

INSTITUTE OF
**PARTICLE SCIENCE
& ENGINEERING**



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Control of Concentrated Oxide Particulate Waste Streams

Presented by: Ms. Qanita Omokanye

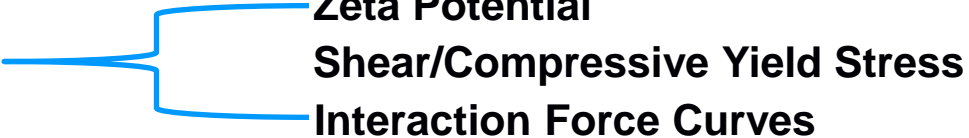
Supervisor: Prof. Simon Biggs

21st July, 2009.

EPSRC

Engineering and Physical Sciences
Research Council

KNOO
Keeping the Nuclear Option Open

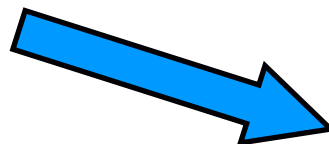
- **Research Motivation**
- **Aim**
- **Background & Theory**
- **Objectives**
- **Research Approach**
- **Sample Results** 
 - Zeta Potential**
 - Shear/Compressive Yield Stress**
 - Interaction Force Curves**
- **Conclusions**

Research Motivation

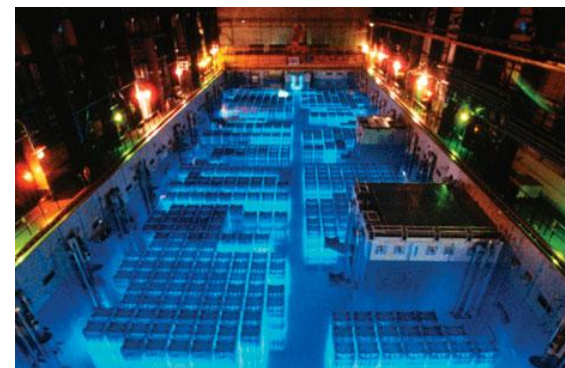


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Industrial interest for **Oxide Dispersions in Concentrated & Complex Electrolyte** conditions over a wide pH range.



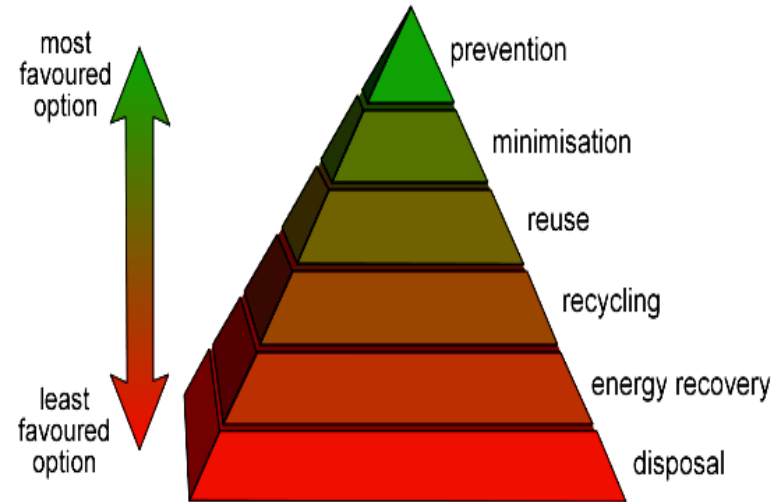
Contents of these ponds include; **Oxide dispersions**, Magnox sludge (corroded fuel cans), Organic waste (slimes), Miscellaneous junk, Insoluble carbonates etc.



In all cases, the waste particles dispersions will be subject to treatment & disposal. Therefore, an understanding of the behaviour of such sludge materials is crucial.

To develop fundamental knowledge that will facilitate new approaches to managing particulate wastes

in **concentrated electrolyte solutions.**

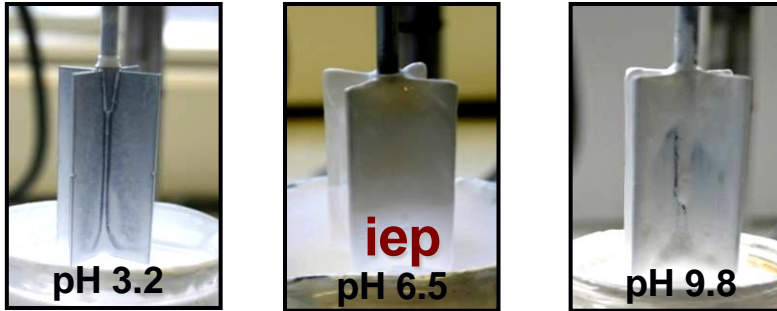


Background & Theory

Shear yield stress (τ_y) vs. Zeta (ζ) potential



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Theory Describing Particle - Particle Interactions: DLVO (Derjaguin, Landau, Verwey and Overbeek) 1940s'.

$$V_T = V_A + V_R \quad \text{Equation 1.}$$

$V_A > V_R$ – Unstable

$V_R > V_A$ – Stable

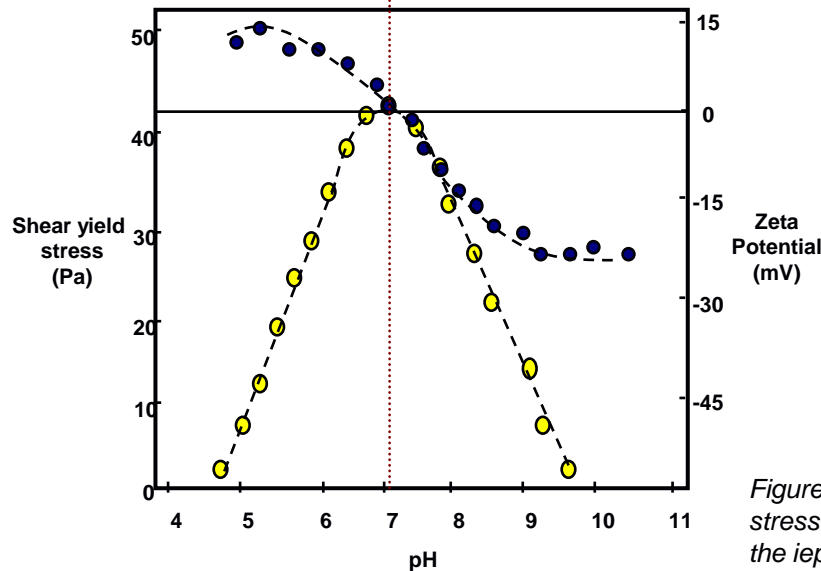


Figure 1. Total interaction energy curves.

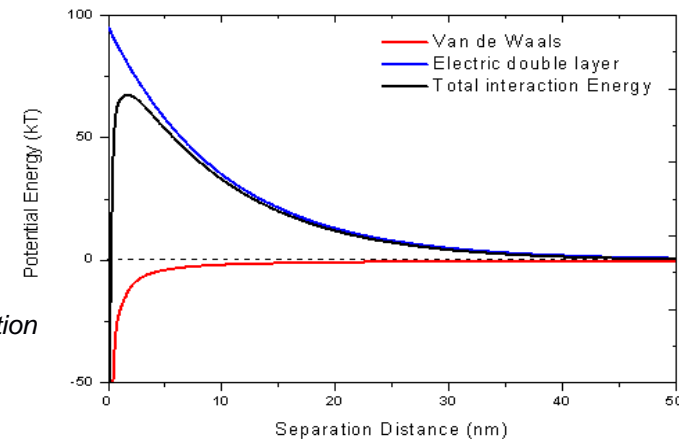


Figure 2. Schematic representation of zeta potential and shear yield stress versus pH – cationic and anionic surface adsorption showing the iep.

- **Determine the Zeta (ζ) potential of titania (TiO_2) in mixed salts e.g. $\text{NaCl} + \text{Na}_2\text{SO}_4$: ζ – related to ψ_0 , surface potential
 κ – Influenced by conc., ionic valency**
- **Determine the Shear/Compressive yield stress – relate to energy of contact of TiO_2 in mixed salts.**
- **How Ion adsorption and other interactions NOT given by DLVO affects the surface of a particle in solid-liquid interfaces; important in optimising re-suspension processes.**

Research Approach



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- Simulant “model” metal oxide (TiO_2) titania $\sim 1.1 \mu\text{m}$
- Measurements:
 - Zeta Potential (*Colloidal Dynamics ZetaProbe*)
 - Shear Yield Stress (*Brookfield Viscometer*)
 - Compressive Yield Stress (*Filtration Compressor*)
 - Force Curves (*NanoScope IV Digital Instruments Atomic Force Microscopy*)

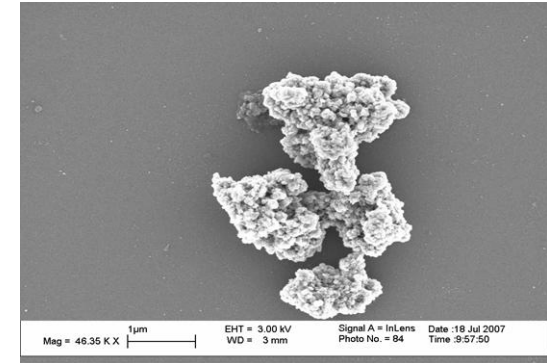


Figure 3. Scanning Electron Micrograph of TiO_2 in Milli. Q. Water

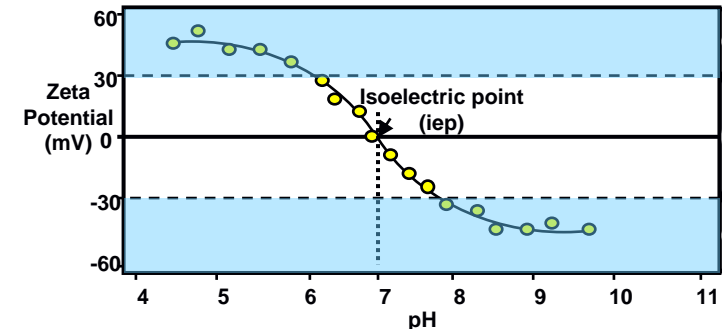


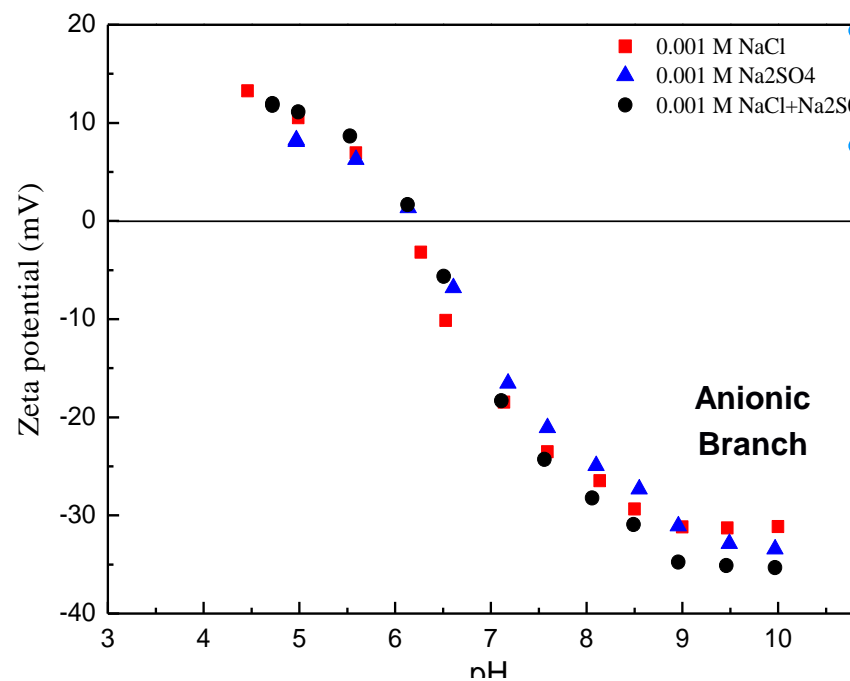
Figure 4. Schematic representation of zeta potential versus pH – cationic and anionic surface adsorption showing the iep

Zeta Potential

Single & Mixed Electrolytes



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• No iep for; ▲-Na₂SO₄, ●-NaCl+Na₂SO₄

• Na₂SO₄ dominates over NaCl

Figure 5. Zeta potential of 4 wt% titania as a function of both pH and electrolyte type 0.001 M single and mixed (1:1 cationic molar ratio).

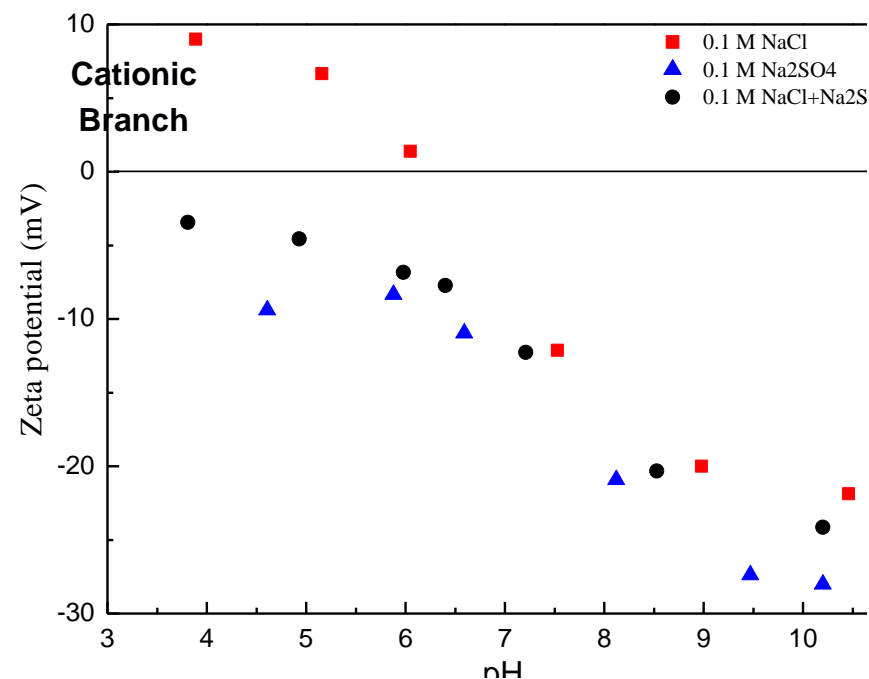


Figure 6. Zeta potential of 4 wt% titania as a function of both pH and electrolyte type 0.1 M single and mixed (1:1 cationic molar ratio).

Yield Stress

Shear & Compressive



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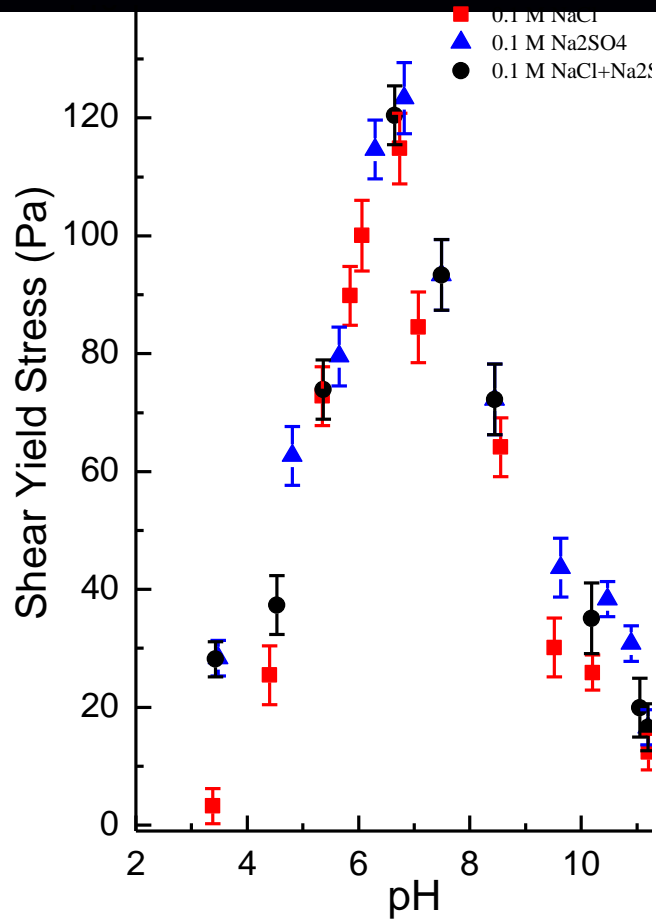


Figure 7. Avg. Shear yield stress of 47 wt% titania as a function of pH & 0.1 M electrolyte.

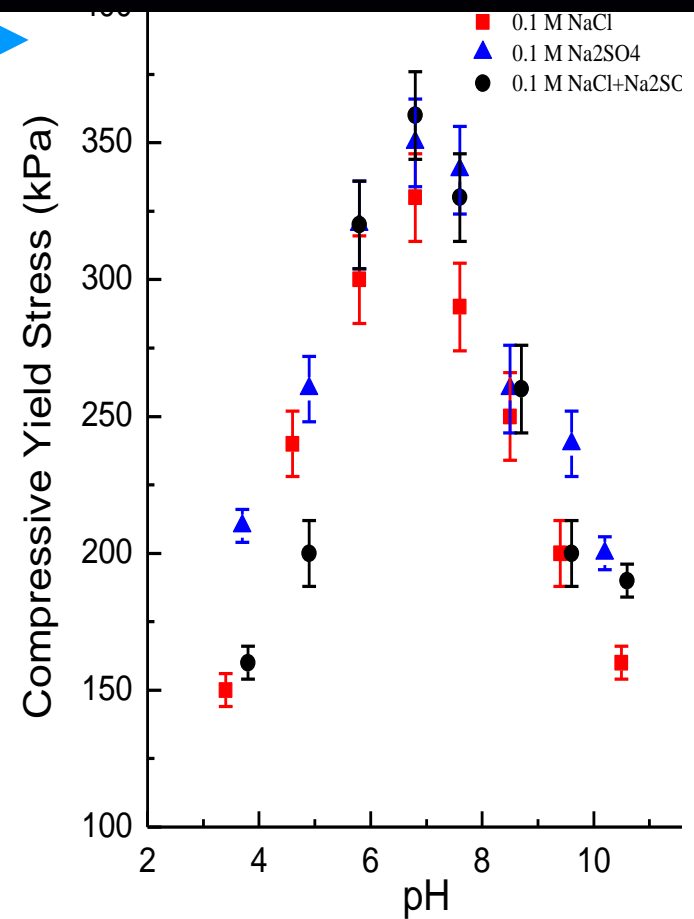


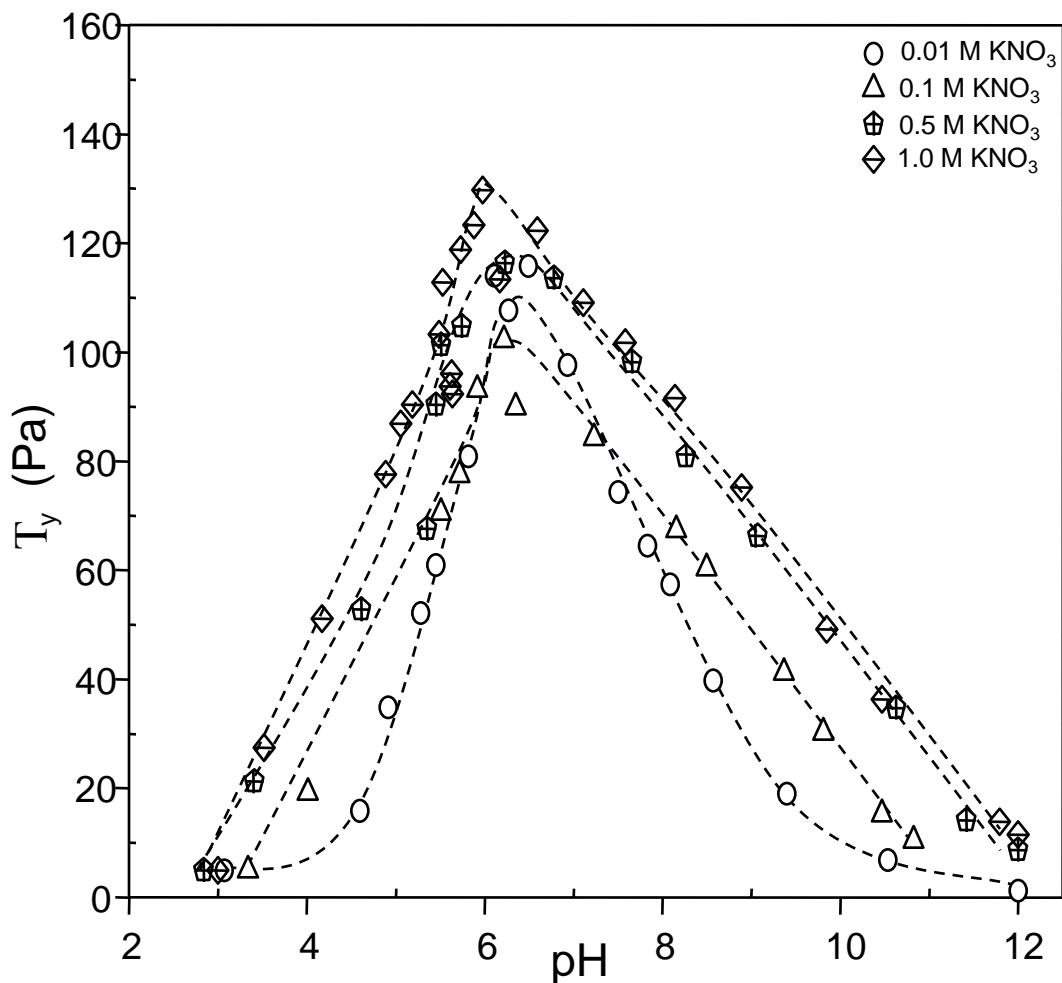
Figure 8. Avg. Compressive yield stress of 47 wt% titania as a function of pH & 0.1 M electrolyte.

Shear Yield Stress

Concentration Effect



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Peak increase with Concentration.

Figure 9. Effect of Shear yield stress as a function of both pH and concentration of KNO_3 (0.01 M ~ 1 mM – 1.0 M ~ 1000 mM) in 47 wt% titania (The dotted lines are drawn to guide the eye)

Interaction Force

Single & Mixed Electrolytes



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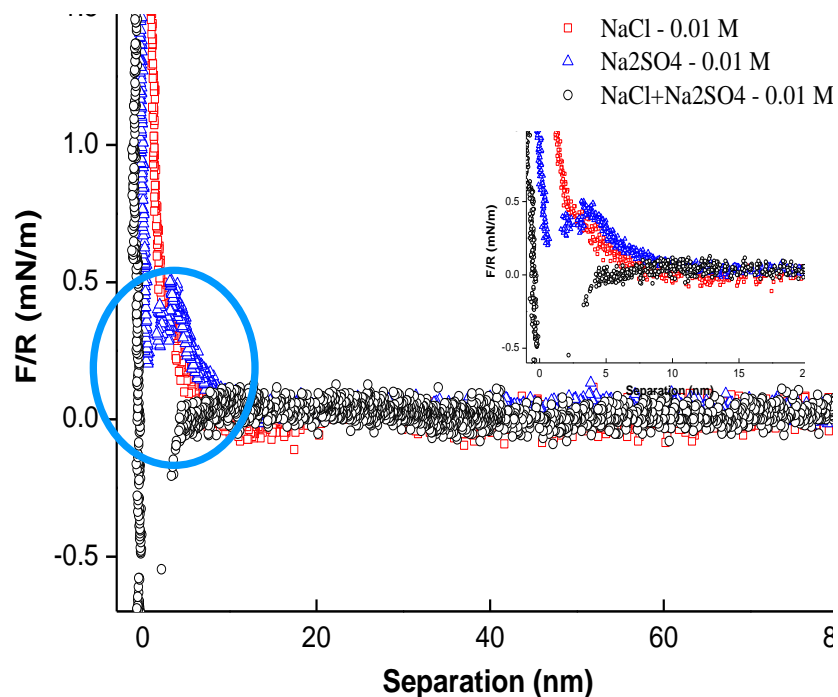


Figure 9. Approach force curve for rutile titania colloid probe tip with rutile titania surface in the presence of 0.01 M total concentration for salts as indicated in the figure.

Force-distance curves determine structure
firmness & resilience

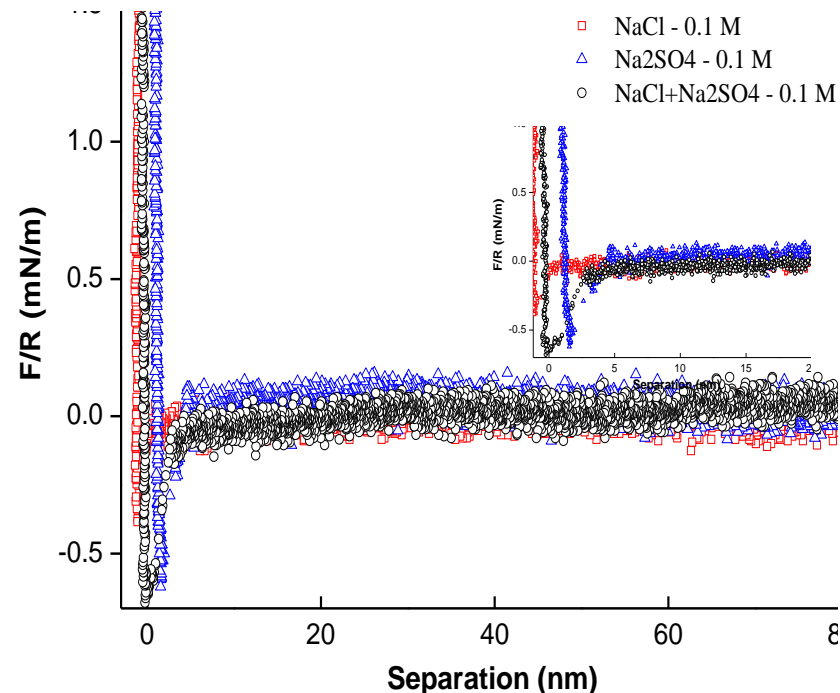


Figure 10. Approach force curve for rutile titania colloid probe tip with rutile titania surface in the presence of 0.1 M total concentration for salts as indicated in the figure.

Conclusions



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- Under certain pH, No repulsive forces and the particles have a net zero ζ potential, offering a route to **enhanced solid – liquid separation**.
- The **divalent ions** screens the electrical fields more effectively than monovalent ions.
- τ_y of suspensions are shown to fall in an approximately linear fashion with ζ^2 .
- Ion specific adsorption is important to understand to optimize. **Limitations to the DLVO.**

Acknowledgement



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My Supervisor - Prof. Simon Biggs



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Colloid and Polymer Engineering Group (CPEG)

Thank you all for your Attention!

Zeta Potential

Concentration Effect



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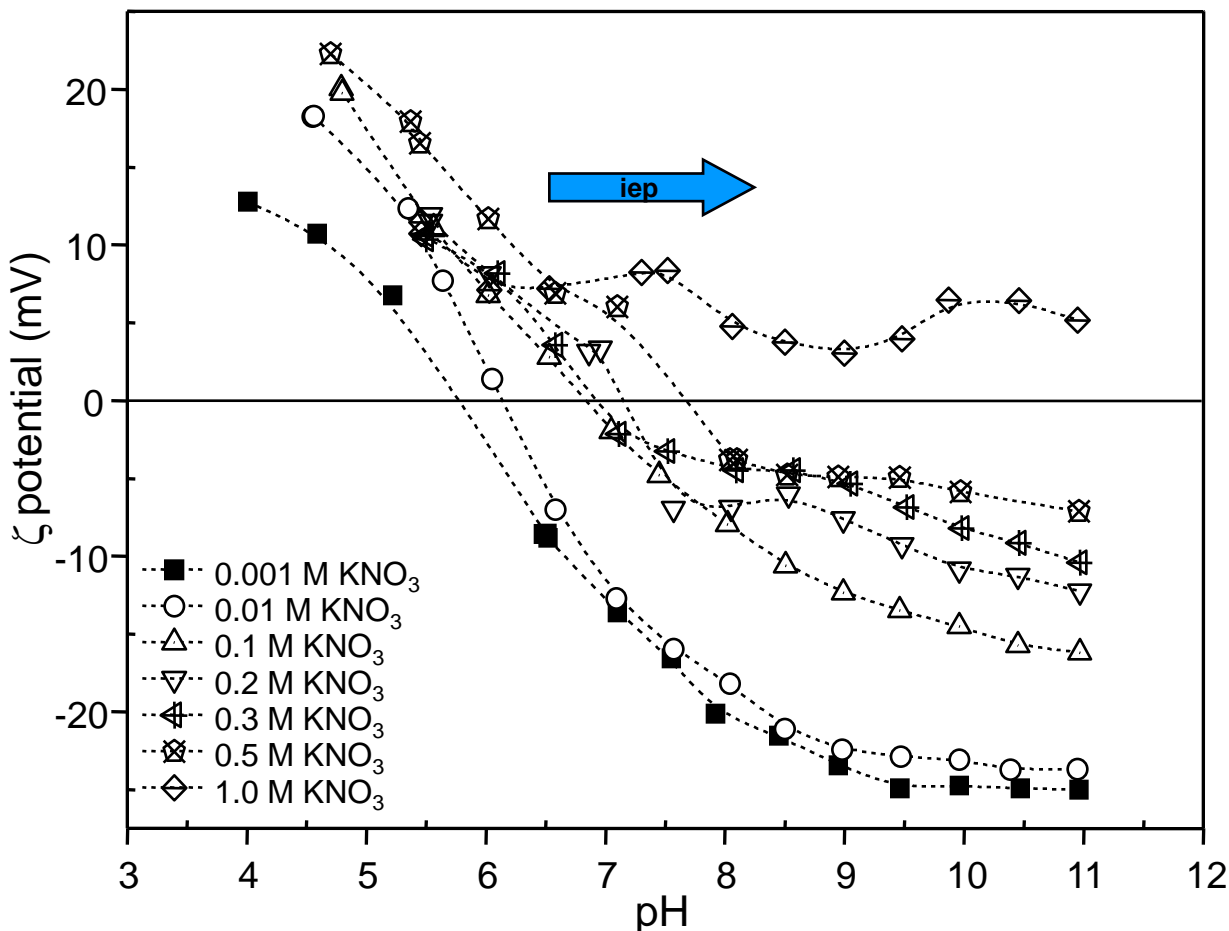


Figure 5. Zeta potential of 4 wt% titania particles as a function of both pH and aqueous solutions of KNO₃ (In agreement with Kosmulski and Rosenholm, 2004)

- Change / shift in iep out as conc. **INCREASES**

- **Specific adsorption** -Metal ion-surface interactions

Zeta Potential

Single Electrolytes



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- **Specific adsorption effects evident: above iep**

- Adsorption $K^+ > Na^+ > Li^+$
- Relates to Charge densities

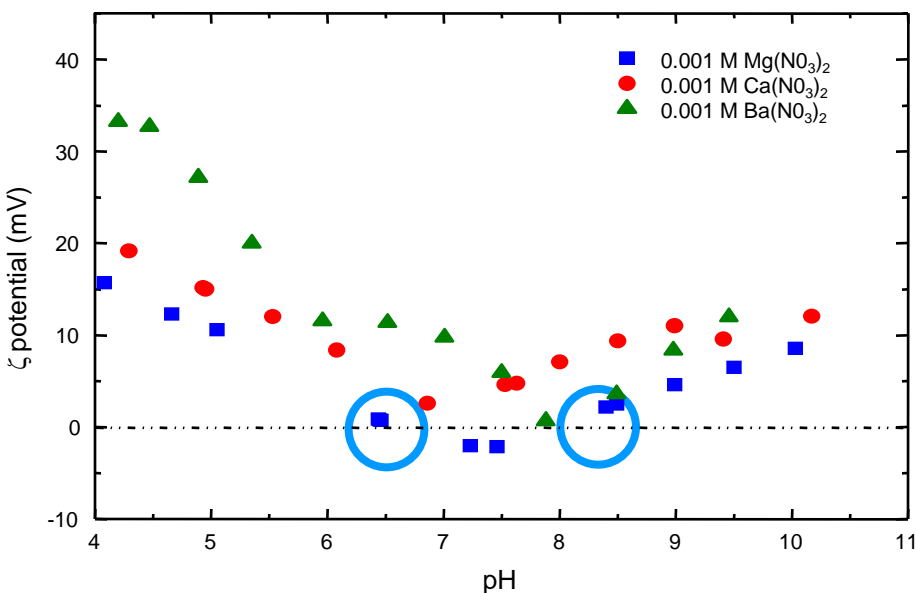


Figure 7. Zeta potential as a function of pH for titania at 4 wt% in **divalent electrolytes** (concentration 0.001 M ~ 1 mM)

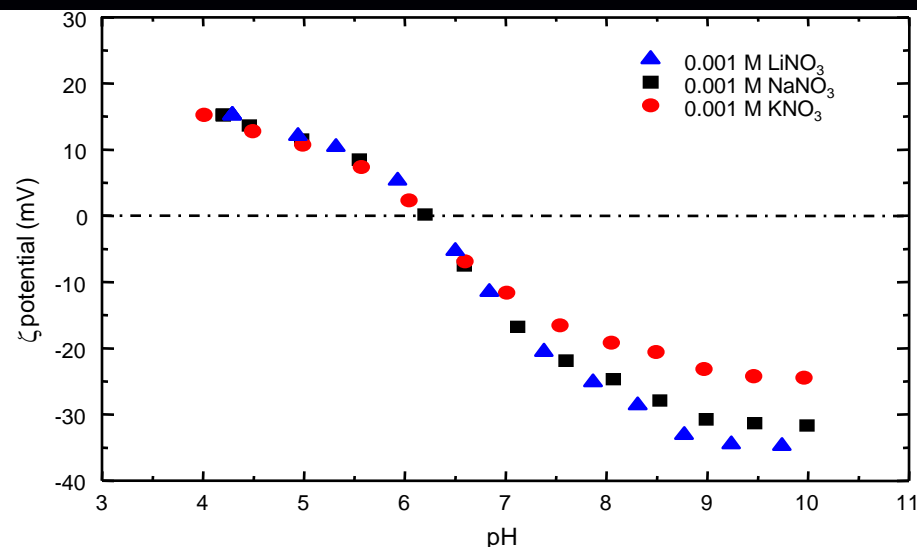


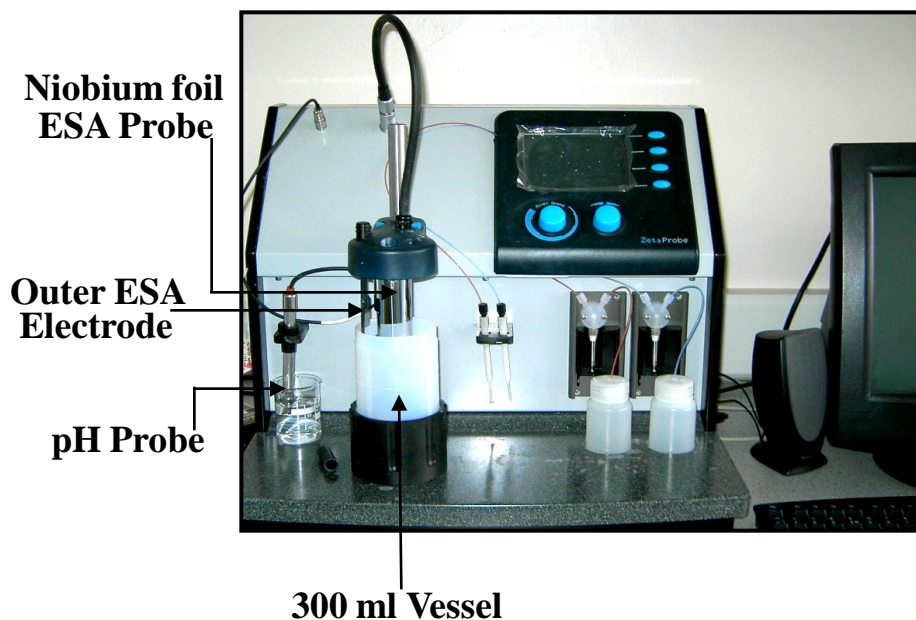
Figure 6. Zeta potential as a function of pH for titania at 4 wt% in **monovalent electrolytes** (concentration 0.001 M ~ 1 mM)

- **Specific adsorption effects evident: Mostly ANIONIC effect**

- Adsorption $Ba^{2+} > Ca^{2+} > Mg^{2+}$
- No point of zero charge
- Mg^{2+} and Ca^{2+} ions

Electroacoustic Effect

- **Signal generated** - application of an electric field across electrodes.
- **Signal results in a sound wave** - hits transducer.
- **Sound wave causes transducer to vibrate and generates voltage.**
- **This voltage allows us to determine the characteristics of the sound wave.**

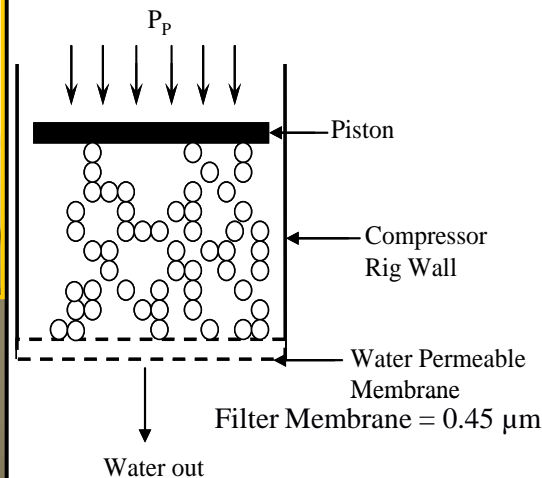
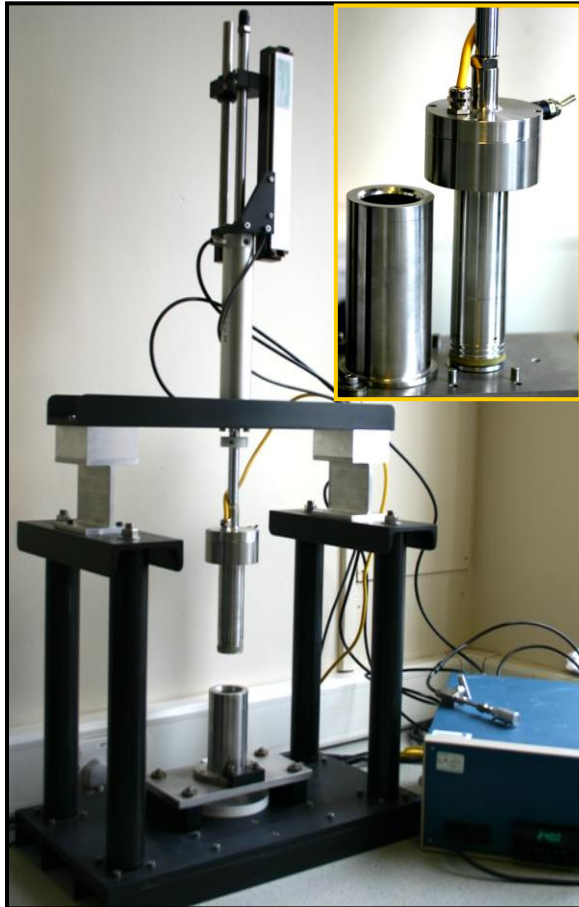


Compressor Rig

Concentration Effect



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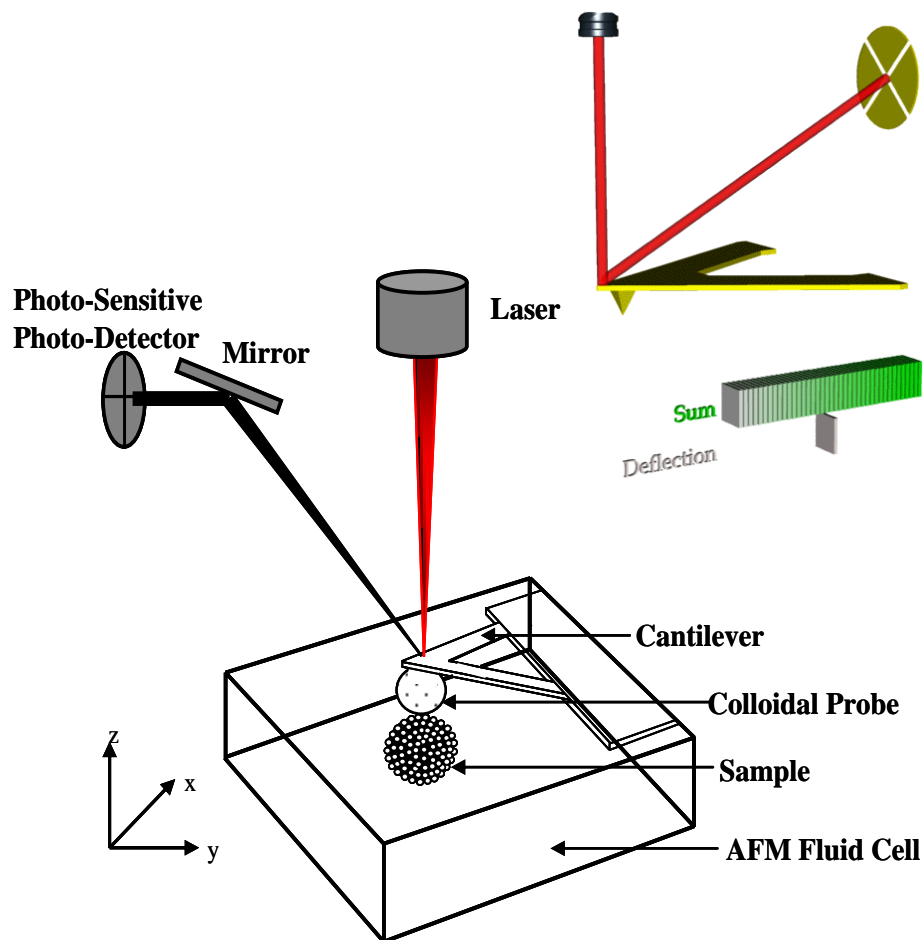
- Pressure is stepped up in each filtration run to determine compressive yield stress $P_y(\Phi)$.
- Employs a pneumatic cylinder to apply pressure to a sample contained in a compression cell.
- Pressure control system allows pressures ranging from 1 - 400 kPa.

AFM

NanoScope IV Digital Instruments



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- Interaction between cantilever and probe causes deflection.
- Detected via laser beam reflected off the back of cantilever via a mirror, onto a photo-detector.