The foamability of aqueous solutions of polypropylene glycols (molecular weight of 192, 400 and 725 g/mol) was characterized. For each surfactant, the foamability increases to a maximum and then decreases, as surfactant concentration increases. Static surface tension of surfactant dictates the rate of increase in foamability at low surfactant concentration, while the dynamic surface tension dictates the maximum foamability. The decrease in foamability at high concentration is attributed to the presence of surfactant droplets (phase separation is detected by dynamic light scattering). There is an apparent relation between the surfactant conformation at the interface and foamability. Shorter surfactants (PPG192 and PPG400) adopt a flat conformation at the water/air interface, while longer chain surfactants (PPG725) tend to coil. Our hypothesis is that surface diffusion of coiled PPGs, driven by the surface tension gradient presents on the bubble surface, is slower. As a result, the interfaces are partially mobile which promotes liquid drainage and hence reduce foamability. Currently, we are investigating the thin film drainage rate by measuring the coalescence rate between two captured bubbles. This will provide a test for the proposed mechanism.

Colloidal Aspects of Milk and their Affect on Stability

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Milk and milk alternatives such as soymilk, oat milk and rice milk are colloidal emulsions and suspensions. The emulsions are the small fat or oil droplets, stabilised by surface-active agents such as proteins and lipids. The suspensions are the insoluble solid particles, such as undissolved proteins, starch, fibre and other cellular material that are suspended in the aqueous solution. The presence and size of solid particles can have a significant effect on the emulsion stability, perceived texture and the efficiency of bacterial reduction and therefore the shelf life of the milks. There is limited literature relating the particle size to the stability of milk or milk alternatives. The aim of this study was to compare the particle size, stability, texture and viscosity of cow's milk, soymilk, oat milk and rice milk, and to evaluate the relationship between these factors. Particle sizing was conducted with a Malvern Mastersizer, emulsion stability was determined with a Turbiscan (Quickscan) light scattering analyser, and the texture and viscosity of the milks were evaluated by a sensory panel. The particle size and emulsion stability of the milks were highly correlated. The $d_{3,2}$ values ranged from 0.42 μ m to 2.52 μ m and varied for the different milks. The level of stability and type of destabilisation (creaming or sedimentation) of the milks also varied, but could be predicted from the particle size distributions and terminal velocity calculations. Despite variations in particle size distributions, the sensory panel was unable to detect a difference in texture between the fresh milks. The panel detected a difference between the viscosities of the milks, and these results were correlated with the size of the particles in the milks.

Treatment of Printing Wastewater using Adsorbing Colloid Flotation

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Printing wastewater is characterised by high levels of chemical oxygen demand (COD), biochemical oxygen demand (BOD) and colour. It contains a combination of heavy metals, pigments, suspended solids as well as organic solvents and is difficult to treat by conventional sedimentation. The aim of this project is to investigate the feasibility of the use of a novel development in adsorbing colloid flotation (ACF) to treat printing wastewater.

Conventional adsorbing colloid flotation involves adsorption and/or coprecipitation of contaminants onto metal hydrous oxide colloid, followed by removal of these solids from solution by flotation. Surfactant is used to render the solids sufficiently hydrophobic to be collected at liquid/bubble interface, and to establish a foam. The nature of the interaction between the removed contaminants and the bubbles in the foam is such that, soon after generation of the foam, the solids are released to the drainage liquid. It is therefore important to keep the volume of this liquid in the foam to a minimum. This, in turn, limits the rate at which the conventional ACF treatment process can be operated.

An alternative mode of running the ACF process involves the choice of conditions such that a solids-stabilized foam is generated. Under these conditions, the drainage from the foam is as clean or cleaner than the liquid in the flotation cell. This allows operation of the process at a much faster rate since the foam liquid content does not need to be minimized. It also allows much higher levels of dewatering of the removed contaminant solids to take place.

Batch mode flotation studies were conducted on industrial printing wastewater samples that were dosed with surfactant (CTAB) and a single or mixed hydrous metal oxide adsorbing colloid. The adsorbing colloid metals investigated were Fe(III), Fe(II) and Al(III). The influence of pH, ratio of adsorbing colloid metals, concentration of adsorbing colloid, surfactant concentration and wastewater solids content were investigated. Continuous mode flotation studies were also conducted. The influence of surfactant concentration, wastewater throughput and airflow rate were investigated.

The Influence of Dissolved Gas and Surface Hydrophobicity on Particle Coagulation

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Mawson Lakes Campus, 5095 South Australia, Australia
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The colloidal stability of synthetic silica spheres with clean, methylated and dehydroxylated surfaces was studied under different concentrations of dissolved gas and KCL electrolyte at a fixed pH of 4.2. A classic stability ratio/electrolyte concentration analysis shows that hydrophobic methylated particles undergo faster rates of aggregation with increasing concentrations of dissolved carbon dioxide. Similar data for hydrophilic particles and dehydroxylated particles show no change as a function of dissolved carbon dioxide concentration. Zeta potential data behave similarly, showing a strong influence of dissolved gas only for methylated particles. The results are interpreted in terms of DLVO theory, with the surface to surface interaction dominated by the presence of very small bubbles.

Is Cassie Correct?

Craig Priest, Rossen Sedev, John Ralston

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The Cassie Equation describes the contact angle of a liquid on a chemically heterogeneous surface according to the surface coverage of each component. Although the equation is theoretically sound, for some systems Cassie's equation fails to predict the experimentally determined contact angle. In many cases, this difference may be due to sample characteristics that are inappropriate for the experimental system. In published literature, the effects of line tension and domain (or drop) sizes are frequently cited to explain deviations from the Cassie Equation. These effects are often only discussed with limited data and seldom prove to be the whole answer to the origin of the observed contact angle deviations. One important parameter that is frequently overlooked is the state of the contact angle. Most publications consider the static advancing (in preference to static receding) contact angle, however calculations using the Cassie Equation require the static equilibrium angle. In this present work we have used a vibration technique to examine the static wetting behaviour of chemically patterned surfaces using advanced, receded and "vibrated" angles. The vibrated angle is consistent regardless of the initial state of the droplet, indicating that the angle may represent a close-approximation of the equilibrium angle. Our heterogeneous surfaces consist of hydrophilic domains in a hydrophobic matrix. These domains are 10 and 20µm in size and are prepared using UV-photolithography of alkanethiol self-assembled monolayers on gold. Both circular and square domains of precise area fractions have been investigated for comparison with the Cassie Equation. Data from these samples indicate that a deviation from the Cassie Equation is present even when line tension effects are negligible and the state of the contact angle is considered. Our results show that the Cassie Equation is not always a good predictive model, depending on the type of chemical heterogeneity being considered.

Direct visualisation of biological processes using atomic force microscopy

J. Collie and K.M. McGrath

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The ability of the atomic force microscope (AFM) to image non-conducting specimens in an aqueous environment is of enormous significance to the life sciences. It opens the door to visualisation of biomolecules in their native state and allows a glance into nanometer scale biological events. We are interested in observing the preovulatory surge of luteinizing hormone (LH) from anterior pituitary cells when stimulated by peptide hormones oxytocin and gonadotrophin releasing hormone (GnRH). By observing this process in real time using the AFM there is the potential for the development of new infertility treatments and the replacement of steroid-based birth control pills.

At present the focus of the project is on the experimental set-up for imaging living cells. There are three key aspects to be considered. The first is immobilization of the cells to ensure they are not displaced by the tip during imaging. Second is the choice of physiological media, required to maintain cell functionality and finally the load on the tip needs to be considered to minimise the amount of damage incurred by the cells while achieving maximum resolution.

With respect to immobilization strategies, two different substrates are being considered; glass coverslips coated with poly-D-lysine and silicon nitride platforms with cavities just large enough for the cells to be held within. The advantage of using the platforms is that the cavities are numbered; so individual cells can be identified. To maintain cell functionality, a simple physiological buffer known as Krebs buffer is being used. To minimize the load on the tip the "tapping mode" of operation is being explored. This involves oscillating the tip at its resonance frequency while it raster scans across the sample. The tip only comes into contact with the sample during the downward part of the oscillation cycle eliminating lateral forces and allowing the imaging of soft samples, such as cells, which are difficult to image when the tip is in hard contact with the surface.

Results collected so far are promising in terms of setting up a system for the direct visualization of cells in a physiological environment and exploring their surface activity.

Effects of Pesticides on the Adsorption of Zn(II) to K-Wyoming Montmorillonite

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The adsorption of a trace element, Zn(II), to K-Montmorillonite from Wyoming in the presence of pesticides (3-amino-1,2,4-triazole (AT) and 2-aminopyridine (AP)) was carried out at 25°C and background electrolyte of 10 mM KNO₃. This investigation aims to understand the reaction mechanisms, which are responsible for the adsorption in these systems.

Zn(II) adsorption to the clay mineral increased as the pH was raised. About 50% of Zn(II) adsorbed to the surface at pH 3, and 65% at pH 7. The adsorption then increased rapidly, reaching 100% by pH 8.

The presence of AT and AP significantly inhibited the adsorption of Zn over the pH range 3 to 7, but did not affect the adsorption at higher pH values. AT, which adsorbed much less to the clay mineral, had less effect on the adsorption of Zn(II) than AP. Depression of Zn(II) adsorption to be appears due to competition between Zn(II) and the pesticides for adsorption sites, and will be discussed later.

From the laboratory to Antarctica: Flocculation and dewaterability analysis for low temperature water treatment

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A clean up of an abandoned waste disposal site in the Thala Valley near Casey station, Antarctica, will involve the extraction of wastes and contaminated sediments for treatment and disposal in Australia. The presence of significant quantities of melt-water during clean-up activities may result in entrainment and dispersal of heavy metals and some hydrocarbon pollution from the site into the surrounding environment. A portable multi-stage water treatment system incorporating coagulation/flocculation, settling, filtration and ion exchange will be in operation during the clean up to prevent the escape of contaminants from the Thala Valley site.

Pilot testing of water treatment equipment in Antarctica is difficult due to its climate and isolation. Alternative methods of testing and prediction of system performance are needed. In this investigation a combination of laboratory scale baffle reactor, batch settling and pressure filtration experiments will be used to model and predict the performance of the water treatment system under various conditions. The study will look at the effect of temperature on the effectiveness of coagulation and floc formation as well as dewaterability of flocculated suspensions. The impact of seawater/salinity on flocculation and dewaterability will be investigated, as the landfill site is located in close proximity to the marine environment.

The laboratory equipment and associated mathematical modelling required for flocculation analysis and dewaterability characterisation have been developed by researchers from the Particulate Fluids Processing Centre. The development of the water treatment system is a collaborative project with the Australian Antarctic Division. It is envisaged that this project will help set the standard for contaminated site remediation in Antarctica. Water treatment modelling techniques used for the Antarctic project may also be applied to other cold regions as well as contributing to the knowledge base of water treatment applications such as potable water, sewage and minerals processing.

Self-Assembly of Nonionic Gemini Surfactants

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In recent years both academic and industrial interest has been expressed for many new forms of surfactants. Of particular interest are surfactants that have low critical micelle concentrations (CMCs) and enhanced surface activity. Usually both of these aims are achieved by a class of surfactants called gemini surfactants, which consist of two conventional surfactants joined by a spacer at the head groups. Several non-ionic gemini surfactants have been synthesised with poly(ethylene oxide) head groups of different sizes. Their self-assembly and phase behaviour has been studied as a function of the head group size.

The gemini surfactant CMCs were measured using a fluorescence probe technique. The CMCs were found to be much lower than conventional surfactants. The mixing behaviour of the gemini surfactants with conventional surfactants was also studied. They were found to mix ideally with both conventional ionic and conventional nonionic surfactants.

The shape and size of the gemini surfactant micelles were measured in solution using Small Angle Neutron Scattering (SANS). The micelles are generally spherical, or near spherical, at room temperature. However, the surfactants with the smallest head groups (ie shortest poly(ethylene oxide) groups) were found to undergo a sphere to rod transition as the temperature was increased. This observation is supported by a large rise in the solution viscosity, which is caused by the formation of entangled rod-like micelles.

Surface Protonation and Proton Promoted Dissolution of Manganite (g-MnOOH)

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Manganite (g-MnOOH) is a metastable manganese (III) hydroxide assumed to be one of the most common natural manganese hydroxides in lakes and rivers and is, thus, expected to play a role in the geochemical cycling of metals, organic and inorganic ligands.

Crystalline manganite has been synthesised and characterised using XRD (X-ray Diffraction) and HRTEM (High-Resolution Transmission Electron Microscopy) techniques. The surface composition was studied with XPS (X-ray Photoelectron Spectroscopy) between pH 0.4 and 11. Special pre-cooling procedures were developed to keep the manganite/water interface intact before and during XPS measurements. The ratio between O and OH groups differs between pH 6 and 11 in agreement with the acid/base characteristics of the surface. The surface charge arising at high and low pH is compensated by the counter ions Na⁺ and Cl⁻, respectively. The acid/base characteristics will be discussed together with surface charge of the manganite.

Below pH 6, manganite starts to dissolve and, consequently, drastic changes can be seen in O 1s XPS-spectra. In order to qualitatively determine the dissolution products, XRD measurements were performed on the remaining solid, below pH 1. The results indicate formation of different Mn(IV) oxides, as can be expected from a disproportionation reaction of Mn(III).

Effect of Mixing on Heterocoagulation

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Heterocoagulation plays an important role in many industrial and natural processes, such as mineral processing and pulp and papermaking. Heterocoagulation commonly takes place under orthokinetic conditions, making the mixing time and intensity important parameters for obtaining optimum flocculation. A detailed study on the effect of these properties will lead to a greater understanding of the optimum conditions required for heterocoagulation. Flocculation experiments have been carried out using alumina particles as a model system. Silica particles of various sizes were chosen as the flocculant. A standard mixing vessel equipped with Rushton impeller was used. The mixing effects were quantified by measuring the optimum flocculant dosage required to achieve supernatant clarity and by monitoring in-situ the size (chord length) of the flocs being formed using Focused Beam Reflectance Measurement technique. One of the important findings is that the optimum silica dosage halved on increasing the mixing time from 5s to 30s. Possible mechanisms leading to this observation will be discussed.

The influence of pH and temperature on adsorption of PEO, hence flocculation and dewatering behaviour of kaolinite dispersions

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Dewatering of colloiddally stable mineral waste tailings is an important problem, which presents technological and environmental challenge to mineral processing industry worldwide. Flocculated, gravity assisted thickening processes are currently used by industry for dewatering but these are far from being efficient, requiring further development and optimisation for improvement. A high molecular weight, anionically charged polyacrylamide polymer which is normally used for flocculation of kaolinite dispersions, produces fragile flocs, which are not amenable to compaction. The use of a "flexible" polymer such as non-ionic polyethylene oxide (PEO) as an alternative flocculant has been of considerable interest over the past few years. In this paper, recent studies carried out to modify the interfacial chemistry, manipulate particle interactions and improve the dewatering behaviour of flocculated kaolinite clay dispersions are investigated. It is shown that changes in pH and tempera!

ture of kaolinite dispersions strongly influences the adsorption behaviour of a (PEO), impacting upon surface chemistry, flocculation performance, shear yield stress and dewatering behaviour. Flocculation and dewatering of kaolinite dispersions with PEO was more enhanced at acidic pH (3.5 -7) than at alkaline conditions (9.5). The primary mechanism responsible for this observation is linked to increased Bronsted acid sites at the kaolinite edge faces at lower pH which facilitates the adsorption of PEO, a Lewis base. The influence of solution temperature (20-60°C) on non-ionic polyethylene oxide (PEO) adsorption and flocculation was also investigated. An increase in the magnitude of zeta potential of kaolinite particles, at a fixed PEO concentration with increasing temperature reflecting a decrease in the adsorbed polymer layer thicknesses was observed. The adsorbed amount of PEO however, was found to increase with increasing temperature suggesting that the significant reducti!

on in solvency accompanied by polymer conformational changes occurred at higher than lower temperature. Flocculation and settling tests showed that increasing temperature from 20 to 40°C enhanced the flocculation performance and improved the concomitant settling and consolidation behaviour of kaolinite dispersions. A further increase in temperature beyond 40°C was detrimental to the flocculation and dewatering processes. The yield stress of the pulp decreased with increasing temperature leading to decreased bridging forces between the particles. The results demonstrate that pH, temperature and conformational change play an important role in flocculation and dewatering of kaolinite dispersions.

pH Dependent Polymeric Micelle Adsorption

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In recent years there has been increasing interest shown in the possible use of polymeric micelles in drug delivery systems. As these polymeric micelles are often diblock copolymers it is important to understand their behaviour before and after micellisation, particularly in regards to their adsorption at an interface. This study has used the diblock copolymer poly(2-vinylpyridine)-b-poly(ethylene oxide) (P2VP-b-PEO), which has been shown to form pH responsive polymeric micelles.

The Atomic Force Microscope (AFM) has been used to study the interaction forces of P2VP-b-PEO adsorbed onto silica surfaces. Experiments were undertaken in a number of different solution conditions with a constant polymer concentration. The pH of the solution was increased from a low pH to a high pH with a constant background electrolyte concentration. Experiments were also conducted to examine the effect an increase in electrolyte concentration would have on the adsorbed polymer layer following adsorption at a low pH and a high pH. The results obtained indicate the importance of the anchoring block on the adsorbed conformation of the diblock copolymer as a function of varying solution conditions.

The Effects of Surface Roughness On Wetting: Wetting To Dewetting Regimes

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The contact angle that a liquid drop forms when placed on a solid substrate is the result of three competing surface tensions: namely the solid/vapour, solid/liquid and liquid/vapour surface tensions. In 1936 Wenzel proposed a relationship that correlated surface roughness and the effect this has on the contact angle of a liquid. This relationship has been widely accepted as being correct, however, the vast majority of scientific literature concerning this relationship has either been theoretical or if experimental it has been qualitative rather than quantitative. This presentation addresses the Wenzel relationship in a quantitative and experimental manner.

Chemically homogeneous surfaces are prepared by dip coating glass slides with an amorphous fluoropolymer (Teflon AF1600). The surface roughness is altered by incorporating various amounts of 40mm diameter silica spheres into the polymer coating. The surface roughness is then quantified by optical and SEM techniques. Contact angles of several pure liquids are determined by the Wilhelmy Plate technique as the roughness of the substrate is varied. At contact angles significantly below and above 90° the Wenzel relationship is followed as expected. However, angles in the vicinity of 77° show a departure from the Wenzel relationship, hence the influence of surface roughness on static wetting cannot be uniquely described by Wenzel's roughness factor, r .

Second-order phase transition from and oil-in-water to a water-in-oil emulsion in the triton/toluene/water system

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An emulsion is defined as a mixture of two immiscible liquid phases (oil plus water) where one of the phases is dispersed in the other in the presence of emulsifier. In this project toluene/water/triton emulsions were prepared with a constant oil surfactant ratio of 5:1 over the entire range of oil to water ratios. This allowed us to prepare both oil-in-water and water-in-oil emulsions. Stable emulsions were prepared for a maximum of 80% oil through to a minimum of 0.5% oil.

The method of preparation strongly affects the structure of the emulsions. Emulsions were prepared by three methods, mixing by hand, blending and microfluidising. Each of the methods supplies a different input energy to the system. We have found that there is a correlation between the microstructure of the emulsion and the amount of energy supplied to the system during emulsion formation.

The results were studied by conductivity, Rheology, PFG-NMR and Freeze-fracture TEM. TEM indicates a high degree of polydispersity in the droplet size. All results indicate a second-order phase transition occurs at a surfactant weight percentage in the range of 6-8%. In addition, under limited preparation conditions a bicontinuous emulsion is formed between the oil-in-water and water- in-oil emulsions.

Adsorption of 12-s-12 Gemini Surfactants at the Silica-Aqueous Interface

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The adsorption of gemini surfactants, of the form alkanediyl-a,w-bis (dodecyldimethylammonium bromide), to a silica substrate are investigated using optical reflectometry (OR) and atomic force microscopy (AFM). The adsorption isotherms and kinetics of adsorption have been determined for spacer sizes of 2, 3, 4, 6, 8, 10, and 12. The maximum surface excess correlates strongly with the size of the spacer group. The smallest spacer size yields the largest surface excess.

Soft contact AFM imaging has shown that flattened ellipsoidal aggregates are present on the surface at the shortest spacer lengths. Images of the adsorbed layer cannot be obtained for spacer sizes greater than three, due to a strongly attractive force regime. The origin of the attraction is attributed to proximal desorption induced by the approach of the tip towards the substrate. The linear increase in the area per adsorbed molecule with spacer size suggests that the aggregate structures become flattened with increasing spacer size but are otherwise similar.

Dependence of disjoining pressure on surface chemistry

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Surface forces play a vital role in many fields, e.g. colloid chemistry, materials science, tribology and biology. The surface force apparatus (SFA) has, for many years now, been used for force measurements based on the use of Fringes of Equal Chromatic Order (FECO) interferometry, which enables resolutions of 1 to 2 Å in separation to be achieved. A modified SFA that incorporates a fluid surface (mercury drop) has been developed during Jason Connor's PhD work at the University of South Australia recently, and making use of this experience, a different version of the SFA (the Mark II) at the Max-Planck Institute for Polymer Research has been modified so that it has the same capability. Two types of experiment are planned in this project. First, one can modify a silicon surface by colloidal assemblies and measure the interaction against a mercury drop. This will enable simultaneous force measurements between different surfaces in terms of surface properties (e.g. surface charges, hydrophobicity etc). The second series of experiments is designed to be conducted in a system, which has a bare mica surface against a modified mercury drop. The mercury surface can be modified by the use of so-called self-assembled monolayers (SAM) of alkane thiols. SAMs have been studied extensively on gold surfaces due to their well-organized monolayer structure, and many applications are envisaged in biology, chemistry, medicine and molecular level electronic devices. However, these SAMs are not perfect, generally containing holes and other defects inherited from the underlying gold surface. The use of a mercury substrate enables a defect-free and atomically smooth monolayer to be formed in comparison to the gold substrate. Force measurements provide information of the basic mechanism of the formation of the SAMs, as well as their surface chemistry after formation. Other possibilities include modifying the mercury surface by SAMs of thiols with different functional terminal groups, and by a phospholipid monolayer on the top of an alkanethiol monolayer to mimic a biomembrane surface.

"Nanoparticle" Adsorption at the Emulsion Droplet-Water Interface

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Interactions between silica nanoparticles and polydimethylsiloxane (PDMS) droplets have been investigated as a function of silica hydrophobicity, droplet deformability (controlled by internal cross-linking) and the surface electrical properties of both particles and droplets. Particle adsorption mechanisms were probed through adsorption isotherms with complementary freeze-fracture cryo-SEM imaging of the adsorbed layer structure. Hydrophilic silica adsorbed onto PDMS droplets with surface coverages less than an equivalent close-packed monolayer.

The adsorption affinity and plateau surface coverage show a minor dependence on pH, but are dramatically increased by salt addition. This behavior is attributed to the magnitude of particle-droplet and particle-particle interactions and is subtly controlled by electrostatic and hydration forces, and hydrogen bonding.

In contrast, hydrophobic silica adsorbed onto PDMS droplets with surface coverages in excess of an equivalent monolayer. Sigmoidal isotherms have been obtained for deformable droplets and "affinity increase" isotherms for non-deformable (crosslinked) droplets. Adsorption is pH dependent and increased by salt addition (may rigidify the interface) and considered to be controlled by a balance of electrostatic and hydrophobic interactions. Of further note, the initial adsorption affinity was significantly greater for deformable than non-deformable droplets. This relates to differences in the interfacial layer structure and penetrability. The adsorption mechanisms proposed have implications for the development of particle-stabilised emulsions.

Nanorhology using a Modified Commercial Atomic Force Microscope

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The conventional Atomic Force Microscope has been used for the force-based characterisation of many systems. We have modified a commercial AFM instrument so as to enable the collection of high-resolution nano-rheological information. The addition of small amplitude sinusoidal oscillations to the drive voltage of the Piezoelectric scanner allow us to excite the system and then measure the response of a colloidal probe attached to a cantilever. The information obtained can be used in the calculation of amplitude response, an indication of the energy dissipated in the system; and phase response, indicative of the “stiffness” of a rheological system. Through hydrodynamic modelling it is also possible to determine the spring constant for a colloid probe using a solution of known viscosity. Once this is determined we can then use the system to measure the viscosity of unknown solutions both in the presence and absence of polymer brush layers.

3D Poly(D,L-lactic-co-glycolic acid) Scaffolds for Soft Tissue Engineering

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The future development of soft tissue engineering strategies aims to circumvent the problem of donor scarcity by utilising a polymer scaffold that acts as a vehicle for transplantation of healthy cells. The creation of scaffolds with a controlled, connected porous structure is crucial to understanding the effects of pore size and distribution on the development of a vascular system and growth rate of the tissue within the scaffold.

In this work, poly(D,L-lactic-co-glycolic acid) (PLGA), 1,4-dioxane and water were used to fabricate various porous biodegradable scaffolds suitable for soft tissue engineering, based on a thermally induced phase separation (TIPS) technique. A homogenous polymer-solvent solution was thermodynamically demixed into a polymer-rich phase and a polymer-poor phase by cooling the solution below a binodal solution curve. The quenched polymer solution was subsequently freeze-dried to produce a highly porous structure. This manufacturing method has significant potential for large-scale production of polymer scaffolds for soft tissue engineering.

Morphological study of the 3D PLGA scaffolds for soft tissue engineering has been carried out. Various highly interconnected, open porous structures have been obtained by adjusting process parameters, such as polymer concentration, solvent/non-solvent ratio and quenching temperature. Results showing that both porosity and pore size vary with the process parameters will be presented. Most scaffolds have pore sizes ranging from a few to 200 μm . Such pore sizes are appropriate for nutrient and oxygen transport, as well as cell ingrowth. Some scaffolds have a preferential orientation that may benefit growth of certain tissues like nerves and muscle.

It is necessary to remove the residual solvent from the 3D PLGA scaffolds to ensure materials are safe for implantation. A relatively low level of 0.04% dioxane residue, which is two orders of magnitude lower than achieved in previous work,¹ can be reached via a washing process in combination with freeze-drying, approaching FDA requirements for veterinary medical products.² Results of *in vitro* cell toxicity tests using these scaffolds will be presented, along with preliminary results from animal *in vivo* trials using the vascular loop model developed by the Bernard O'Brien Institute of Microsurgery.^{3,4}

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A Study of Very Small Bubble Formation on Solid-Water Interface using Tapping-Mode Atomic Force Microscopy

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We have studied the formation of very small bubbles (so called nanobubbles) on structured solid-water interfaces. The technique of tapping mode atomic force microscopy (TMAFM) has been used to image the bubbles. Si oxide wafer surfaces were prepared with different degrees of surface roughness and hydrophobicity. The results indicate that small bubbles do not form on either smooth hydrophilic or dehydroxylated Si oxide wafer surfaces immersed in aqueous solutions under known levels of gas supersaturation. However, randomly distributed small bubbles have been observed by TMAFM imaging on both smooth and rough methylated Si wafer surfaces. The existence of these very small gas bubbles on the surface was further demonstrated by the observation of bubble coalescence with time and “Ostwald ripening”. The macroscopic contact angle, measured with respect to the aqueous or gas phase, is very different from the microscopic contact angle detected by TMAFM. We show that this difference apparently lies in the influence of line tension at the three phase contact line.

Interaction of poly (acrylic) acid dispersant with titania pigment particles

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Dispersion of titania pigment particles is very important in industrial processes, such as in the manufacture of paints, paper and plastics. In water-based paints, for example, poor dispersion can result in low gloss, opacity and tint strength, and also lower storage stability. Dispersion can be increased 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 a better choice of dispersant and pulp conditions.

In this study, the interaction of poly (acrylic) acid with titania pigment particles is reported. Adsorption isotherm, electrochemical measurements and also Fourier Transformed Infrared spectroscopy (FTIR) have been used to investigate the nature of the interaction. Adsorption occurs under neutral solution condition, when the particles and the dispersant molecules have opposite charge and also under alkaline condition, when both are negatively charged, although through different mechanisms. Therefore the interaction strongly depends on the pH of the dispersion medium. FTIR confirms that the interaction between poly (acrylic) acid and pigment particles is via physisorption.

Liquid Droplets to Viscoelastic Particles: An AFM Investigation of Interaction Forces, Deformation and Nano-rheology

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The influence that deformation has on colloid interactions is of both academic and industrial interest. AFM was used in-situ to characterise the repulsive forces between a silica probe and cross-linked silicone (PDMS) droplets, with different levels of bulk rheology and hence deformability. At relatively large separations the repulsive forces can be described by electrical double layer interactions for rigid particles. At smaller separations the repulsive force increases less rapidly than expected for rigid bodies and droplet deformation is evident. Thus the zero of separation can be established by shifting the force data such that weak force tails coincide with the known rigid body interaction at large separations¹. The level of droplet deformation can therefore be quantitatively determined.

Highly cross-linked droplets exhibit force curve hysteresis and a significant drive velocity and loading force dependency. This behaviour is due to the viscoelastic response of the PDMS soft colloids and has been theoretically modeled to characterize the nano-rheological parameters². For liquid-like colloidal droplets, with moderate amounts of cross-linking, droplet deformation exhibits a Hookean response with no velocity dependence or hysteresis observed. The deformation and nano-rheology of colloidal droplets are controlled by either the bulk rheology or interfacial tension.

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Photo-induced reversible wetting behavior on structured surfaces

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Surface substrates such as gold and quartz can be chemically modified with self-assembling pyrimidine molecules, attached to long alkyl chains terminated by an appropriate functional group such as thiol. When irradiated with discrete wavelengths of light these surfaces undergo substantial reversible wetting changes over a number of irradiation cycles. The novel aspect of this system is that neither the liquid phase nor the solid phase needs to be altered in order to effect a large change in contact angle. The pyrimidine molecules utilized in the system are C-5 modified uracils that are able to dimerise when irradiated with light of a specific wavelength. The wetting behavior is brought about by a change in the surface charge that, in turn, is highly dependent on the functionality of the pyrimidine groups. Surfaces patterned with monolayers incorporating these dynamic molecules exhibit different wetting properties depending upon the wavelength of light the surface is exposed to. The significance of this work is discussed with respect to molecular structure and possible applications.

Notes

Antimicrobial Coatings - Relationships between Coating Architecture and Antimicrobial Efficacy

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The formation of bacterial biofilms and subsequent infections can cause serious complications in the use of biomedical devices such as catheters, and broadly, effective technology is lacking. Nature has, however, addressed very effectively the problem of microbial colonization of surfaces. For instance, the red algae *Delisea pulchra* secretes brominated furanones that prevent its microbial colonization. These compounds are thought to interfere with bacterial quorum sensing by their chemical similarity with homoserine lactone, an important bacterial regulator. Work to date has produced substantial reductions in bacterial colonization, but not to the high degree required in clinical applications.

This PhD study will investigate how the performance of coatings of antibacterial compounds of the furanone class varies with the mode of immobilization, the architecture, orientation, and interfacial properties of the antibacterial layer, and the properties of the underlying attachment surface. It will also be investigated how molecular interactions between the furanone coatings and molecular components of biological media, particularly proteins, affect the efficacy and longevity of furanone coatings. In this way, fundamental information will be obtained that will enable the designed optimization of the efficacy of such coatings for various applications, particularly against gram-negative and gram-positive bacteria.

Surface Characterisation of Fungi

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Substrates such as chitin (the structural component of many cell walls) and various fungal types have recently been investigated as adsorbent surfaces for heavy metals, with high levels of removal being obtained. The use of biological substrates as a replacement for metal oxide substrates has the advantage that they are renewable, are relatively inexpensive, and are often produced as by-products in fermentation processes. These substrates contain different surface functionality than that found on metal oxides, with carboxyl, phosphate, phosphate diester and, importantly, amino groups being present in addition to the hydroxyl groups. This dissimilar surface functionality has been shown (in some cases) to enhance the removal of heavy metals. The surface properties of these substrates have not, however, been studied extensively.

The aim of this study is to measure the surface properties (surface area, zeta potential and surface charge) of dead biological substrates (of fungal origin).

The surface sites present on a substrate strongly influence the extent of metal ion adsorption. Whilst it has been demonstrated that carboxyl functionality enhances adsorption, the effect of the other surface site types present on biological substrates can be of considerable scientific importance. From an industrial perspective, the development of a biological system utilising a renewable and inexpensive substrate for the removal of aqueous heavy metals is desirable, and a thorough understanding of the surface characteristics is central to the development of this process.

Structure of Mixed Polymer and Surfactant Adsorbed Films on Mica

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The structure of adsorbed surfactant films containing tethered polymers is being studied with the aim of elucidating the role of polymer morphology and anchoring mechanism on the adsorbed layer structure. Atomic Force microscopy (AFM) and Surface Force Apparatus is being used to observe and measure structure of and interactions between such mixedadsorbed layers.

Using soft-contact AFM imaging, the adsorption of dodecyltrimethylammonium bromide (DTAB) monomers onto mica from solutions below the critical micelle concentration (CMC) have revealed an adsorbed layer comprised of short cylindrical micelles just below the CMC lengthening with increasing concentration. Currently the effect of uptake of long-chained polyoxyethylene di-block copolymers with various hydrophobic moieties by bilayers of double-chained surfactant didodecyldimethylammonium bromide (DDAB) on mica is being studied and compared with previous results for uptake into DTAB adsorbed layers. (1)

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The Preparation of Model Sulfide and Oxide Surfaces for Surface Enhanced Raman Scattering

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Surface enhanced Raman scattering (SERS) has been used for many years in the fields of electrochemistry and catalysis.^{1,2} The requirement of using a metal surface (such as gold or silver) to give the surface enhancement has limited the application of SERS to other areas. Here, we present two methods for producing model sulfide and oxide overlayers on gold surfaces that will allow SERS to be applied to the area of mineral processing. The first technique involves the preparation of sulfide overlayers by the sulfidation of evaporated zinc metal on rough gold surfaces. The second involves the coating of gold surfaces with oxide-coated gold sols. Both techniques give rise to surfaces that are suitable for SERS and provide a means of using SERS in studying polymer and collector adsorption onto model mineral surfaces.

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2. M. Weaver, 'Surface-enhanced Raman spectroscopy as a versatile in situ probe of chemisorption in catalytic electrochemical and gaseous environments', *J. Raman Spectrosc.* 33, 2002, 309-317

The Effect of Electric Fields of Thin Liquid Films

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Thin liquid films between mica surfaces can be investigated in the surface forces apparatus (SFA). By soldering wires to evaporated silver on the outer surfaces of the mica, potentials can be applied across such liquid films in order to study their behaviour in an external electric field. Experiments are planned on aqueous solutions to see whether the electrical double layer, and hence the interaction between the mica surfaces, is modified by the field. It will also be of interest to see if an electric field has any influence on surface charging effects in nonpolar liquids. Techniques for making these measurements, along with any results obtained, will be presented.

Surface Modification of Ester-Based Biomaterials

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Poly(lactic-co-glycolic acid) is one of the most popular biocompatible, biodegradable polymers currently in use for tissue engineering research, due to its high biocompatibility, good mechanical properties and processability. However, the surface properties of the polymer are less than ideal for the adhesion and proliferation of cells. This poster presents an overview of the approaches taken in the past, and gives details of the method of controlled partial surface hydrolysis, which we are currently exploring in detail.

Molecular Adsorption onto Gold Sols: A Surface Enhanced Raman (SERS) Study

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Surface enhanced Raman scattering (SERS) is an established technique for studying adsorption onto gold surfaces and colloids.

1. The goal of this work is to apply SERS to the study of molecular adsorption at oxide overlayers on gold colloids.
2. However, optimisation of the SERS effect for bare gold colloids is necessary for this demanding application as the SERS enhancement attenuates in the presence of an oxide overlayer.
3. The enhanced Raman signal at a gold surface is due to the creation of surface plasmons in the substrate by the electric field of incident light. The excitation of surface plasmons is related to the roughness of the surface being investigated (or particle size for a gold colloid) and the wavelength of the light used to induce Raman scattering. We present a surface enhanced Raman study of the adsorption of pyridine onto gold colloids, as a function of the size of the gold particles and the wavelength of the incident laser.

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2. L. M. Liz-Marzan, M. Giersig, P. Mulvaney, 'Synthesis of Nanosized Gold-Silica Core-Shell Particles', Langmuir, 12, 1996, 4329-4335.
3. S. A. Wasileski, Shouzhong Zou, M. J. Weaver, 'Surface-Enhanced Raman Scattering from Substrates with Conducting or Insulating Overlayers: Electromagnetic Model Predictions and Comparisons with Experiment', 54, 2000, 761-772.

Adsorption Behaviour of the Various Crystal Structures of TiO₂

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Previous studies have been undertaken to adsorb aqueous heavy metals onto many colloidal substrates in an attempt to model metal removal from industrial waste. One theory is that metal ions must hydrolyse, thus lowering their charge, prior to adsorption. This lowers the change in solvation energy involved in adsorption which otherwise prevents the free (unhydrolysed) metal ion from adsorbing.

TiO₂ is an unusual oxide, in that some of its crystal structures have a dielectric constant similar to that of water and thus there is little difference to the solvation energy of an adsorbed or free metal ion. There should, therefore, be little solvation energy involved in adsorption and metal ions should not need to hydrolyse prior to adsorption. Speculatively, adsorption would now be largely controlled by the electrostatics of the system. The surface charge of the TiO₂ becomes increasingly negative with increasing pH, thus increasing the "driving force" for adsorption.

Experimentation found that adsorption of metals onto TiO₂ (anatase crystal structure, dielectric constant ~ 31) is not driven by electrostatics alone. There is a weak trend between adsorption and metal hydrolysis, but specific affinities appear to be present between individual metals and the substrate. The order of adsorption with respect to pH was found to be Pb²⁺ > Cr³⁺ > Cd²⁺ = Zn²⁺ > Ni²⁺ > Mg²⁺ > Ca²⁺. This closely matches the order for adsorption onto coal, where carboxyl surface functionality is believed to be the driving force for adsorption.

Future work will concentrate initially on measuring and, if possible, controlling the dielectric constant of a TiO₂ sample so that the dielectric constant of the sample will approach that of water (~78). Thus adsorption will be studied on chemically similar, but physically dissimilar, substrates.

Interaction between liposomes and surfaces studied by attenuated total reflectance

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Liposomes are important vehicles for drug delivery and knowledge of their interaction with surfaces is critical in understanding targeted delivery^{1,2}. Fourier transform infrared (FTIR) spectroscopy in attenuated total reflectance (ATR) mode was used to study the interaction of liposomes with model surfaces, and to study the influence of liposome properties on the kinetics and thermodynamics of surface interaction. Upon liposome adsorption at the (modified) ZnSe internal reflection element - water interface, IR bands due to the liposome components (C-H stretch & scissor and C=O stretch) were observed to increase with time. The quantification of these absorbances enabled adsorption amount G vs time data to be obtained. The liposomes under investigation were composed of lecithin, PEG-lipid and cholesterol, and prepared using either sonication or membrane filtration. The liposome composition & size, solution conditions and the characteristics of the adsorbent surfaces have been shown to influence liposome adsorption kinetics and interaction mechanism (e.g. liposome to bilayer transformation).

Reference:

- 1 H Egawa, K Furusawa, 1999. Liposome adhesion on mica surface studied by atomic force microscopy. *Langmuir*, 15:1660-1666
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Novel Explosive Formulations Using Ionic Liquids

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Ionic liquids (IL's) are a relatively new class of compounds whose properties are well explained through the generic name.

These compounds are based on organic molecules although they have ionic functionalities. This project aims to synthesise new and established IL's, and characterise their physical properties including phase behaviour, surface activity and solubility. The IL's will then be formulated with existing ANFO emulsions and tested as possible new explosive products.

The Extraction of Gold From Low-Grade Ores

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The Cut-off grade for extraction of gold from ore is one of the main factors in the economic viability of all gold mining ventures. Over the years various advances in technology, from the development of cyanidation in the late 19th century to more recently the introduction of Carbon-in Pulp and Carbon-in-leach techniques in the 1970's has led to major reductions in cut-off grade from around an ounce per tonne down to a gram per tonne. The focus of my work in this area will be to develop a method to further reduce this cut-off grade and make viable the mining of deposits that were previously uneconomic. The work will be done on three ores from the Pilbara region of northwest Western Australia with focus set on the development of sustainable mining.

Surface Chemistry of Bacteria Associated with Foams in Wastewater Treatment

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One of the most efficient ways to treat wastewater is by activated-sludge. But this has a major drawback in that nearly all plants at some stage produce stable foams. This is a major problem because it can affect the quality of the effluent, depending on where in the plant the site of the foaming incident has occurred, and the foam can also contain pathogenic microorganisms that become wind-borne creating potential health risks.

This project aims to determine the role played by microorganisms in the production and stabilisation of foams in wastewater treatment plants. To do this we are studying the nature of the bacterial surfaces using contact angle measurements and the Microbial Adherence to Hydrocarbons (MATH) assay to ascertain the hydrophobicity of those microorganisms found to be present in stable foams. Further characterisation of cell surfaces will be conducted using Attenuated Total Reflectance – Fourier Transform Infrared (ATF-FTIR) spectroscopy. Correlation of the hydrophobicity and ATR-FTIR data may provide information on the underlying surface characteristics that determines why some bacteria are found in foam and others not.

The Influence of Polymers on 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 solid particle involves the formation of a finite contact angle. The sessile-bubble technique will be used to measure the contact angle between a bubble and specific solid surfaces. The surfaces studied embrace model systems (SiO_2 , TiO_2 , ZnS, PbS), whilst the polymers are well-defined polyacrylamides and polysaccharides. The contact angle will provide important information regarding whether or not a bubble will adhere to a surface and the stability of the bubble-particle attachment. The contact angle will be linked to force-distance data where bubble-particle interactions are determined by colloid probe AFM measurements.

Photo-induced wettability of metal oxides

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It has recently been found that the wettability of TiO_2 is influenced by irradiation (Hashimoto et al). This wettability change has been observed for both anatase and rutile surfaces, in the form of either a single crystal or as polycrystals. It is believed, although a clear explanation is yet to be given that the wettability change is due to structural change of TiO_2 itself, rather than the well-known photocatalytic decomposition of contaminant organic compounds.

In our research, photo-induced wettability changes of TiO_2 are investigated. TiO_2 surfaces exhibit a water contact angle of 70° . UV irradiation converts the surface to a hydrophilic state, i.e. the water contact angle is 0° . The surface returns to a hydrophobic state after irradiation with visible light. This conversion was found to be reversible over numerous cycles. We have shown that the process applies to flat surfaces as well as to small particles attached to a hydrophilic host surface such as silica.

In order to gain further insight into this phenomenon it is necessary to expand this research from TiO_2 to other metal oxides. A mechanism for the wettability change is not completely known, mainly due to the fact that the surface states responsible for these wettability conditions are ill-defined. The influence of surface morphology, light intensity, surface defects, etc for the case of TiO_2 and other metal oxides is being studied, with a strong emphasis on identifying the surface states responsible for these remarkable wettability effects. A relationship between the photo-induced wettability changes and photocatalytic activity is also being investigated.

De-aeration of Liquids Under an Ultrasonic Field

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There has been a growing interest in the industrial application of ultrasound, especially in the de-aeration of process fluids such as, carbonated drinks, molten metals, photographic and electroplating solutions, and food products [1]. The presence of certain additives such as, surface active materials and viscosity modifiers can greatly influence the de-aeration by affecting the mass transfer rate of the gas through the air/liquid interface of the bubble wall. Coupled with ultrasound, the system becomes complicated.

The primary objective of this investigation is to gain a better understanding of how ultrasound, surface tension and interfacial viscosity or elasticity affect the dissolution rate of bubbles in liquids. During the de-aeration process in the presence of ultrasound for example, bubbles can (i) undergo cavitation, a process by which the bubbles grow by rectified diffusion [2] to a critical size within a few acoustic cycles, collapse and break into smaller bubbles (transient cavitation) or become stable and oscillate over a number of acoustic cycles and then collapse (stable cavitation) and (ii) grow above a certain size range (by rectified diffusion or bubble coalescence) when they can be expelled via buoyancy force.

Ultrasonic degassing may involve several processes. The bubble collapse by cavitation process can lead to the breakup of larger bubbles into smaller nuclei that are below certain size range that would not affect the properties of the product being considered. The stable bubbles can be kept levitated by an ultrasonic field, long enough to self-dissolve. The bubbles can be grown to large enough sizes, to be expelled by buoyancy force. A number of people have looked at the rate of growth of a single bubble as a function of acoustic pressure amplitude, and the effects of surfactants on the rate of mass transfer across the oscillating interface of the bubble, both experimentally and theoretically [3]. However, very few studies have been carried out on rheologically complex systems where the bulk as well as the interfacial rheology becomes important. This presentation will include the progress to date on the construction of an experimental unit with a cell and an imaging unit, which will allow the changes in radius of a single bubble in the presence and absence of ultrasound to be monitored. The effects of surface tension, viscosity and pressure on the rate of de-aeration of liquids will be discussed. Some preliminary experimental data on the rate of bubble dissolution in the absence of an ultrasonic field and on the interfacial rheology of fluids will also be presented.

References:

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- [2] L.A. Crum, *Ultrasonics*, 1984, 22, 215-223.
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Applications of vibrating AFM cantilever

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Atomic Force Microscopy (AFM) is a powerful tool for studying the surface forces between two surfaces or to image a surface. Sader (Journal of Applied Physics 1998, 84, 64-76) produced the first detailed theoretical analysis which models the vibrational behaviour of the cantilever beam when excited by an arbitrary driving force immersed in a viscous fluid.

Using the theoretical analysis carried out by Sader, we are trying to:

1. Examine torsional forces
2. Use the lever as a rheology probe
3. Build a chemical sensor

Results of these studies will be the subject of my poster

Ultra fine particle-bubble interactions

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Extensive research to date has focused upon particle-bubble interactions in the collision or interception flotation regime, which typically occurs for particles greater than 3µm in diameter.

However, as particles decrease in size and approach molecular dimensions, Brownian diffusion becomes the primary interaction mechanism.

Previous experimental research conducted at the Ian Wark Research Institute has showed evidence of a minimum in collection efficiency (E_{coll}). This is dependent upon the particle size and physico-chemical conditions within the flotation cell. As the particle size decreases further, an increase in collection efficiency is observed and currently, the fundamental mechanism for this 'turnover' is not yet fully understood. It will certainly depend upon diffusion at least, as well as other factors.

The aim of the proposed research is to:

- Investigate the mechanisms governing ultrafine particle flotation.
- Investigate the effect of varying the bubble size, particle agglomeration/flocculation and power input upon ultrafine particle flotation.

Develop a capture model and rate constant for ultrafine hydrophobic particles.

We will outline interactions between bubble and ultrafine particles, the proposed experimental research and an overall research strategy.

A COMPARISON BETWEEN NORMAL AND ALCOTHERMALLY TREATED SBA-15

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In 1992, the invention of the M41S family of mesoporous materials by researchers at Mobil Corporation opened a new chapter in the area of protein adsorption. SBA-15 is a mesoporous molecular sieve which possesses a regular hexagonal array of uniform pore openings with large pore diameters and is a potential adsorbent for proteins in applications such as wine haze protein removal. However, the stability of mesoporous molecular sieves in aqueous solutions is a significant limitation to their use in industrial applications. Alcothermal treatment of SBA-15, involving a thermal treatment in alcohol at 100°C, has been proposed as a promising method of improving its stability. In this work, a comparison between normal and alcothermally treated SBA-15 was performed. The structure of the materials with and without the alcothermal treatment was characterised by X-ray diffraction and gas adsorption analysis. The structural stability of these materials was then tested in buffer solutions of relevance to food and pharmaceutical processing. The materials were found to compare favourably to the M41S materials with enhanced stability.

Light scattering by natural waters

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Remote sensing is a technique that is very useful in determining the concentration of phytoplankton, bacteria's, silt and other constituents of natural waters over large ranges. To be put into practice, more information is needed about the light scattering properties, specifically the intensity form factor.

The form factor measurements of T.J. Penzold[1] on San Diego harbour water have been used as a standard for the light scattering properties of natural waters throughout the world. Recently, however, it was found that this was not satisfactory for Australian waters.

Form factor measurements have been taken on two samples of Australian natural waters, supplied by CSIRO, over the angular range of 15° to 140°. The samples were found to have a similar trend but not close enough to the Penzold measurements, thus concluding that it is not a suitable standard for the light scattering properties of Australian natural waters.

The objective now is to determine the scattering functions of several samples. These can then be used for remote sensing purposes at CSIRO.

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Layer-by-layer Assembled Mixed Spherical and Planar Gold Nanoparticles: Control of Interparticle Interactions

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Gold nanoparticles (NPs) were prepared by reduction with salicylic acid in aqueous solution. The resulting dispersions were found to contain a mixture of flat triangular/hexagonal and smaller close-to-spherical NPs. Such nanocolloids display two clearly differentiated surface plasmon bands at 540 and 860 nm associated with spherical and anisotropic triangular/hexagonal NPs, respectively. Layer-by-layer (LBL) assembly was used to deposit thin films of the Au colloids. UV-visible data indicate preferential adsorption of the flat particles on polyelectrolyte films. Importantly, a new band developed at 650 nm as the number of the Au NPs bilayers increased. This finding indicates that there exists a strong interaction between the NPs in adjacent layers, resulting in the surface plasmon absorption at a new wavelength. The insertion of extra polyelectrolyte or montmorillonite layers between the Au bilayers was shown to reduce the interlayer interaction.

b-Cyclodextrin-Alcohol mixed systems as a reaction media

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The kinetics of the acid hydrolysis of N-methyl-N-Nitroso-p-toluensulfonamide (MNTS) were studied in media containing different alcohols and b-cyclodextrin (b-CD). The results were interpreted in terms of the proposed kinetic model. The model takes into account the formation of both b-CD/alcohol and b-CD/MNTS complexes and also the presence of free cyclodextrin. The variation of the apparent b-CD/MNTS binding constant in the presence of alcohol will allow us to obtain the different b-CD/alcohol association constants.

Investigation of bubble - surface interactions using SFA

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A knowledge of the electrical potential on bubble surfaces is important to gain deeper insight into the structure of gas-liquid interfaces, and to quantify the forces between bubbles or between bubbles and particles. From previous research the data on surface charge of bubbles are very often contradictory because of side effects of the electrokinetic methods used, and impurities at the interface. Taking into account these facts, 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 research into fluid surfaces, including air bubbles. This project will aim to determine the double layer potential on the bubble surface from analysing double-layer forces measured between a bubble and mica, whose potential will have been established from previous experiments. The eventual plan is to conduct experiments with different electrolyte solutions and different gases to investigate their effects on the gas/water interface potential.

Time Resolved Evanescent Wave Induced Fluorescence Spectroscopy on Bovine Serum Albumin adsorption to Hydrophilic Silica.

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Time-Resolved Evanescent Wave Induced Fluorescence Spectroscopy (TREWIFS) is a technique which utilizes interface sensitive evanescent waves, produced through a total internal reflection geometry, to photo-excite fluorophores within the interfacial region. Hence, if the interfacial region consists of adsorb macromolecules, the fluorophore can be used to probe the surface conformation of the macromolecule species. The fluorescence emission, in particular the time-resolved fluorescence, yields information intrinsically related to the nature of the fluorophore and its immediate environment, thus providing highly sensitive spatial probing of the interfacial phenomena. When coupled with Time-Resolved Anisotropy Measurements (TRAMS) motional information on picosecond time scales of the excited state fluorophore can be obtained, allowing the study of rotational motion of macromolecules in solid/liquid interfacial regions.

These techniques have been utilized to characterize the adsorption of the protein Bovine Serum Albumin (BSA), non-covalently bonded with the fluorophore 1-anilinonaphthalene-8-sulfonic acid (ANS) onto a hydrophilic silica surface. Serum Albumins are blood proteins known to rapidly cover synthetic materials used for biocompatibility. Therefore considerable interest has been shown in understanding conformation change within proteins upon adsorption.

Adsorption of block copolymer micelles

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In this project the adsorption of a block copolymer [dimethylaminoethylmethacrylate-diethylaminoethylmethacrylate (DMA-DEA 79:21 $M_n \sim 20\ 000$)] at the solid/liquid interface has been investigated. Optical reflectometry was used to examine the adsorption characteristics of this copolymer onto silica as a function of concentration and pH. Of particular interest currently is the adsorption at pH 9 where the block copolymer is known to form micellar aggregates in bulk solution. Pyrene fluorescence probing has also been used to study the aggregation behaviour of this copolymer. The determination of an approximate CMC was carried out by this technique. A critical micelle pH range (CM-pH) was also defined where at low pH the copolymer exists as unimers in solution and above pH 8 micelles are formed.

Particle Wettability

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The wettability of natural and manufactured surfaces is very important as it determines the nature of the contact with various liquids. Thus whether a liquid drop stays on or rolls off a surface may greatly influence the degree of adsorption, chemical conversion or dissolution occurring at the surface during contact. In mineral floatation particles are selectively adhering to bubbles depending on their wettability. The contact angle is a parameter of paramount importance and its appropriate measurement is strongly needed. There are well-established techniques available for contact angle measurement (Sessile Drop, Adhering Bubble, Wilhelmy Plate, Capillary Rise) but in all cases a surface with a large area and small curvature are required. Some techniques can be used for fibers and filaments but they are not applicable to small particles. Typically the wettability of particles is measured by following liquid penetration of liquids through a bed of particles. Although useful information can be obtained for mineral particles the method has some drawbacks. The contact angle value is a complex average over many particles thus differences between particles may be smeared. The powder preparation (drying, storing, packing) may influence the outcome of the measurement and probably affects the surface properties of the particles. This is especially disadvantageous when modeling the efficiency of floatation.

The aim of this project is to reliably determine the contact angle of various liquids on single particles of size range 1-150 μm . The project will be approached in three phases, each of increasing complexity. Phase one will use silica particles of different sizes. In phase two the methodology will be extended to particles of different shape, aspect ratio and chemical composition. In phase three studies will be aimed at characterizing particles with heterogeneous surfaces, wider size distribution, complex shape etc, including several mineral particles. The ultimate goal is to provide reliable values of the contact angle for use in the IWRI floatation model.

Modelling Plate-and-Frame filter presses for the water and wastewater industries

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The sludges produced during water and wastewater treatment have considerable and increasing disposal costs, regardless of the disposal method used. The costs of transport, storage and/or incineration of the products are reduced if the dewatering operations are improved.

One device often used by the industry for the final stage of dewatering is the plate-and-frame filter press. This presentation will outline work undertaken to model the behaviour of two types of presses – fill-only and fill-and-squeeze. The aim has been to take measurements of the material properties and to use them with the model to accurately predict the performance of a given sized press with a known pressure profile, without resorting to empiricisms. The model has been validated through site visits, where improved throughputs and corresponding savings were provided via changes to operation. Several design issues have also arisen.

Structured Fluid From Polyether-modified Poly(acrylic acid): Synthesis and Characterization

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A homogenous gel composed of lightly cross-linked poly(acrylic acid) networks grafted onto polyether chains (Pluronic F127) are synthesised. The cross-linked copolymer gel undergoes thermally reversible phase transitions at the critical micellization temperature (CMT). The cross-linked copolymer contains long chains of Pluronic at temperature below the CMT (25 deg). At elevated temperature, the dangling Pluronic chains rearrange and the hydrophobic poly(propylene oxide) chains aggregate within the cross-linked copolymer, forming a micelle-like structure with distinctive hydrophobic and hydrophilic domains. This thermoreversible copolymer has great potential in drug delivery systems and separation of macromolecules and solutes in response to the various degree of solute hydrophobicity.

Gemini Surfactants Through Polymerisation

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Gemini surfactants consist of two ordinary surfactant units with a short chain linking the hydrophilic heads. Structurally related to these are trimeric and larger oligomeric surfactants, which comprise correspondingly more linked units. These compounds represent a special class of amphiphile, and display interesting and unusual self-assembly and rheological properties. Because they are produced via a specific synthetic process and can therefore be expensive, they are generally utilised in niche applications^{1,2}.

We propose to study the production of a range of oligomeric surfactants via polymerisation rather than synthesis. While the notion of polymerising surfactants has been widely explored for numerous applications, using many different approaches and with widely varying degrees of success^{2,3}, polymerisation has not yet been used with the specific aim of producing oligomeric chains.

In this context, the use of polymerisation techniques could allow a distribution of amphiphilic oligomers to be produced by the same process. Such families of molecules can then be studied either as a series of pure compounds or as mixtures, and their behaviour compared to that of oligomeric surfactants produced by more conventional methods.

We are utilising polymerisable surfactants (or "surfmers") consisting of quaternary ammonium surfactants with allyl or methacrylate groups attached to the nitrogen atom. Polymerisations are carried out in both non-self-assembled (using an organic solvent such as toluene or chloroform) and self-assembled states (micellar solutions), and analysis of the products is performed using NMR and HPLC.

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3. Asua, J.M.; Schoonbrood, H.A.S., *Acta Polymerica* 1998, 49, 671-686

Catalytic Conversion of Mixed Plastics Waste into Useful Chemicals

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Plastics are ubiquitous and the total amount of plastic wastes generated in Australia is growing rapidly. Due to their low biodegradability, land filling can cause serious environmental pollution. In 2000, Australia discarded 753,000 tonnes of waste plastics to landfill. However, waste plastics have great potential to yield useful chemicals and energy from tertiary or chemical recycling. Most studies of chemical recycling of addition polymers have been performed with acid catalysts due to their widespread use in the petroleum industry and their known ability to cleave carbon-carbon bonds. In contrast, the literature available involving the use of base catalysts to effect the degradation process is rather scarce.

In this project, the objective is to develop a novel process, a fixed-bed flow reactor, making use of catalytic systems that have not been thoroughly investigated to date: superbase catalysts obtained by post-synthetic modification of zeolites and novel hydrotalcite materials obtained by coprecipitation. The specific expected outcomes from the project include: determine the cracking products distributions of selected polyolefins over a series of base catalysts; deduce the mechanistic chemistry; and identification of a catalyst capable of achieving high conversion of polymer waste and yielding at least 80 wt% of useful chemicals.

The catalyst systems in this project will be mainly X and Y zeolites containing alkali and alkali earth oxide clusters (specifically those of Li, K, Cs, and Mg) as well as anionic clays with various combinations of divalent and trivalent cations (Ni^{2+} , Al^{3+} , Cr^{3+} , Mg^{2+} , etc.). Catalyst characterisation will include XRD, TGA, BET, Scanning Electron Microscopy, IR, and PSD to determine the fundamental properties of the materials. Cracking reactions will be conducted at temperatures between 500°C and 800°C using polyethylene, polypropylene and polystyrene as model feeds. The study will then be extended to a blend of different polymers and finally to a test batch of mixed municipal plastic waste. Liquid and gaseous products will be analyzed using GC and GC-MS.

"Preparation of Q-PbSe"

Joel vanEmbden

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Melbourne 3010 Victoria, Australia.

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There have been many achievements in the field of semiconductor nanocrystals. The most notable of these has come from the manipulation of CdSe. More recently there has been a push to optimise the synthesis of infrared emitting colloids. Lead selenide allows access to this region, with quantum confinement beginning at $\sim 0.5\text{eV}$. I will present my studies on both lead colloid and lead selenide semiconductor syntheses and optimisation.

Introduction to Electrochemical Double Layer Capacitors

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Electrochemical Double Layer Capacitors are a major section of research in the electrochemical community at present and they are gearing up to be a major component in the electronics industry. At the onset of commercialisation of these components, then, it is important for the wider scientific community to understand the basics of EDLCs, their importance and their differences from batteries and electrolytic capacitors.

This poster presentation then attempts to offer a concise explanation of the technology of Electrochemical Double Layer Capacitors (EDLCs) with advantages and disadvantages when compared to batteries and electrolytic capacitors.

Torsional frequency response of cantilever beams immersed in viscous fluids with applications to the Atomic Force Microscope

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The frequency response of a cantilever beam is strongly dependent on the fluid in which it is immersed. Recently, Sader [J. App. Phys. (1998)] presented a theoretical model for the flexural vibrational response of a cantilever beam, that is immersed in a viscous fluid, and excited by an arbitrary driving force. Due to its relevance to applications of the atomic force microscope (AFM), we extend the analysis of Sader to the related problem of torsional vibrations, and also consider the special case where the cantilever is excited by a thermal driving force. Since longitudinal deformations of AFM cantilevers are not measured normally, combination of the present theoretical model and that of the recent study of flexural vibrations enables the complete vibrational response of an AFM cantilever beam, that is immersed in a viscous fluid, to be calculated.

The surface as a stage for chemical reactions (and the tools we use to watch the stage show)

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Many chemical processes involve the interactions of molecules at surfaces. In order to understand and ultimately control such processes, we need to consider the chemical and physical changes that occur on a molecular level. We can think of the surface as a 'stage' for chemical reactions, where molecules adsorb, diffuse, dissociate and react to form new chemical species. Here, I will discuss how these ideas relate to heterogeneous catalysis research, and to the various spectroscopic tools available for 'watching the stage show' on the catalyst surface. In particular, the adsorption of small molecules, such as CO, on single crystal metal surfaces will be considered. The interactions of much larger molecules, such as proteins and polymers, at surfaces will also be discussed, as will the tools for investigating these complex systems. Specifically, novel time-resolved fluorescence techniques and their ability to reveal information on the conformation, orientation and rotational dynamics of adsorbed macromolecules will be examined.

Colloidal Interactions at the Oil-water Interface in the Presence of Surfactant or Co-polymer

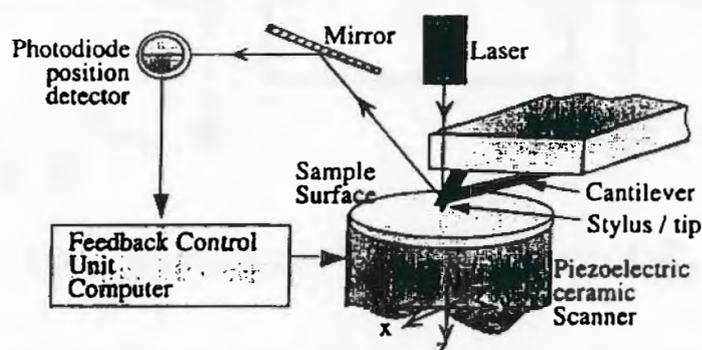
Raymond R. Dagastine, Dennis C Prieve[†], and Lee R White[†]
School of Chemistry, University of Melbourne, VICTORIA, 3010
< rrd@unimelb.edu.au >

AFM measurements of a rigid probe at an oil-water interface have become common place, but interpretation of the data is complicated by the deformation of the surface, requiring a method to deconvolute the colloidal interaction from the effects of interface deformation. The adsorbed species have an enhanced lateral mobility compared to rigid systems allowing for the possibility of rearrangement mediated by interaction forces. We examine the behavior with either an anionic surfactant or a PEO-PPO copolymer adsorbed at an oil-water interface, probed with a rigid sphere. Without any prior knowledge of the force law, we extract the interaction energy between flats from the AFM force measurement. We then compare the interaction energy deconvoluted from the AFM force measurements to theoretical predictions based on independently measured system parameters (*e.g.*, zeta potential, surface tension). The comparisons between theory and experimental results provide evidence of surface rearrangement of charged species or local changes in interfacial tension. We also discuss the behavior of the oil/drop experiment for large drop deformations where the oil-water interface “wraps” around the probe particle.

EXPERIMENTAL TECHNIQUES

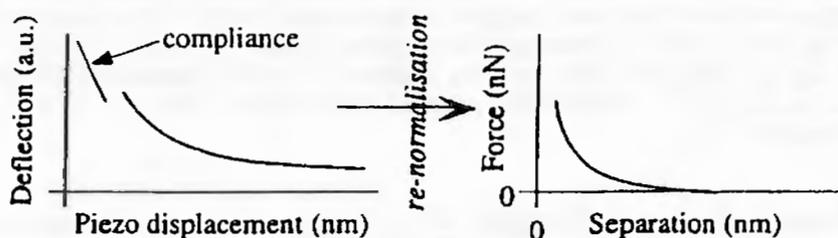
(ATOMIC) FORCE MICROSCOPY

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 $O(\text{pN} - \mu\text{N})$, distance $O(\text{\AA} - \mu\text{m})$ }. Tip composition and geometry may be varied.

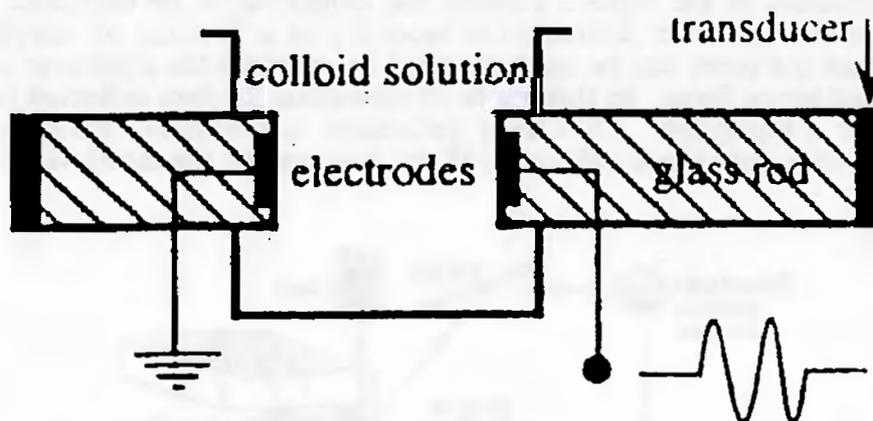


Suggested reading:

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.

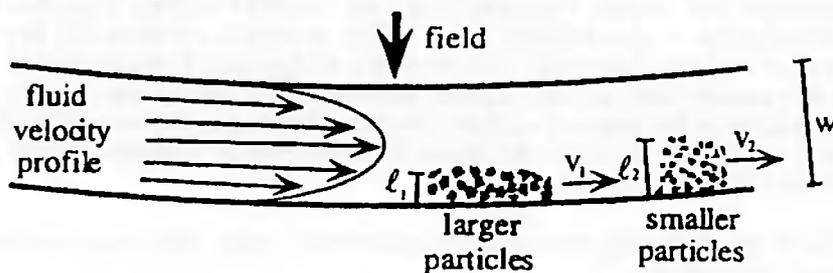
The dynamic mobility is a complex quantity equal in magnitude to the ratio of the particle velocity divided by the applied field. It is complex because there is a phase lag between driving frequency and the response of the particles to that field. As the applied field tends to zero the mobility becomes 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.

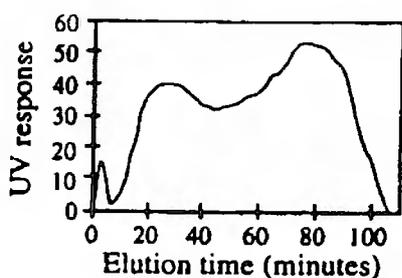


A schematic diagram of a sedimentation FFF channel.

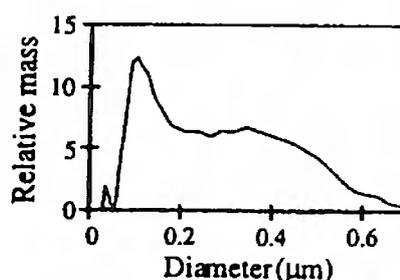
The data obtained is called a fractogram. The elution time can be converted to particle diameter using the equation;

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

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

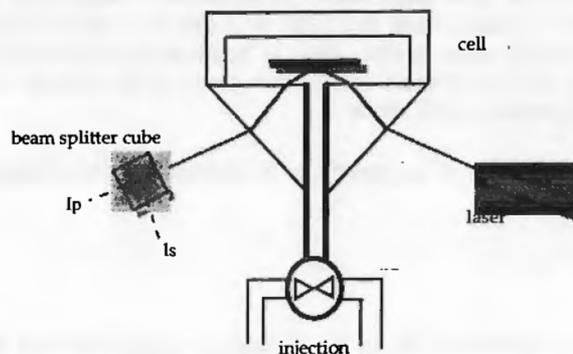
$$\frac{Hc}{R(\theta, c)} = \frac{1}{M_w} \left[1 + \frac{r_g^2 K^2}{3} \right] + 2A_2 c$$

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

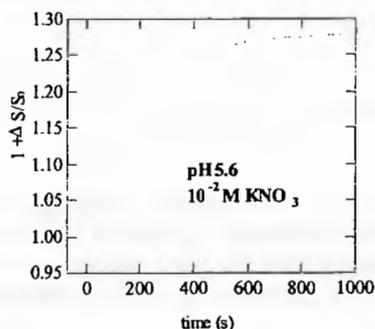
REFLECTOMETRY

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 thick) 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 polymers 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 conformation 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.

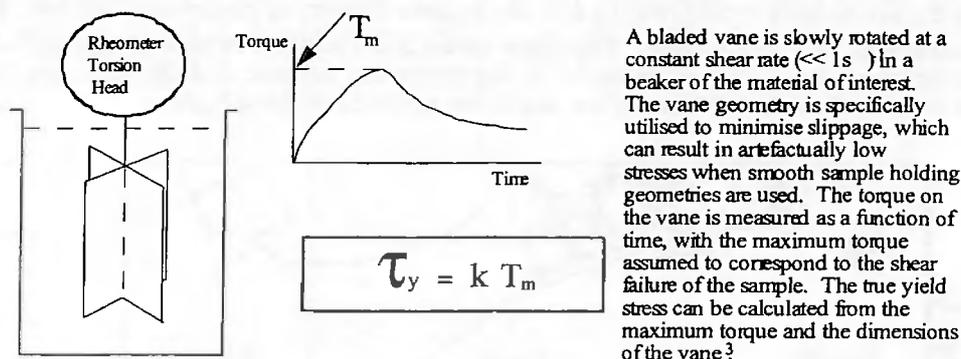
Suggested reading:

Dijt, J.C., Cohen 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

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 measureable 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

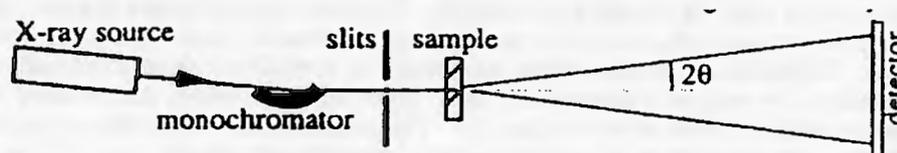
The viscoelastic characteristics of colloidal systems are also characterised by concentric cylinder rheometry, through measurement of the response to application of a sinusoidal stress or strain. G' , the storage or elastic modulus and G'' , the loss or viscous modulus, are determined from the stress-strain inter-relationship and the phase angle (δ) between them: $G''/G' = \tan \delta$. 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

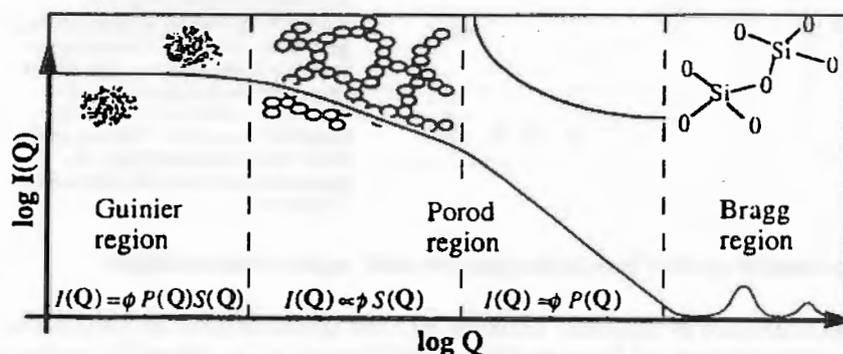
Small angle X-ray scattering allows the physical characteristics - dimensions, surface roughness and spatial arrangement - of particles (\AA 10 - 1000 \AA) in solution to be studied. In practice a collimated beam of monochromatic X-rays is passed through the sample and the intensity of scattering measured 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, $(\rho_1 - \rho_2)$, where ρ_1 and ρ_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 $(\rho_1 - \rho_2)^2$ 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.

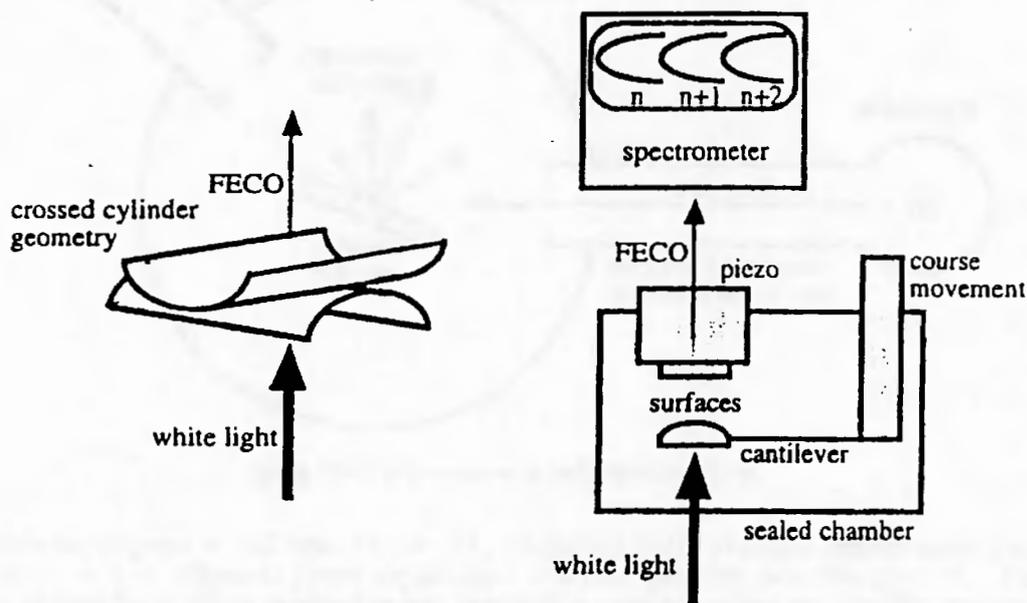
Suggested reading:

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 $O(0.1\mu\text{N-mN})$, distance $O(\text{\AA}-\mu\text{m})$ }.

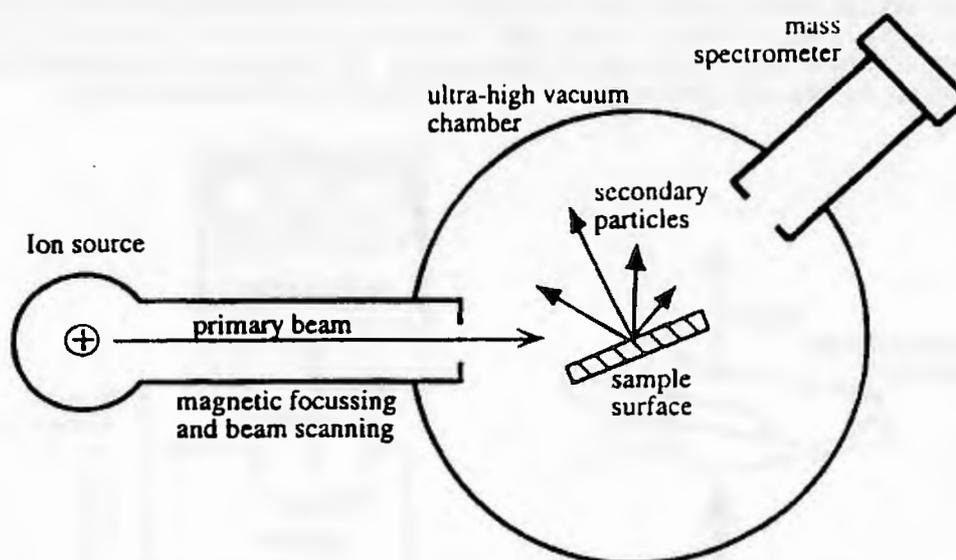
The crossed cylinder geometry approximates well as a sphere, with a radius equal to the geometric mean curvature of the two cylinders, interacting with a plane. Surfaces are restricted to those that can easily form transparent, thin smooth films, e.g. silica, sapphire, mica. Alternatively the interferometer 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 , 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.

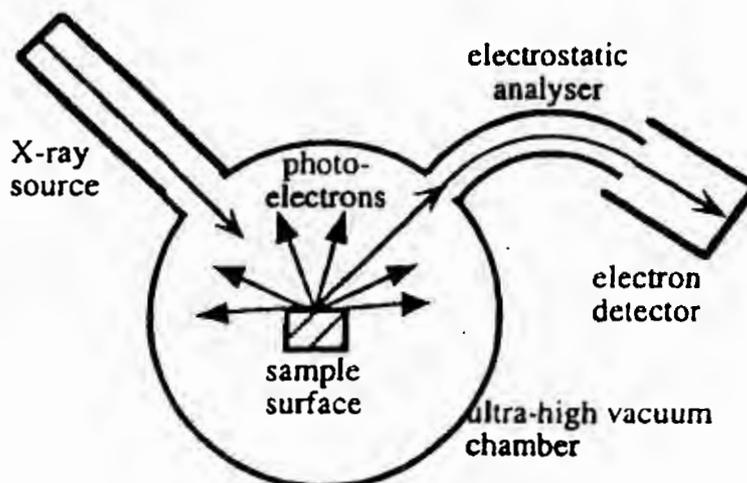
Mass spectrometry not only provides isotopic discrimination, but both negative and positive species can also be analysed.

Suggested reading:

Benninghoven, A., Rüdener, 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.

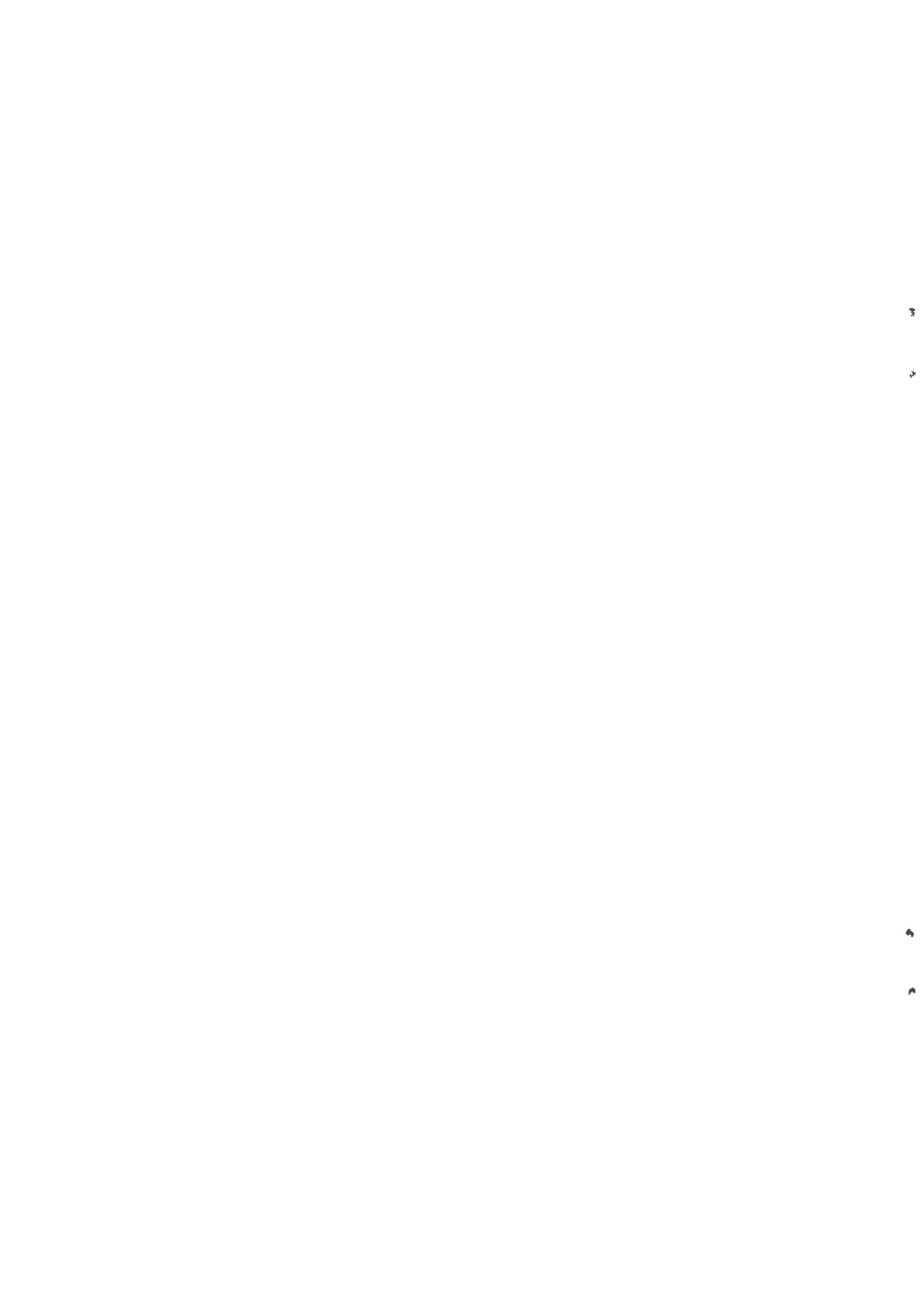
The kinetic energies of the photoelectrons leaving the sample are so low that, in practice, only those from the top 1-5 nm of the surface escape and arrive at the spectrometer, making it an extremely surface sensitive technique. Most elements (except hydrogen and helium) have practical detection limits 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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