

Computational Simulations of Hydrated Strontium Hydroxides

- 15th November 2016
- Olivia Lynes
- Supervisor: Andy Kerridge
- Industrial supervisor: Jonathan Austin

Talk Overview

- Aims of the project
- Previous work
- Aquo Complexes
- Hydroxide Complexes
 - Mono-hydroxide system
 - Di-hydroxide system
- Bulk Oxide- CeO_2

Aim of Project

Computational simulations of storage pond sludge disturbance

- Understanding the interactions of:
 - Strontium, caesium, uranium with particulates.
- *ab initio* molecular dynamics:
 - Large amount of data
 - Novel research method for this area.
- Studying:
 - Hydration Structure
 - Absorption effects/ surface interaction.
 - pH Effects.

Previous Work- Strontium Structures

- DFT gas phase and aqueous phase micro solvation.
- 3 heptahydrates
- 7 CN energetically stable.
- DFT calculations of Sr^{2+} hydroxides with a continuum solvent model.
- Proton transfer mechanism.
- Energy barrier 3.0 kJ/mol

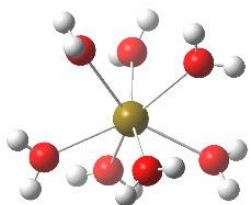


Figure 1. TPSS optimised Sr^{2+} heptahydrate.

A.Kerridge, N.Kaltsoyannis 2011
DOI:10.1002/chem.201003226

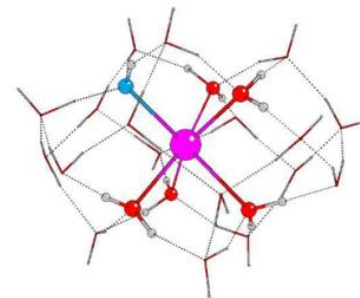


Figure 2. Sr^{2+} 5 Coordinated mono-hydroxide

E. Makkos, A.Kerridge, N.Kaltsoyannis 2015
DOI: 10.1039/C5DT01110H

Methodology

- CP2K (version 3)
 - Periodic planewave code
- *ab initio* molecular dynamic (AIMD) calculations.
 - Total simulation time of 20 picoseconds
 - 5 picosecond equilibration period
 - 0.5 fs timestep- $1/10^{\text{th}}$ smallest vibration
- Gaussian Augmented Planewave method (GAPW)
 - Goedecker-Teter-Hutter (GTH) pseudopotentials
- Nosè-Hoover thermostat

Aquo Complexes

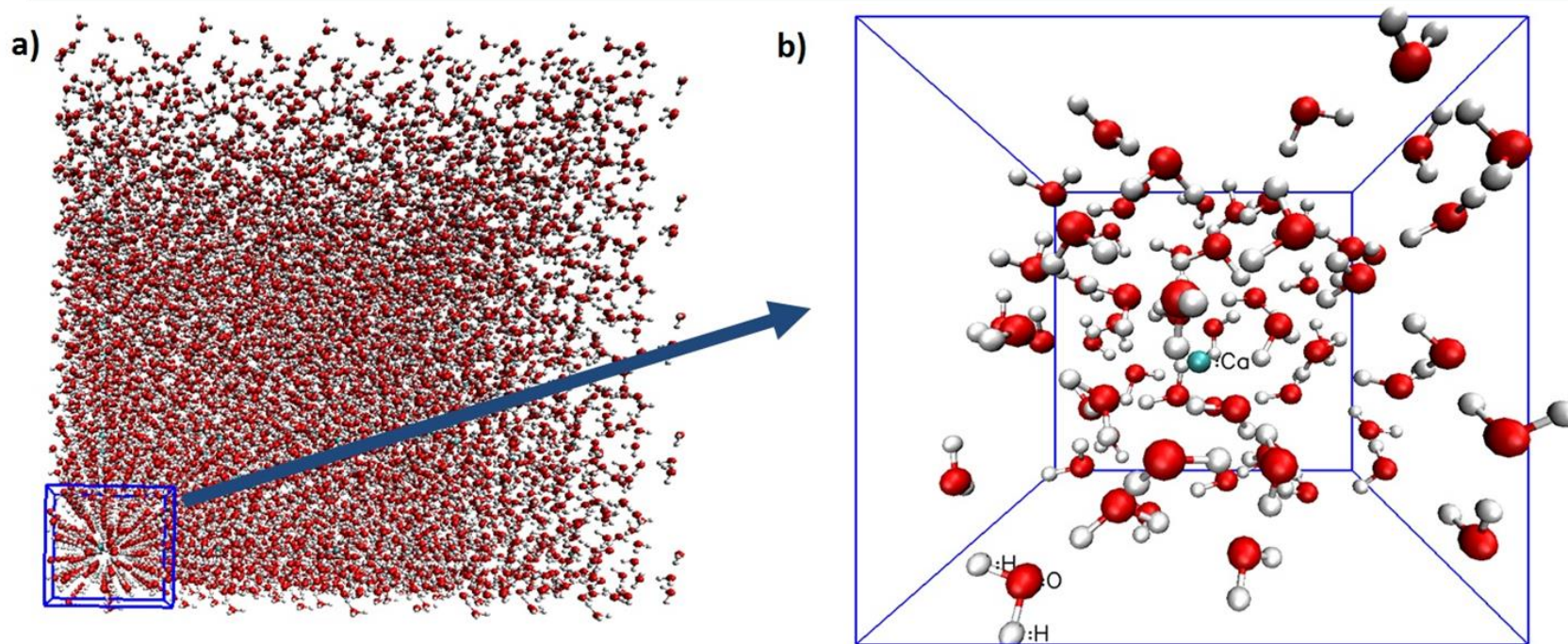


Figure 1: a) Periodic representation of the system. B) Single unit of 64 H₂O molecules with central metal ion (Ca²⁺)

- 11.99 Å box length
- N-H thermostat 400 K
- PBE/DZVP with dispersion

Aquo Complexes

Coordination Number (CN)

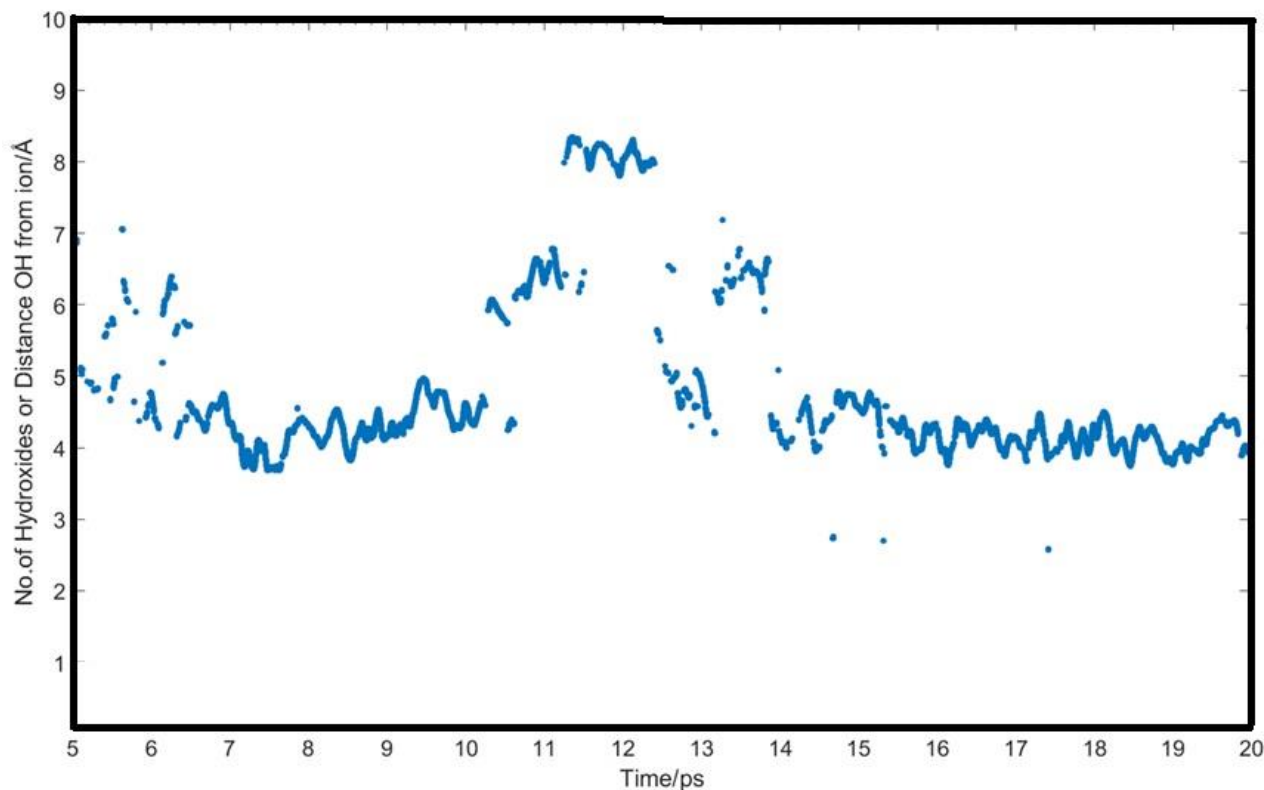
- Coordination of waters to the metal ion calculated at each step.
- Any CN which lasted less than 0.5 ps discounted as “not a true transition”

Bond Length

- Average bond length for the first solvation shell.

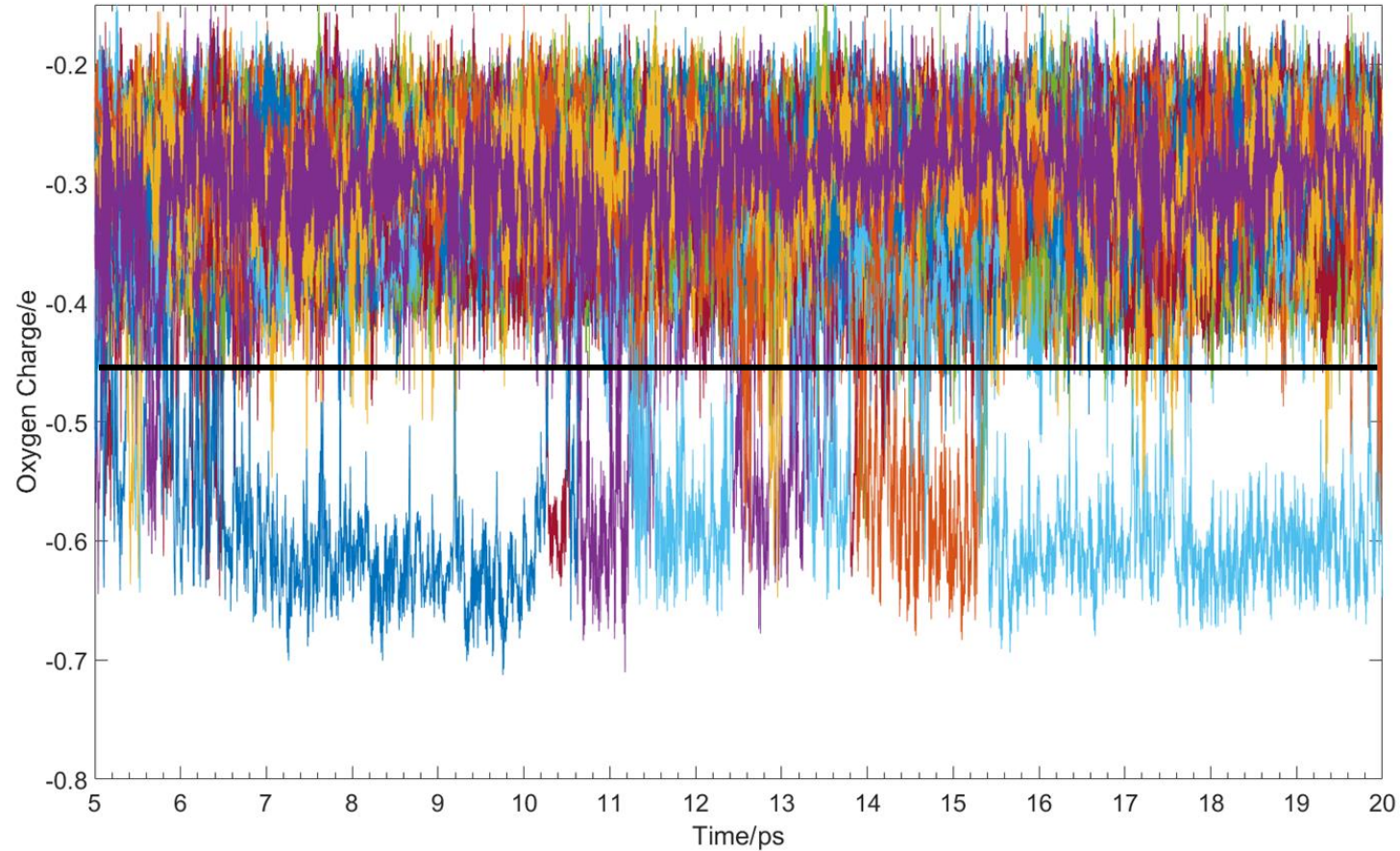
	CN Residence time %			Av. CN	Lit Value	Av. Bond Length/ Å	Lit Value/Å
	6	7	8				
Mg	100			6.00	6	2.20	2.07-2.12
Ca		43.88	56.12	7.56	6-8	2.52	2.35-2.68
Sr			100	8.00	7-9	2.66	2.52-2.69

Mono-hydroxide



- Single hydroxide
- Initially in 1st solvation shell
- Hydroxide at a distance > 4 Å from strontium ion
- Proton transfer or proton migration?

Mono-hydroxide



- Charge less than -0.57 cut off for hydroxide
- 19 separate oxygens considered a hydroxide across the timescale of reaction

Di-hydroxide Complexes-

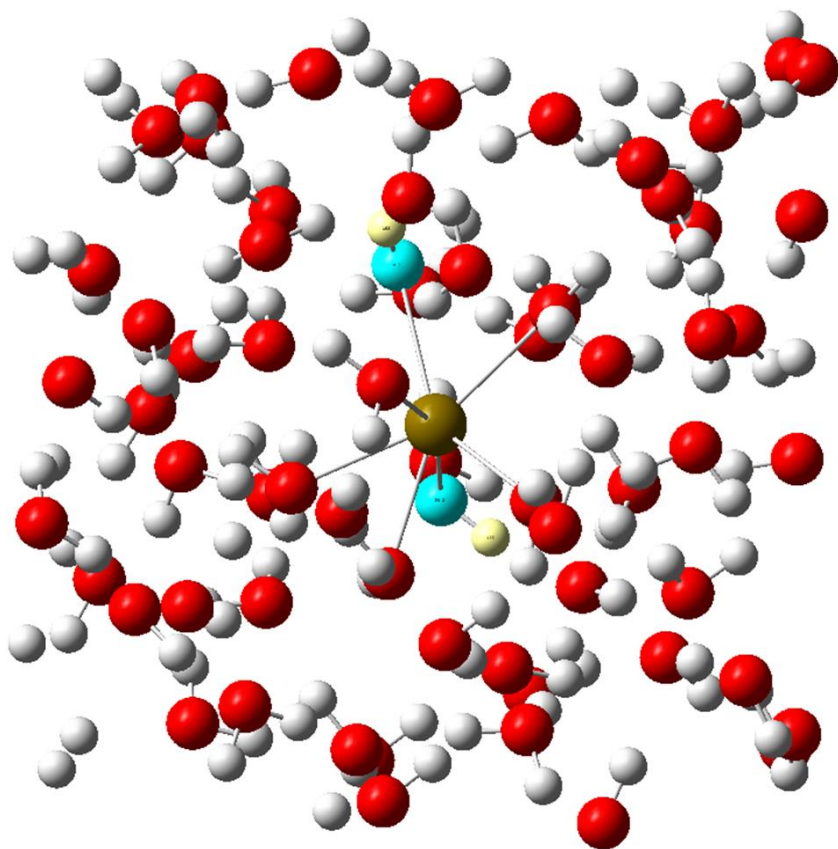


Figure 3: Visual representation of a strontium di-hydroxide. 2 OH⁻ ions (O- blue, H- Yellow) with a Sr²⁺ ion (brown) in the centre.

Initial Di-Hydroxide Systems

Both OH⁻ placed at a distance $> 3\text{\AA}$ away from the metal ion.

Both OH⁻ placed at a distance of $< 3\text{\AA}$ from the metal ion.

One OH⁻ placed at a distance of $< 3\text{\AA}$, one placed at a distance of $> 3\text{\AA}$.

Di-hydroxide Average Bond Lengths

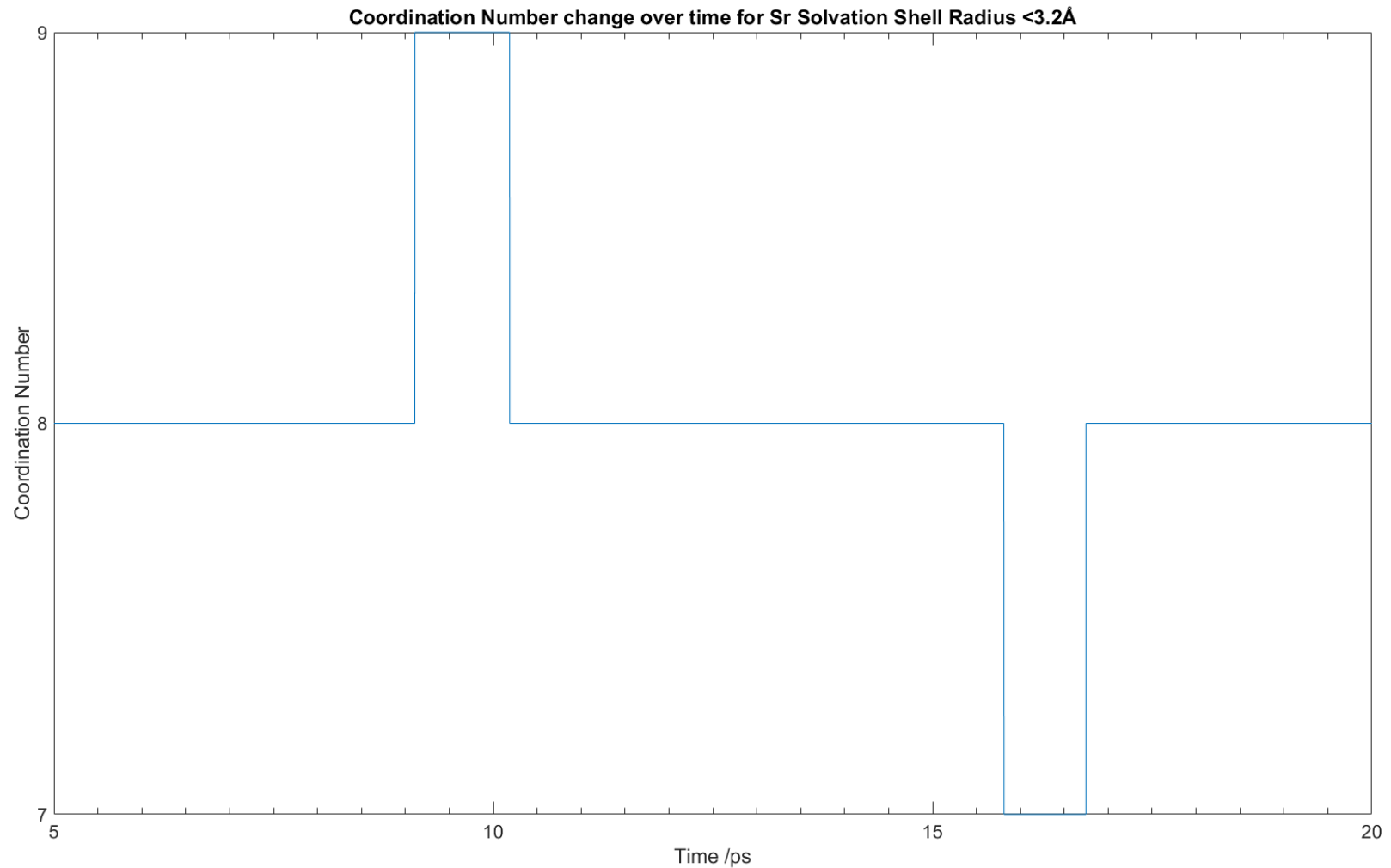
OH Each Solvation Shell		Average Bond Length / Å				Average per "system type" / Å
	Mg	2.16	2.17	2.13	2.20	2.17
	Ca	2.59	2.48	2.46	2.52	2.51
	Sr	2.73	2.63	2.63	2.70	2.67
OH first solvation shell						
	Mg	2.15	2.17	2.24	TBC	2.19
	Ca	2.67	2.42	2.53	2.44	2.51
	Sr	2.62	2.73	2.69	2.66	2.67
OH second solvation shell						
	Mg	2.23	2.15	2.14	2.09	2.15
	Ca	2.58	2.57	2.52	2.47	2.54
	Sr	2.67	2.74	2.64	2.66	2.68

Average Coordination Number

		Average CN				Average CN per "system"
OH in each Shell		1	2	3	4	
	Mg	6.00	6.00	6.00	6.00	6.00
	Ca	6.90	6.47	6.67	6.17	6.55
	Sr	8.01	6.86	7.65	7.48	7.50
Both in first solvation shell						
	Mg	6.00	6.00	5.70	TBC	5.90
	Ca	7.07	6.16	6.54	6.60	6.59
	Sr	7.00	7.45	7.64	7.50	7.40
Both in second solvation shell						
	Mg	5.72	6.00	6.00	5.97	5.92
	Ca	6.98	6.73	6.86	6.83	6.85
	Sr	8.04	7.61	7.46	7.78	7.72

Residence Time

One OH in each Shell



Residence Time OH in each Shell

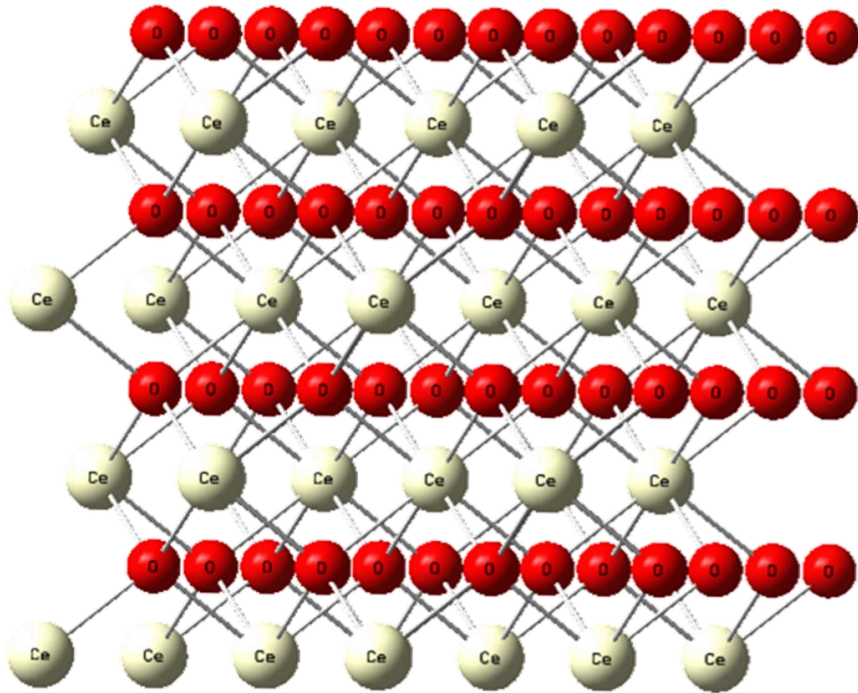
CN	% Residence Time				Average CN 2d.p
	6	7	8	9	
Mg	100.00	0.00	0.00	0.00	6.00
	100.00	0.00	0.00	0.00	6.00
	100.00	0.00	0.00	0.00	6.00
Ca	40.09	29.64	30.26	0.00	6.90
	61.74	29.48	8.78	0.00	6.47
	0.00	92.80	7.20	0.00	7.07
	33.41	35.56	31.03	0.00	6.98
Sr	0.00	6.22	86.59	7.19	8.01
	14.01	85.99	0.00	0.00	6.86
	0.00	36.26	63.74	0.00	7.64
	0.00	22.35	77.65	0.00	7.78

Hydroxide Summary

	Overall Average Bond Length / Å		
	Mg	Ca	Sr
Average Bond Length	2.17	2.52	2.67
Standard Deviation	0.04	0.07	0.04

	Overall Average CN		
	Mg	Ca	Sr
Coordination Number	5.94	6.66	7.54
Standard Deviation	0.11	0.28	0.33

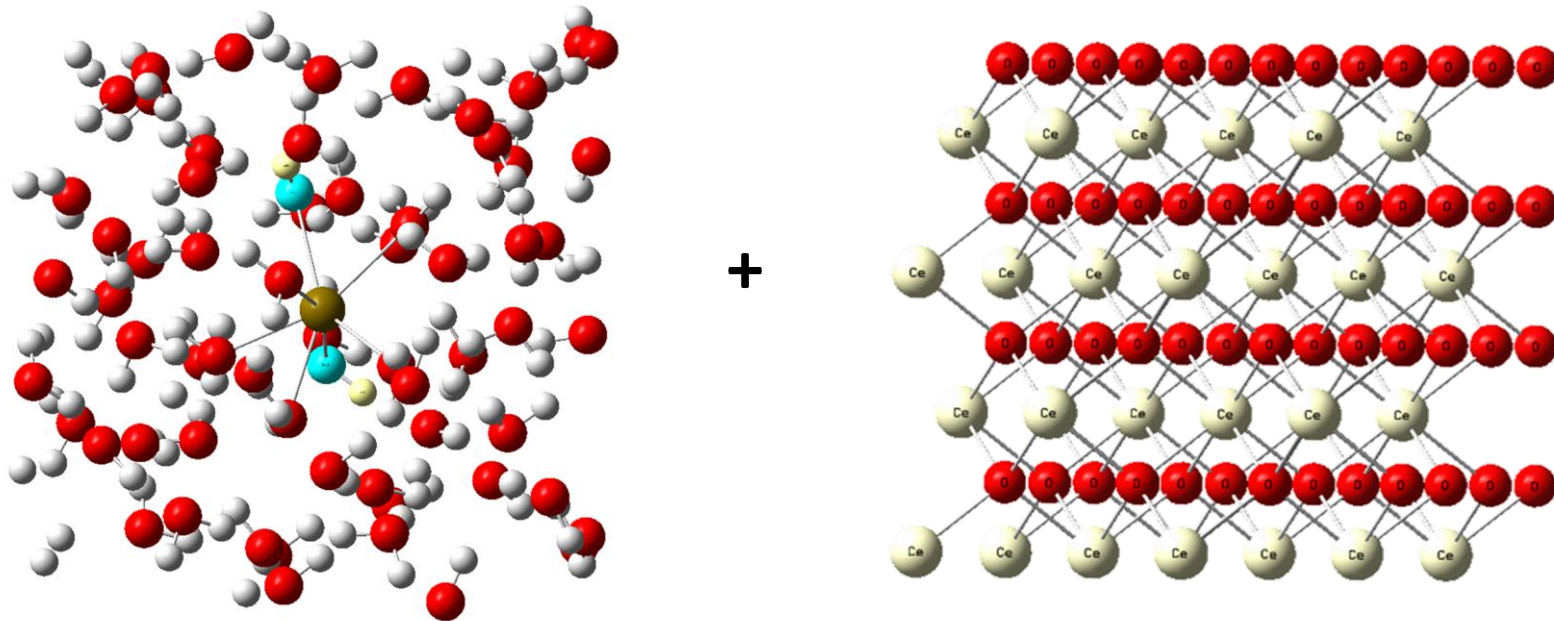
Oxide Surface- CeO_2



Why CeO_2 ?

- UO_2 is one of the main minerals of interest.
- Interested in the absorption of radionuclides onto the mineral surface.
- Uranium dioxide is not easily modelled and not currently available in CP2K.
- CeO_2 is a well known analogue for Uranium dioxide, as it closely resembles the mineral structure of fuel grade UO_2 .
- It is already used both computationally and experimentally with good results.

How it all links together



1. Water model
2. Hydroxide Model
3. Oxide surface
4. Absorption of radio nuclides onto the oxide surface.

Ongoing Work

- Hydroxide Systems AIMD
 - Total simulation time of 100 ps
 - One final set of calculations to run to reach 100 ps
- Water Systems AIMD
 - Currently only 20 ps simulation time.
 - Repetition to 100 ps simulation time.
- CeO₂ Oxide Surface DFT + U
 - Optimisation of surface structure
 - Introduction of hydration models to surface



DISTINCTIVE



Any Questions?

- Theme 3 Annual Meeting
- 15th November 2016
- Olivia Lynes
- o.lynes@lancaster.ac.uk

Principles and effects of dynamic shear in micro and ultrafiltration of chalk suspensions

Keith Schou
Loughborough University

Ultra- & micro- filtration

MF

0.1 - 3 bar
0.1 - 5 μm

UF

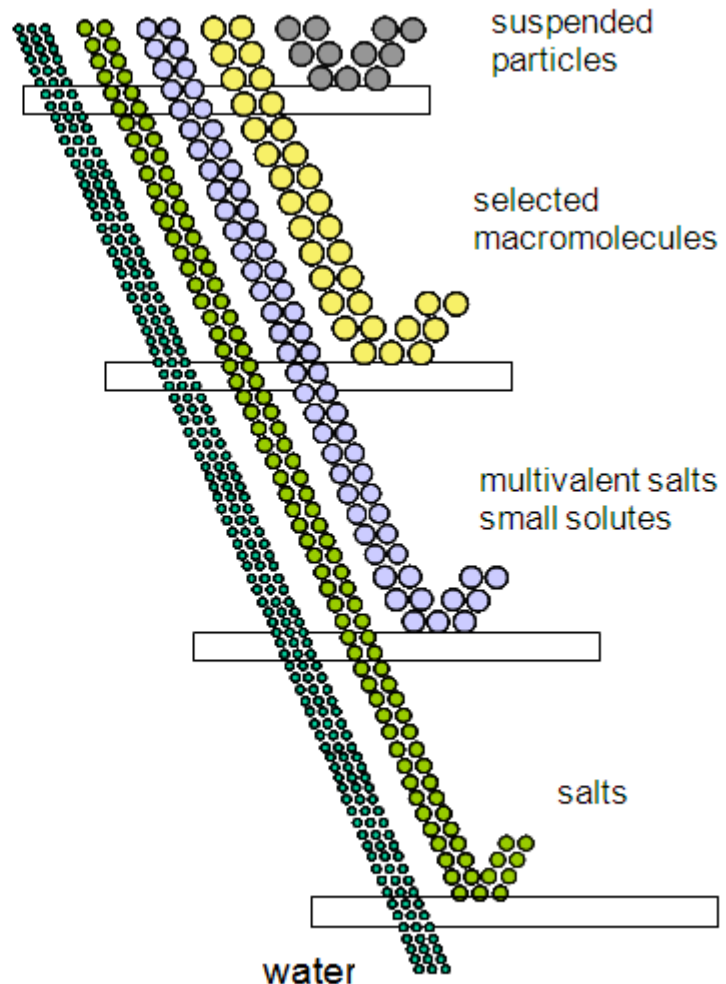
2 - 10 bar
20 nm - 0.1 μm

NF

5 - 30 bar
 $\gg$ 1 nm

RO

10 - 100 bar
0.1 - 1 nm (close)

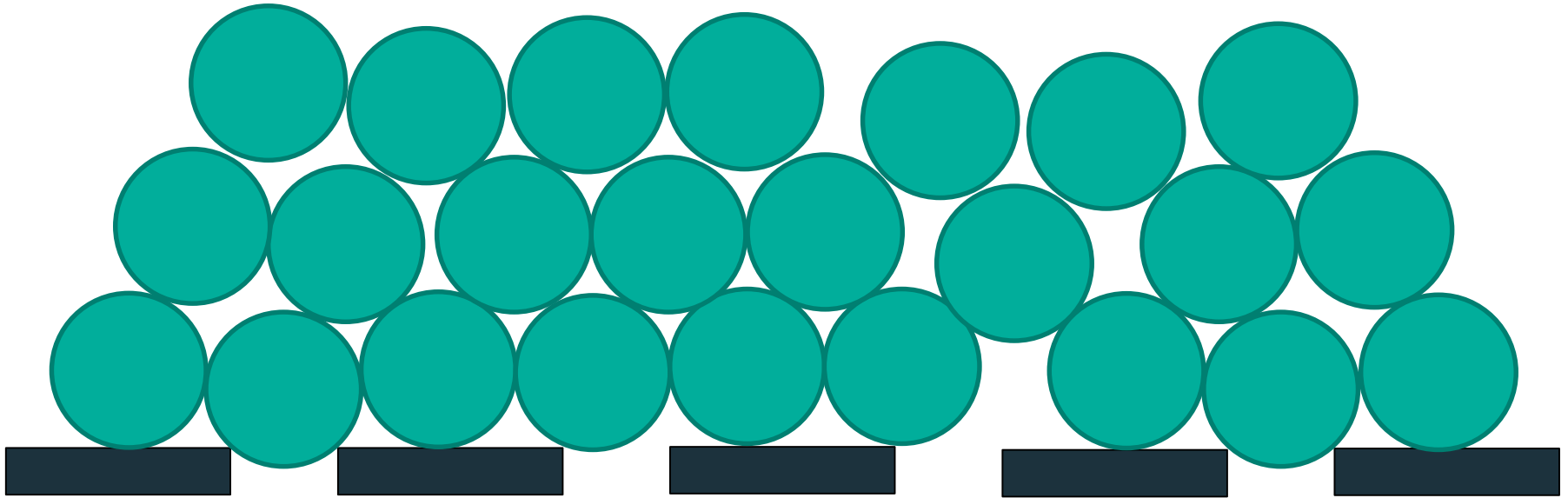


(Emis, 2015), retrieved from

http://emis.vito.be/sites/emis.vito.be/files/data_sheets/migrated/Microfiltration.png

accessed Apr/2015

Filter cake



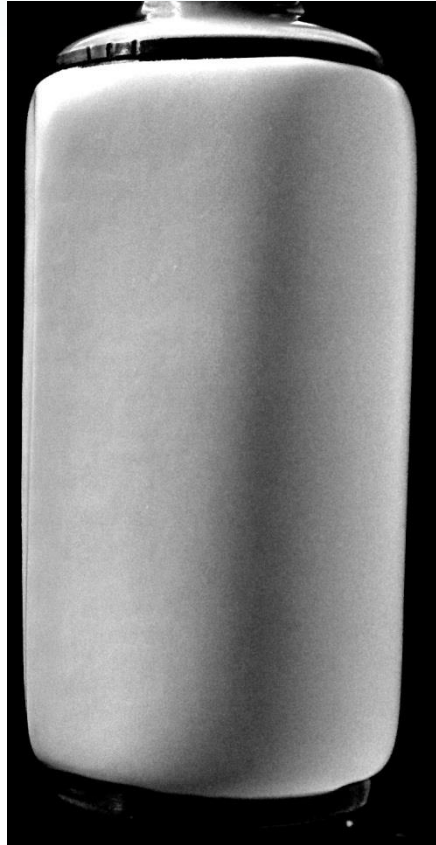
Where is this used

EARP

Enhanced Actinide
Removal Plant

Ferric floc
CMS

What is dynamic shear?



Azimuthal

Dynamic shear systems

Commercial forms

- Vsep, New Logic
- SpinTek
- DYNO, Bokela
- OPTIFILTER, Metso paper
- FMX, BKT
- SSDF, Novoflow
- Westfilia Separator

In development

- Helical membranes
- Magnetically induced membrane vibrations
- Linear with turbulence promoters

Enhanced shear systems

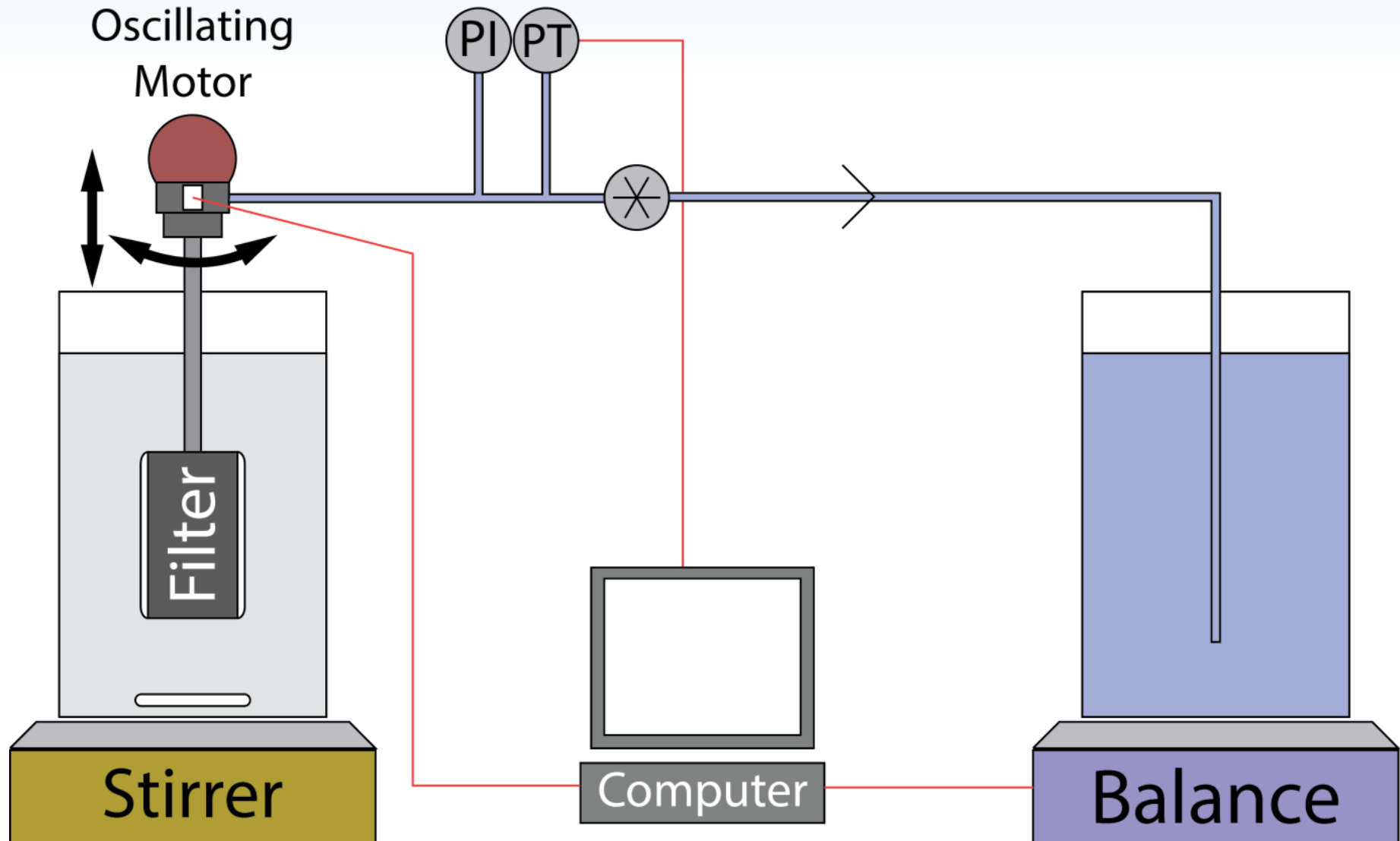
Shear rate, γ (s ⁻¹)	Oscillation type	Flux Enhancement (E)	Reference	System
350	Axial	9.1	(Gomaa, Rao & Taweel 2011)	Linear with TP
2540	Axial	2.7	(Low et al. 2005)	Linear
4600	Axial	2.7	(Li et al. 2013)	Linear
18800	Axial	7.6	(Krantz et al. 1997)	Linear
33600	Azimuthal	2.1	(Kennedy et al. 1974)	VSep
38300	Azimuthal	4.1	(Low et al. 2004)	VSep
39000	Azimuthal	17.1	(Akoum et al. 2002)	VSep

Table 1 The enhancement factor as a function of average shear rate (Zamani et al. 2015)

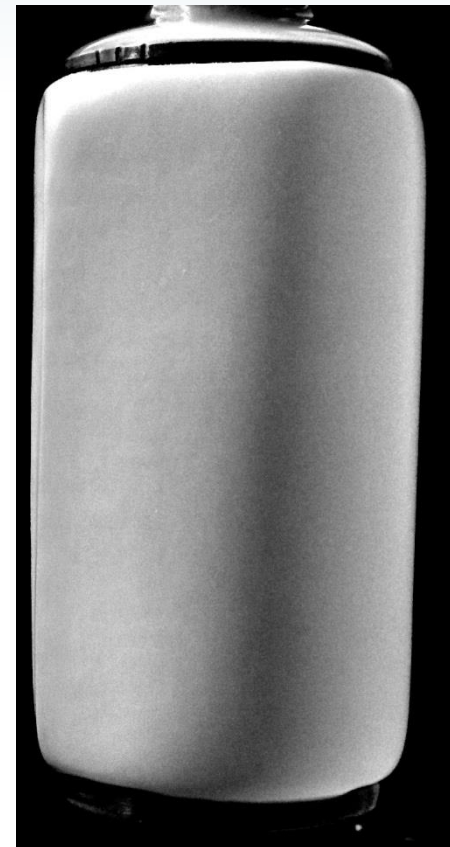
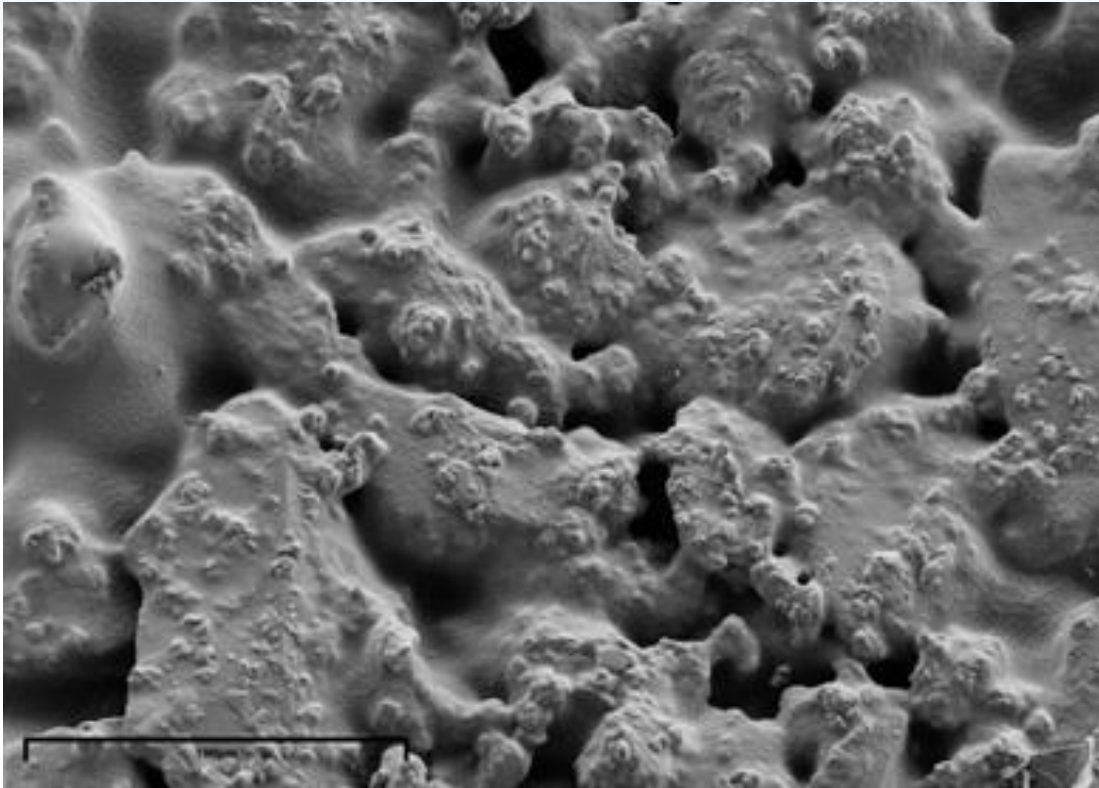
Key research questions

- Does the application of the shear effect this enhancement?
- Does the filter used effect this enhancement?
- What are the underlying principles at work in this enhancement?
- Can these be characterised and modelled?
- Does the localised shear damage the suspension, creating a more difficult to filter suspension?
- How can this be applied in EARP

Experimental system

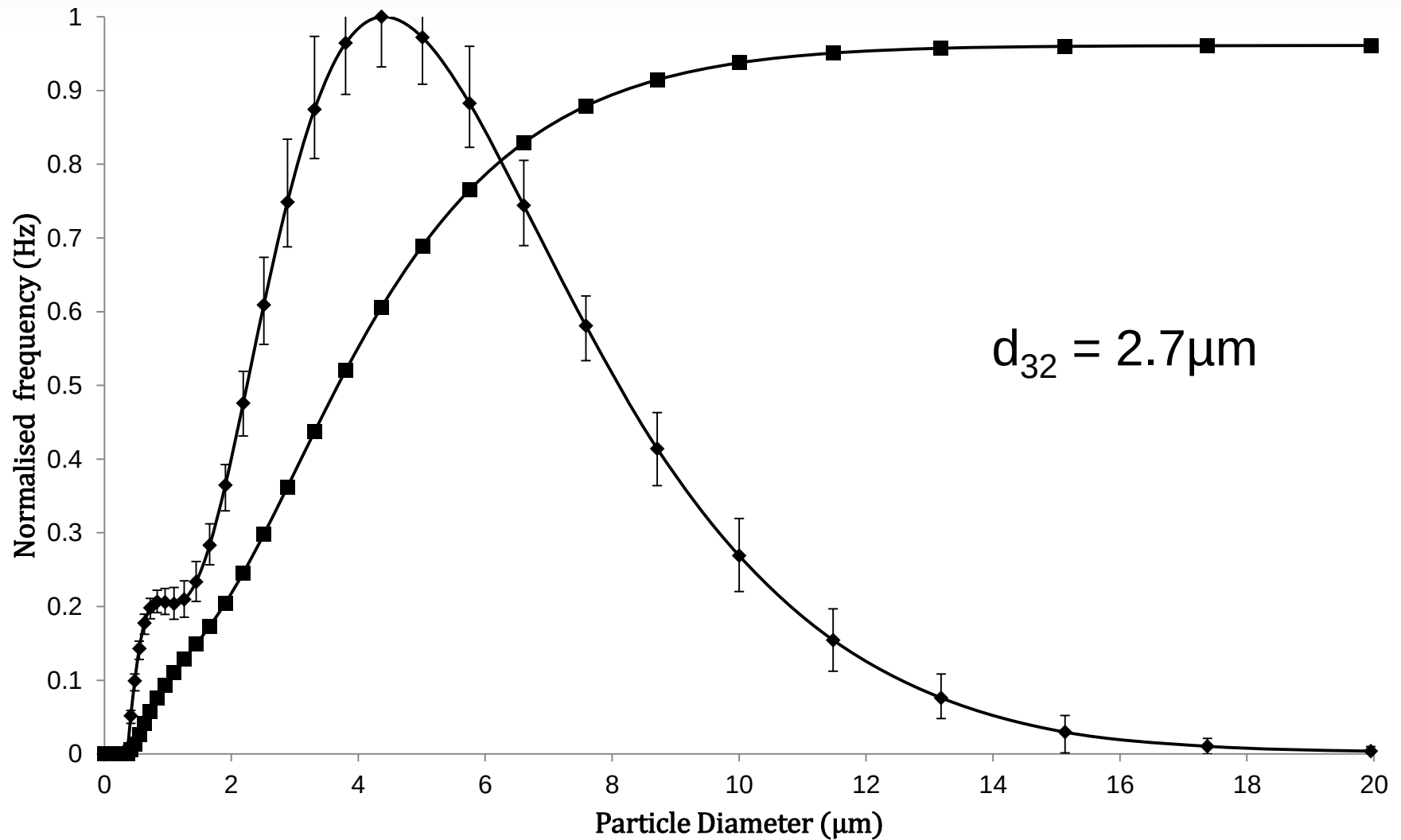


Filter / cake

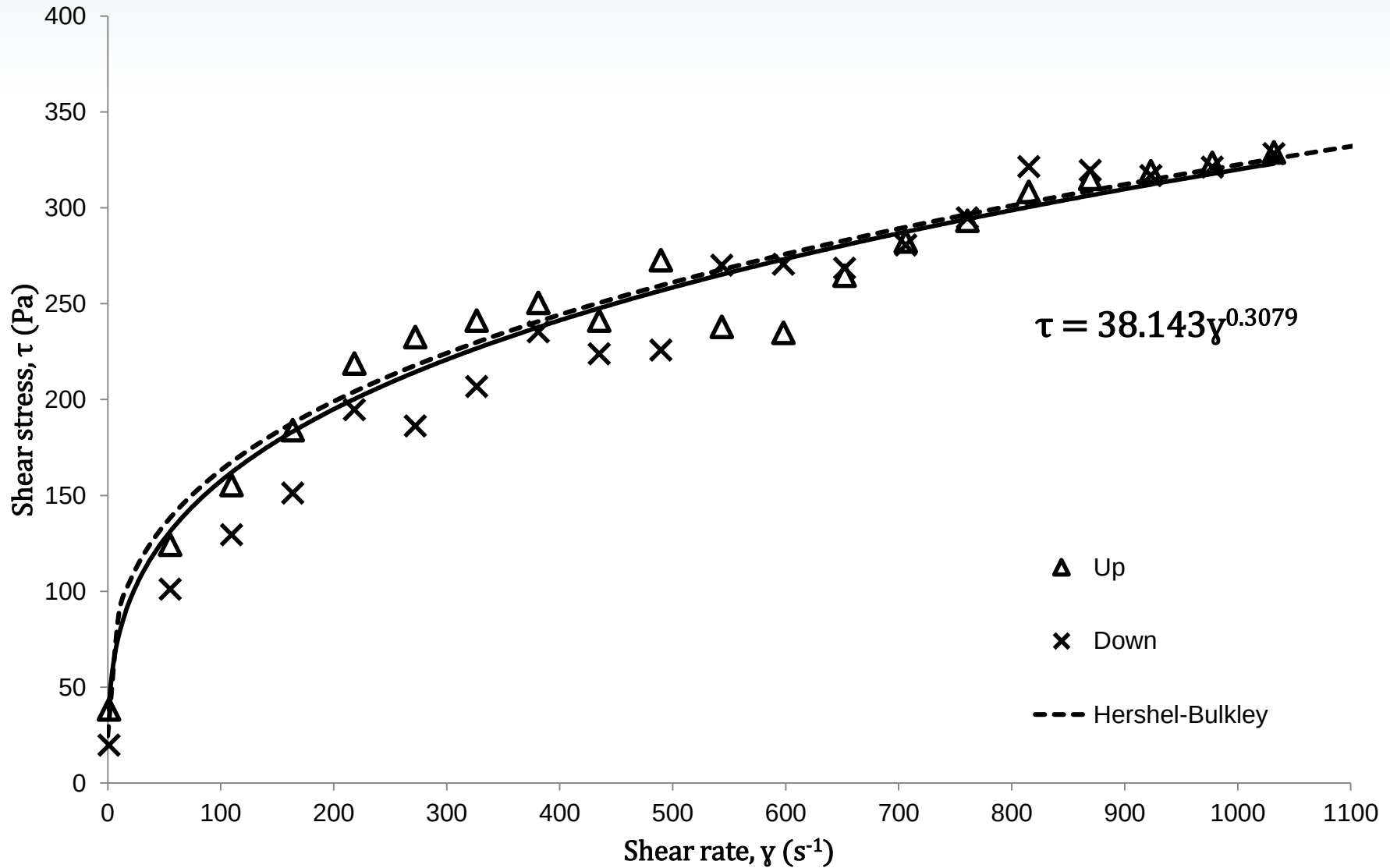


Azimuthal

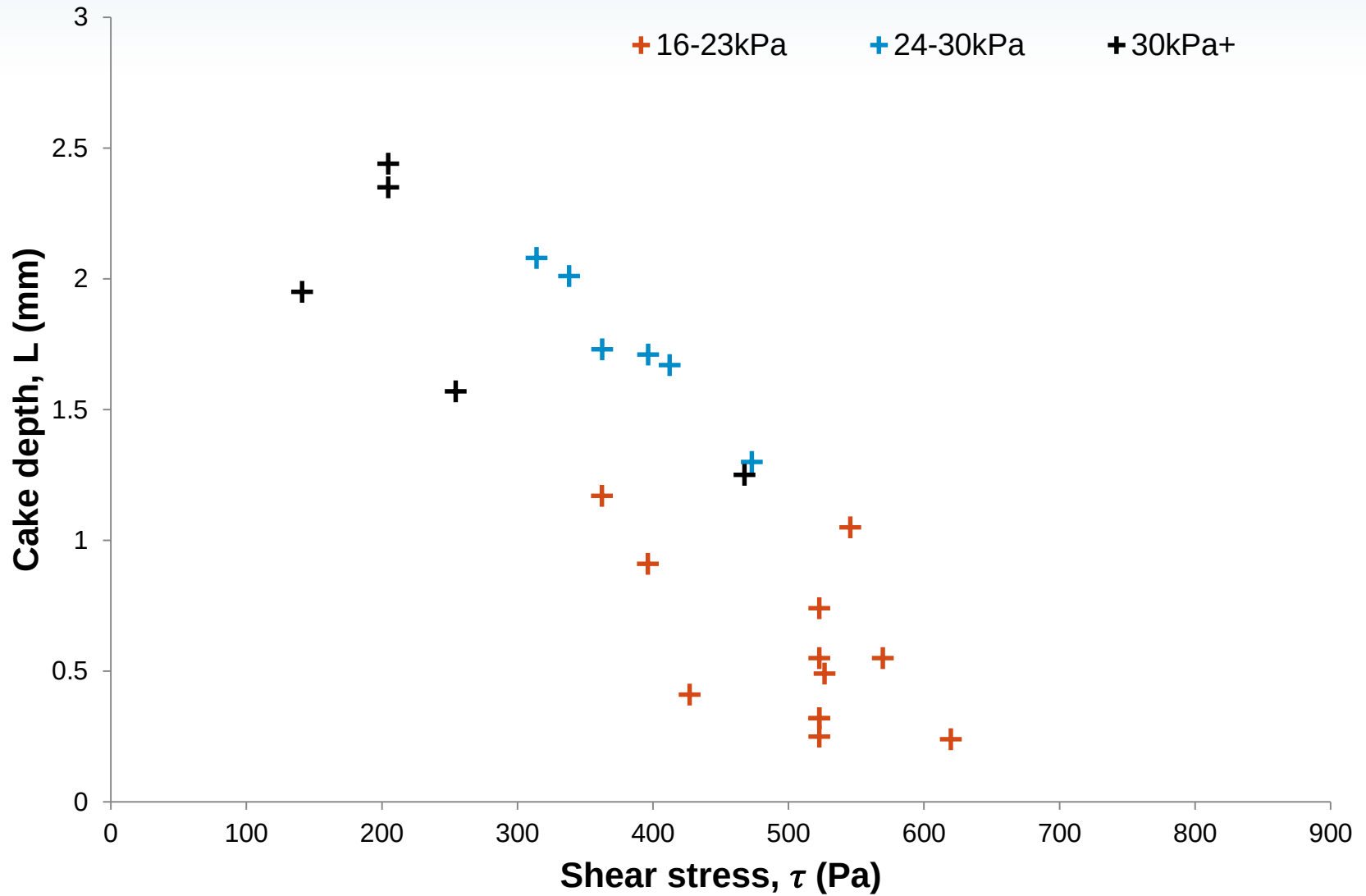
Suspension PSD



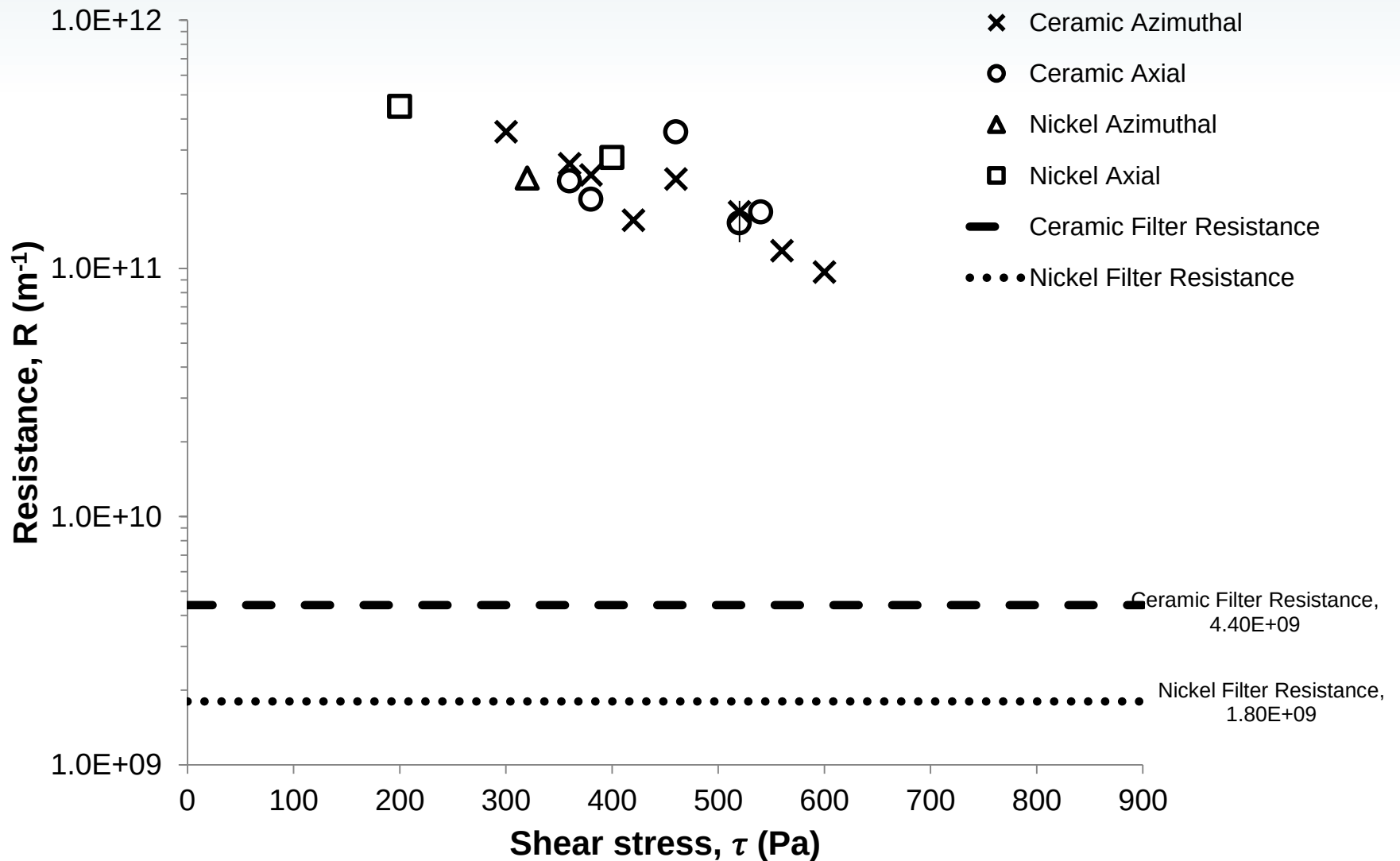
Cake Rheology



Cake Depth



Resistance



Nomenclature

Variable	Symbol	Units
Area	A	m ²
Amplitude	a	m
Cake concentration	C	
Particle diameter – Sauter mean	d ₃₂	m
Frequency	f	s ⁻¹
Flux	J	m ³ .m ⁻² .s ⁻¹
Cake permeability	k	m ²
Cake thickness	L	m
Trans membrane pressure	ΔP	Pa
Volumetric flowrate	Q	m ³ .s ⁻¹
Resistance	R	m ⁻¹
Pore diameter	r _p	m
Shear stress	τ	Pa
Viscosity (permeate)	μ _p	Pa.s
Shear Rate	γ	s ⁻¹

Maths of the system

$$\gamma_{Max} = 2\pi f a \sqrt{\frac{2\pi f}{2\eta}} \sqrt{2}$$

- Wave equation

$$\tau = K\gamma^m$$

- Power law (from rheology)

$$J = \textit{Constant}_1 \cdot \gamma^{\textit{Constant}_2}$$

- From literature

(Jaffrin et al. 2004; Akoum et al. 2002; Petala & Zouboulis 2006; S. P. Beier et al. 2006; Genkin et al. 2006; Goma, Rao & Al-Taweel 2011; Postlethwaite et al. 2004; Akoum et al. 2005)

$$J = K_1 d_{32} \tau + K_0$$

- Friction equation

$$R = \frac{A}{\mu_p} \frac{\Delta P}{Q}$$

$$k = \frac{L}{R}$$

$$Q = \frac{k \Delta P A}{L \mu_p}$$

- Darcy's equation
(different forms)

Maths of the system

$$J = K_1 d_{32} \tau = \frac{k \Delta P}{L \mu_p}$$

Happel and Brenner model

$$k = \frac{\left(2 - 3C^{\frac{1}{3}} + 3C^{\frac{5}{3}} - 2C^2\right) d_{32}^2}{\left(3 + 2C^{\frac{5}{3}}\right) 12C}$$

$$K_1 = \frac{k \Delta P}{L \mu_p d_{32} \tau}$$

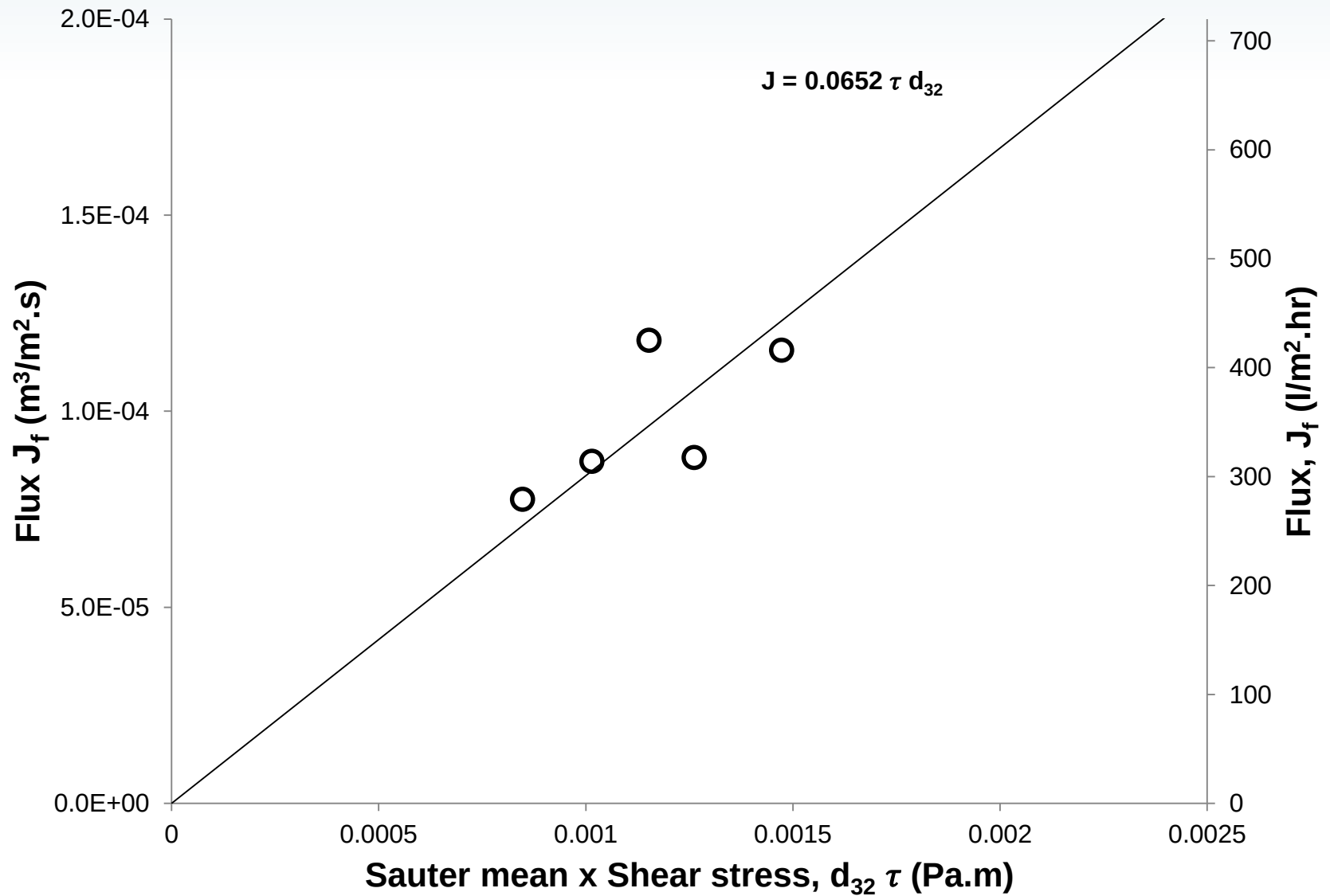
$$L = \frac{d_{32}^2 (1 - C) \Delta P}{90 \tau C^2 \sqrt{d_{32}^2 - 4rp^2}}$$

Maths of the system

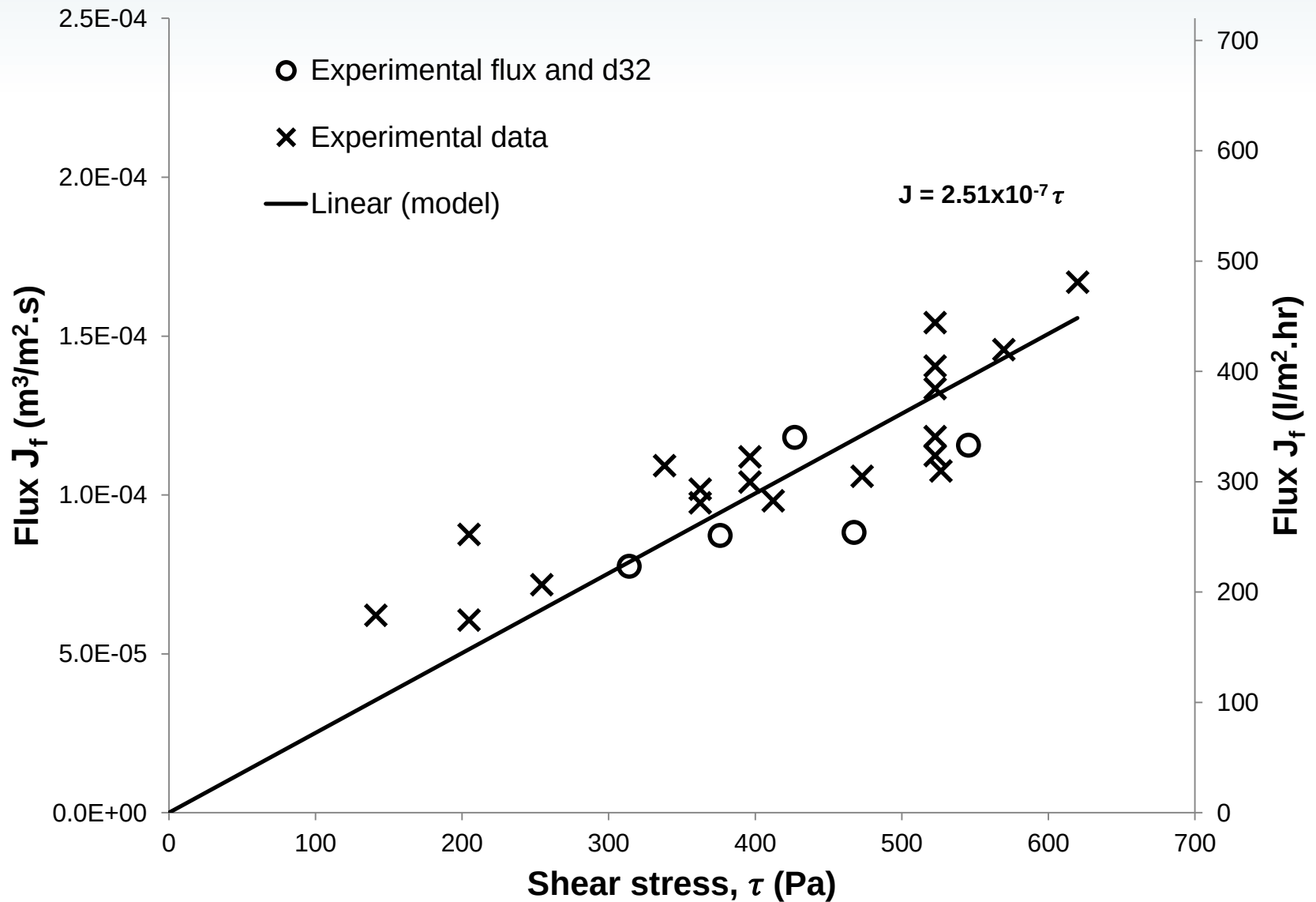
$$J = \frac{15\tau C(2 - 3C^{\frac{1}{3}} + 3C^{\frac{5}{3}} - 2C^2) \sqrt{d_{32}^2 - 2 \left(\frac{\sqrt{9d_{32}^4 + 16d_{32}^2}}{4} - d_{32} \right)^2}}{2(3 + 2C^{\frac{5}{3}})(1 - C)\mu_p}$$

Great!.... But it doesn't work

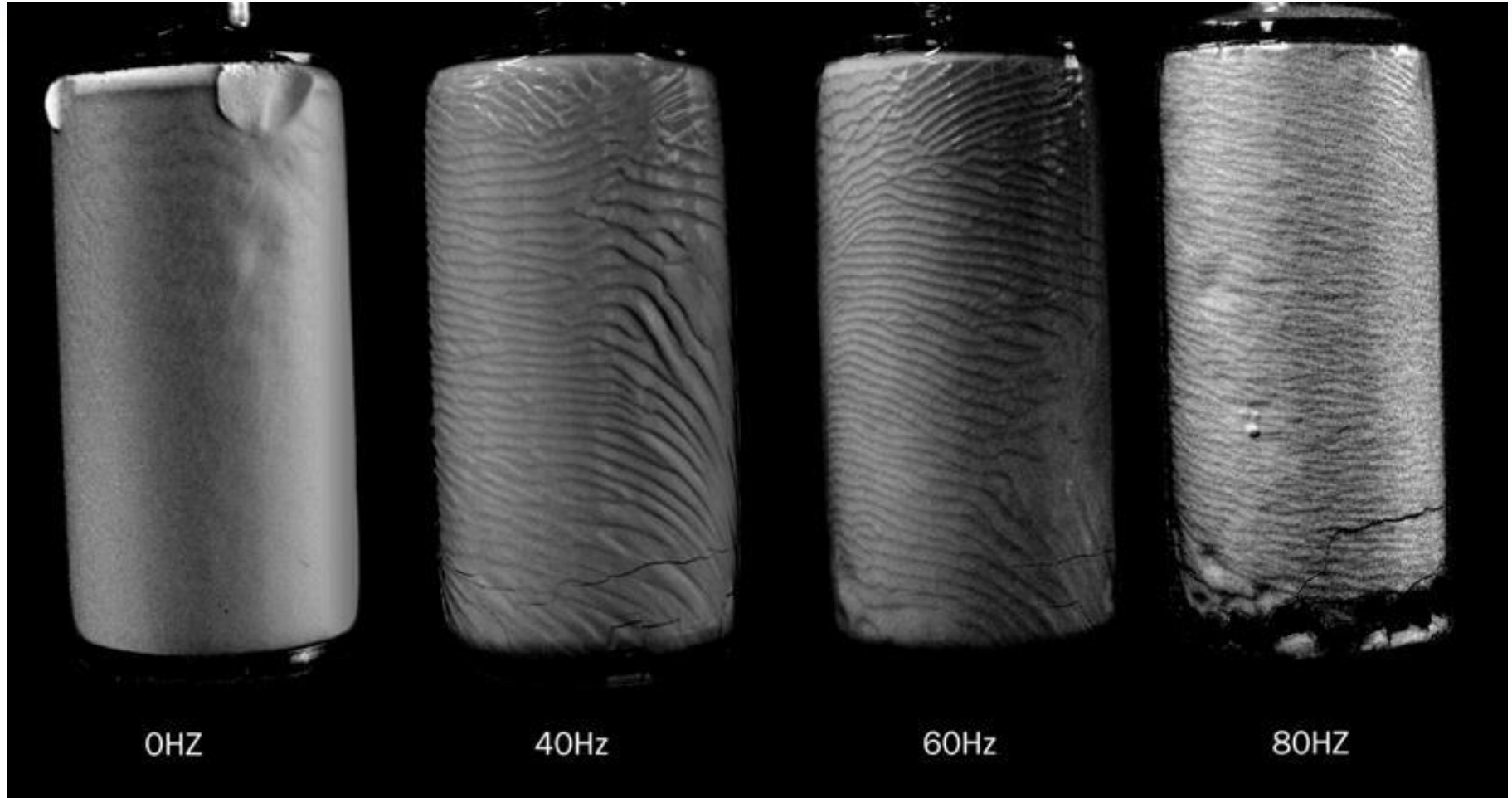
Friction model



Shear vs flux



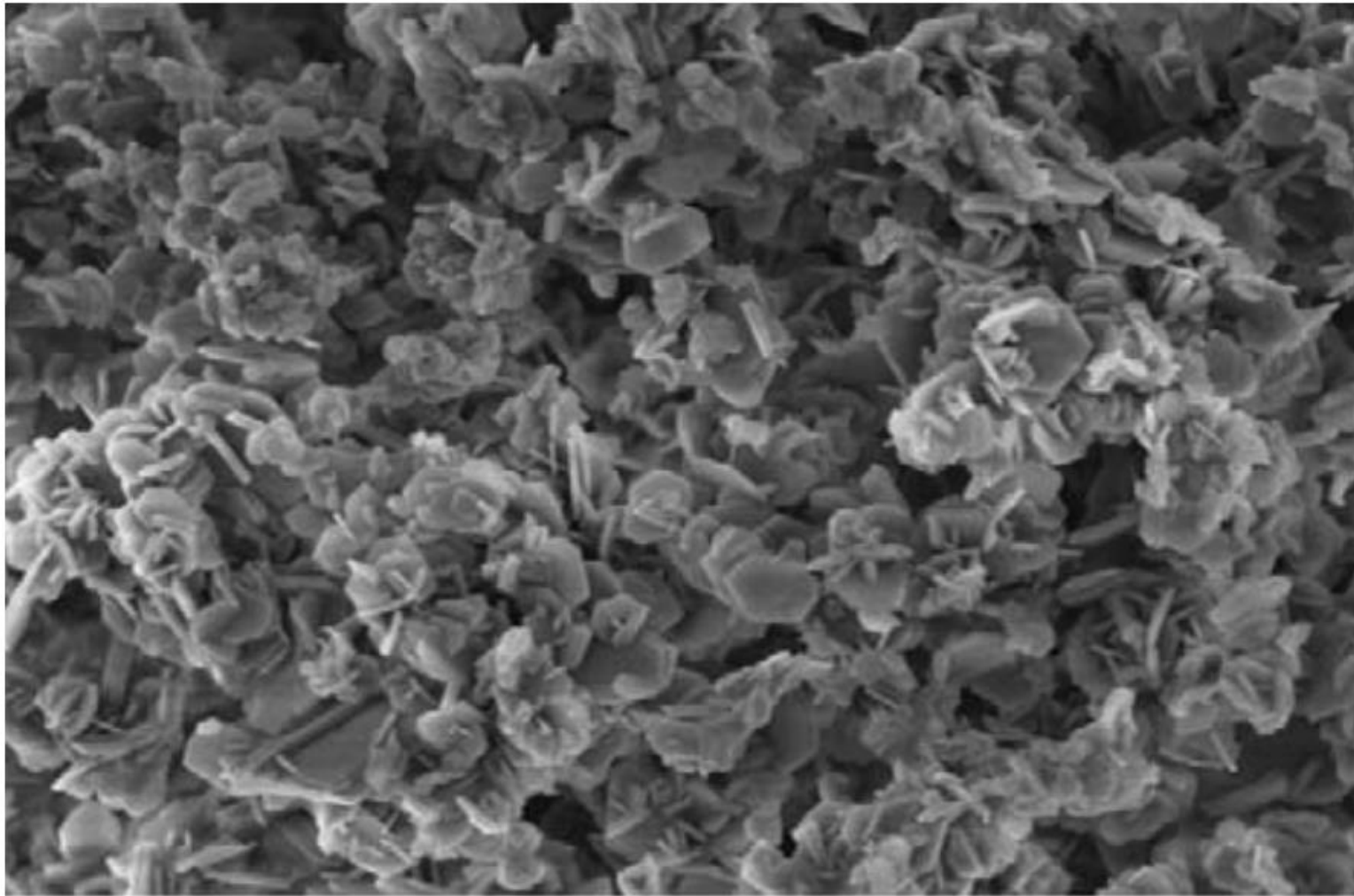
Striations



High Speed Film



MgOH



1 μm

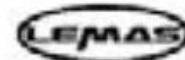


Mag = 50.00 K X

EHT = 3.00 kV

Signal A = InLens

WD = 2.6 mm



Date :11 May 2011

Image taken from Technology Development and Delivery Summary 2011 – Sellafield sites

Ferric floc



- Used in EARP
- Co-precipitates heavy metal ions
- Needs to be concentrated / dewatered
- Is a much 'fluffier' and 'stickier' substance than minerals (i.e. CaCO_3 and MgOH)

Future work

- Intend to investigate the following:
 - Numerical simulation (COMSOL) – to understand shear at the cake surface
 - Cake formation – fundamentals (further work required)
 - $\text{Mg}(\text{OH})_2$
 - Ferric Floc
 - Design application

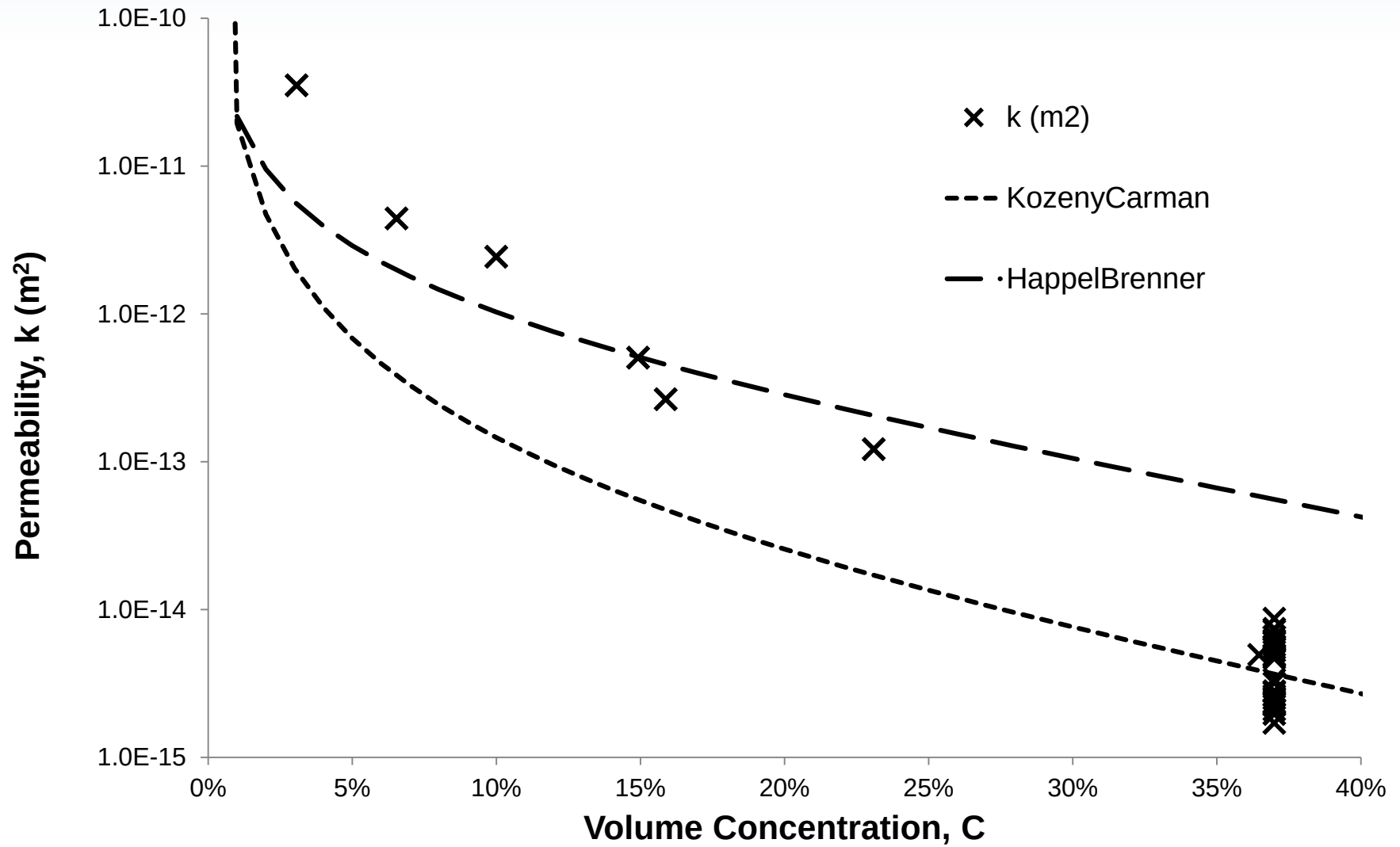
Conclusions

- Particle retention relies on the formation of a filter cake
- The resistance to filtration, and therefore the flux rate are dependent on the cake thickness
- There is a very strong correlation between cake thickness and shear stress
- The friction model is described by a large number of authors, and when applied, along with other derivations a predictive model can be constructed.
- The current predictive model of L is not accurate.
- The formation of the filter cake is independent on the type of shear (azimuthal or axial)
- The type of filter used has negligible effect on the overall resistance, provided a cake is formed, as the resistance is dependent on that cake.

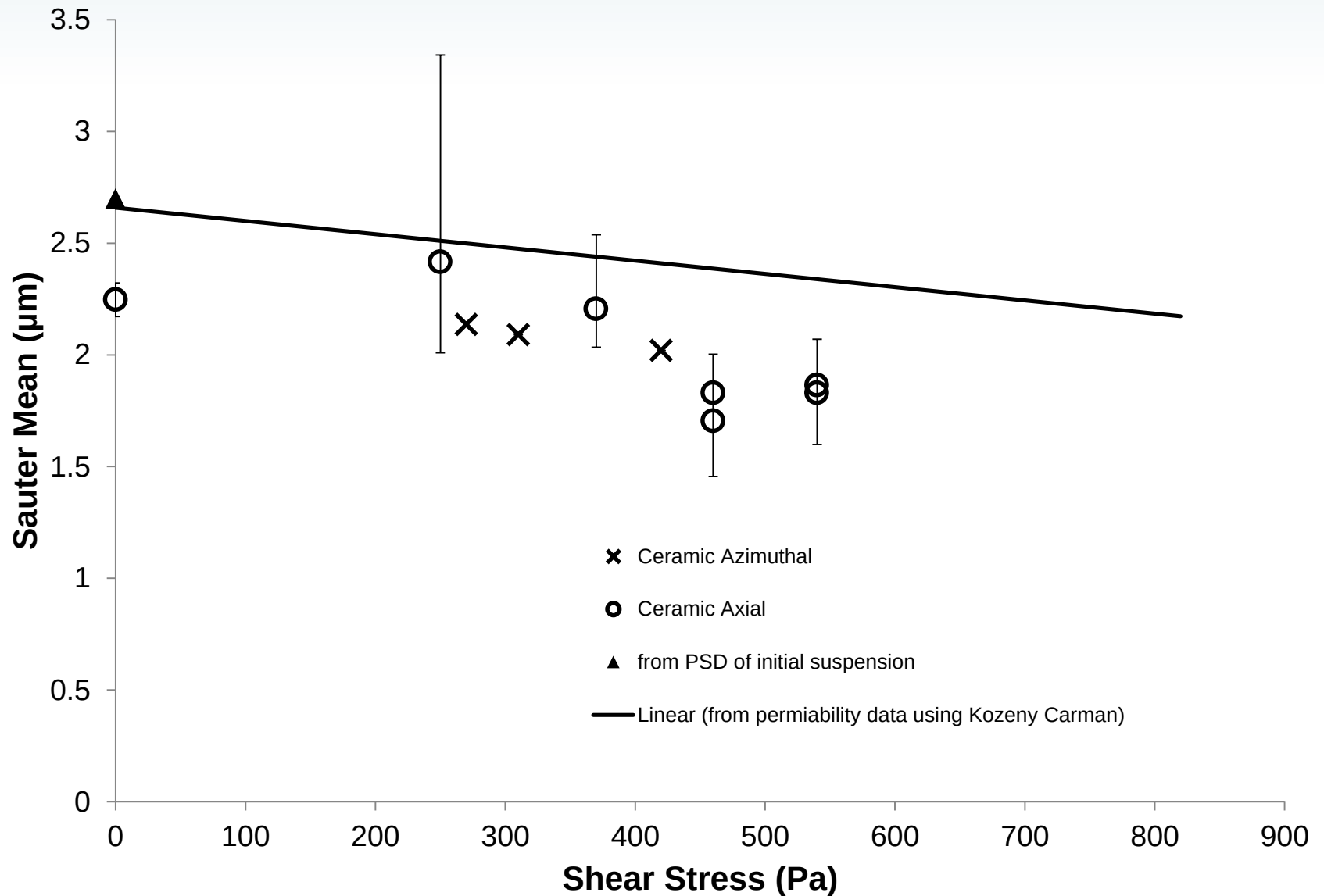
Questions



Permeability



Sauter mean of cake

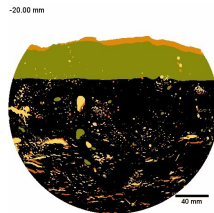
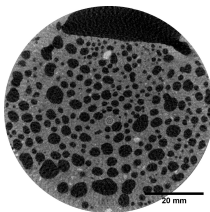
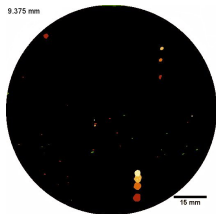


The evolution of bubble populations in Magnox legacy waste

Michael Johnson, Timothy Hunter, David Harbottle, Jeff Peakall,
Michael Fairweather and Simon Biggs

University of Leeds
School of Chemical and Process Engineering

November 2016



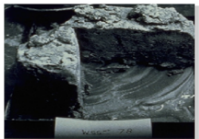
Gas retention in legacy waste



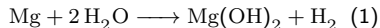
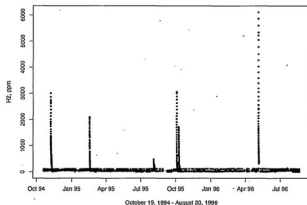
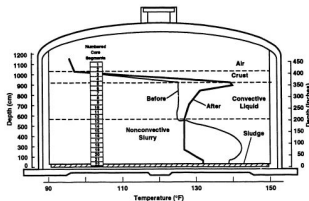
A - Magnox Fuel Can



B - Magnox - Partially Corroded Swarf and Magnesium Hydroxide Sludge



C - Magnox Sludge - Active Magnesium Hydroxide Sludge Sample

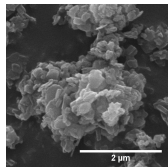


- Magnox cladding corrodes and consolidates into dense beds of CMS
- Hanford engineers have observed periodic upward transfers of decay heat coinciding with spikes in H_2 concentration in the ullage, considered to be *rollover events*

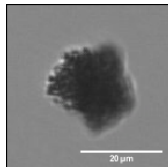
Hastings *et al.* 2007

Allemann 1992

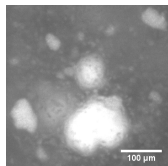
Test material characterisation



SEM

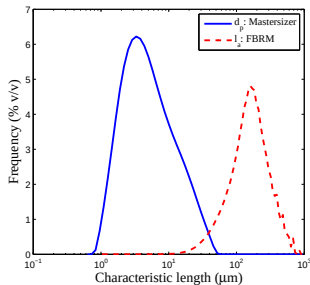
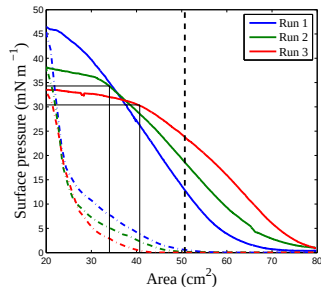
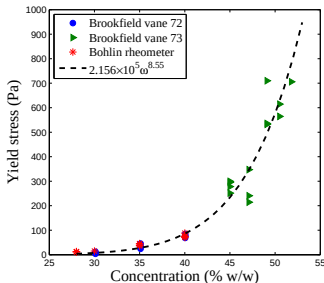


Sysmex

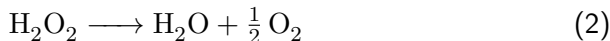
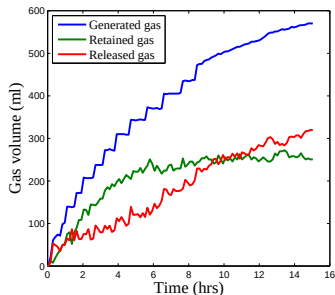
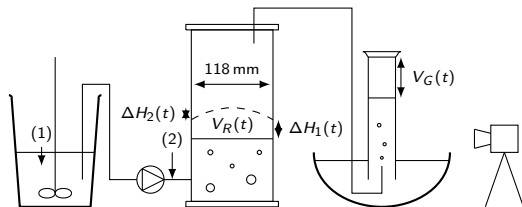


PVM

Johnson et al. 2016



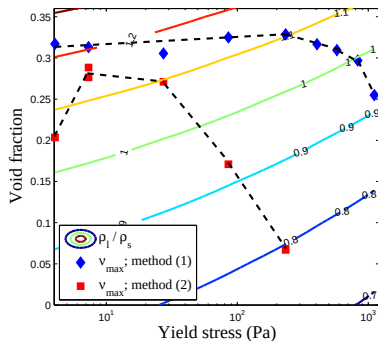
Gas retention tests



$$V_R(t) = \pi R^2 \Delta H_1(t) + \frac{1}{3} \pi \Delta H_2(t)^2 (3R - \Delta H_2(t)) \quad (3)$$

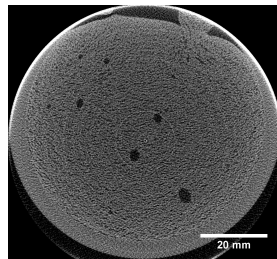
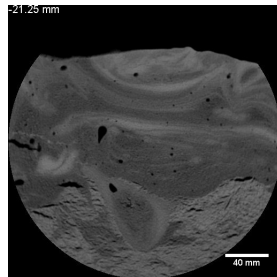
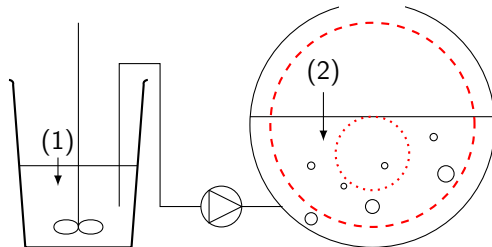
$$V_G(t) = V_R(t) + V_E(t) \quad (4)$$

Feasibility of rollover events



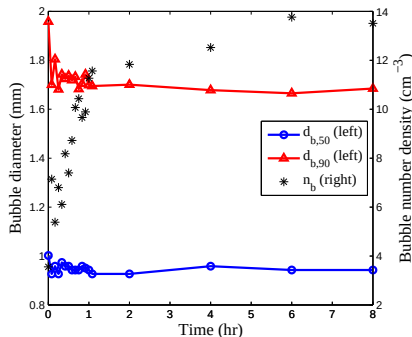
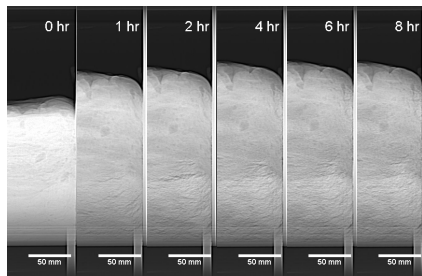
- Void fractions upward of 30 % are observed in sediments of $\tau < 600$ Pa
- Sediments of $\tau < 800$ Pa can experience sufficient *bed swell* to potentially induce *rollover*
- Continuous injection of H_2O_2 into the feedline significantly reduces the capacity for bed expansion, especially in weak ($\tau < 30$ Pa) and medium strength ($80 < \tau < 240$ Pa) sediments

X-ray CT: imaging methodology



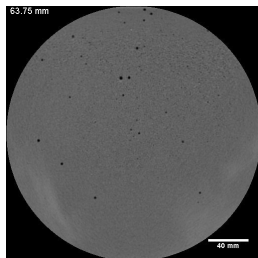
	GE Brivo		Siemens Inveon
	Large FOV	Small FOV	
X-ray voltage (kV _p)	120	120	80
X-ray tube current (mA)	40	79	0.5
FOV diameter (mm)	250	96	81.8
Pixel resolution (μm)	488	250	53.25
Slice separation (μm)	1250	625	53.25
Number of axial slices	112	32	1024
Axial FOV depth (mm)	138.8	19.4	54.5
Total FOV volume (mm ³)	6.81×10^6	1.40×10^5	2.86×10^5

Evolution in bubble size and number

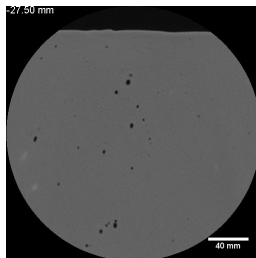


- Bed expansion occurs within the first 4 h
- The median and d_{90} bubble sizes (mature bubbles only) reach a steady state within the first hour
- The increase in voidage is mirrored by the increase in number of mature (millimetre scale) bubbles

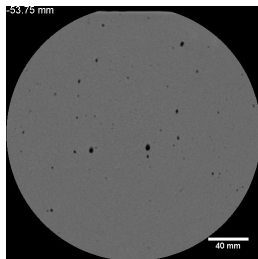
Impact of yield stress on bubble size and shape



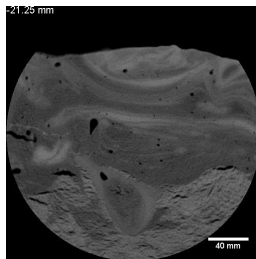
7 Pa



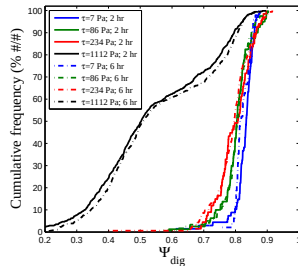
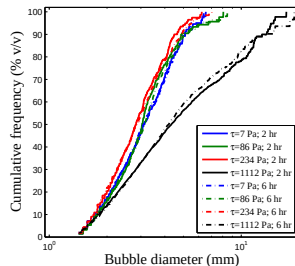
86 Pa



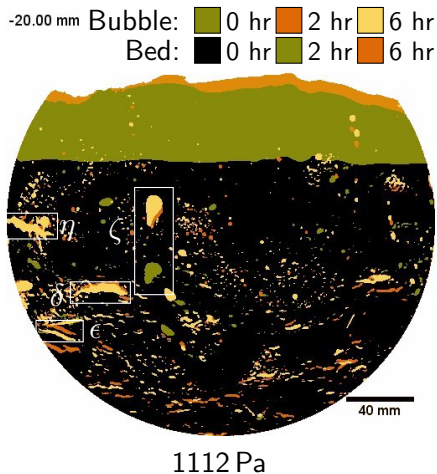
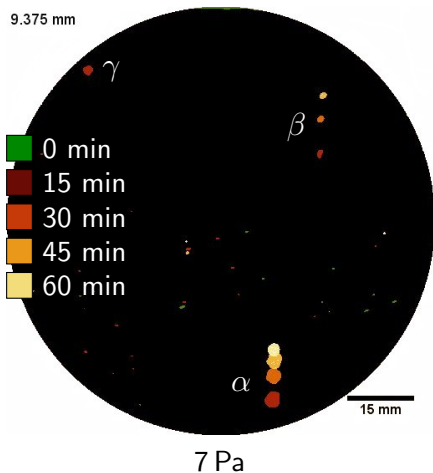
234 Pa



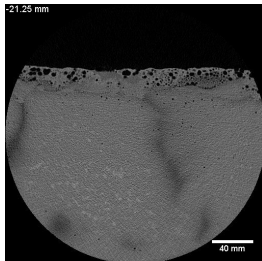
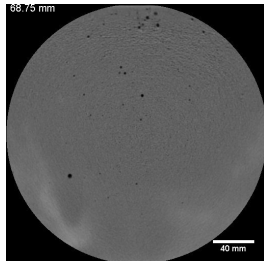
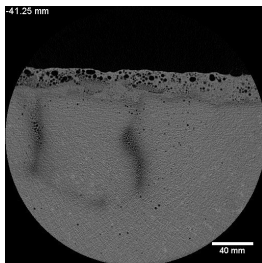
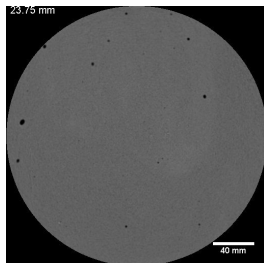
1112 Pa



Bubble motion and residence times



Non-homogeneous gas generation

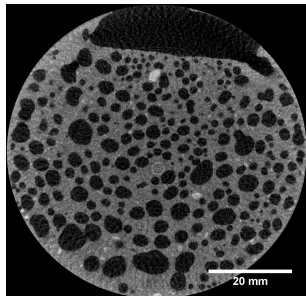
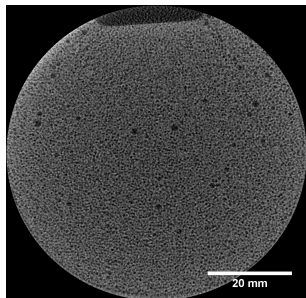


Method (1)

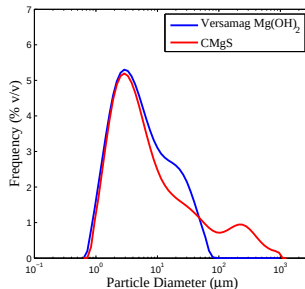
Method (2)

- Homogenous distribution of peroxide results in greater bulk bed expansion (or a reduced rate of gas release)
- Less homogeneous distribution of volatiles resulted in (1) a high porosity foam layer at the surface and (2) dark regions of low radiodensity

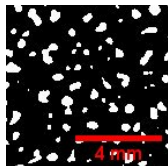
Comparison of test materials



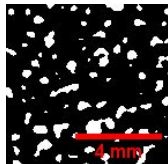
- Higher resolution ($d_{\text{vox}} = 53.25 \mu\text{m}$) CT images captured at the Centre for Advanced Imaging, University of Queensland
- CMgS exhibits a vastly coarser bubble population after 6 h *in situ* gas generation than the commercial $\text{Mg}(\text{OH})_2$ ($\tau \approx 30 \text{ Pa}$ for both samples)



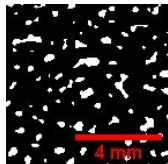
Pore scale bubble growth



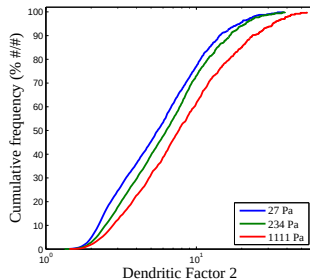
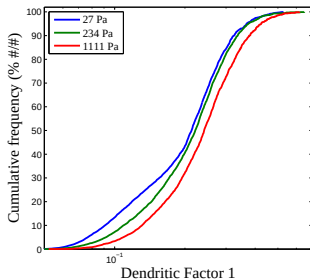
27 Pa



234 Pa



1111 Pa



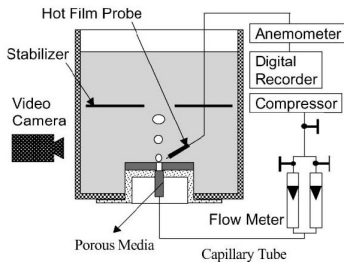
$$DF1 = \frac{\sigma d_i}{d_b} \quad (5)$$

$$DF2 = \frac{V_{Bound}}{V_b} \quad (6)$$

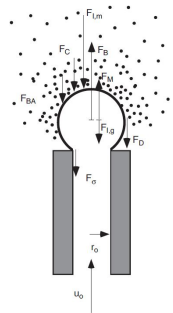
$$\frac{\Delta P_{CI}}{\Delta P_{CE}} = \frac{\frac{\gamma}{r_{th}} \cos(\theta)}{\frac{4}{3} \tau \left(1 + \ln \left| \frac{G}{\tau} \right| \right)} \propto \frac{\gamma \cos(\theta)}{\tau r_{th}} \quad (7)$$

Forthcoming work

- 1 Couple the pore scale bubble growth results with micro-CT characterisation of the two-phase pore structure
- 2 Investigate the mechanics and flow behaviour of gassy soft sediments
- 3 Investigate the growth kinetics and buoyant motion of bubbles from a single submerged orifice



from Zhang and Shoji 2001



from Yang *et al.* 2007

Conclusions

- ① Sediments of $\tau < 800$ Pa can experience sufficient *bed swell* to potentially induce *rollover events*
- ② Continuous injection of H_2O_2 into the feedline resulted in localised pockets of gas generation resulting in low density regions, rich in microbubbles, which appear to facilitate enhanced gas release
- ③ Sediments > 1 kPa exhibit bubble growth by induction of lateral fractures which appear to enable gas transport to the bed periphery
- ④ Sediments of $7 < \tau < 234$ Pa retain virtually identical size distributions of mature bubbles, implying a release mechanism unrelated to bubble buoyancy
- ⑤ Pore scale bubbles appear to become more dendritic with increased bed strength
- ⑥ Corroded magnesium metal sediments appear to support much coarse bubble size distributions than commercial $\text{Mg}(\text{OH})_2$ sediments

Thank you for listening

Supervisors and collaborators



Tim
Hunter



Mike
Fairweather



Dave
Harbottle



Jeff
Peakall



Simon
Biggs

...with thanks also to Geoff Randall and Martyn Barnes of Sellafield Ltd.
and for funding and support from





Novel Characterisation of Flocculated Dispersions using Acoustic Backscatter Systems

Alastair Tonge

Supervisors: Dr Tim Hunter, Prof Steven Freear & Prof. Jeff Peakall

Acknowledgements



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- Sellafield and the NDA
- Martyn Barnes & Geoff Randall
- Hugh Rice and Jaiyana Bux
- Dr Tim Hunter, Prof. Jeff Peakall and Prof. Steven Freear

Motivation - Aims of acoustic characterisation



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*First Generation Magnox
Storage Pond - Sellafield*



Water Treatment Thickener

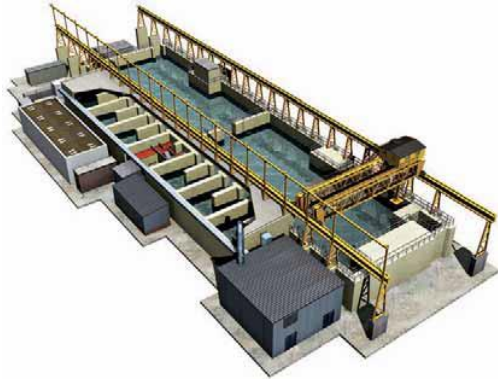
- ❖ Analysis of settling front and concentration profiles in small-scale test settling suspensions.
- ❖ Modelling prediction and *In situ* monitoring of sludges to optimise of processing, transport and separation.
- ❖ Additional applications in water treatment & minerals thickeners

**Major advantage:
Ability to observe
changes in
visually opaque
suspensions**

Motivation – Sellafield Pile Fuel Storage Pond



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- Spent fuel from the first Windscale pile reactors has been stored in the PFSP for > 50 years.
- Corrosion of the waste material and additional organic matter has resulted in a significant build up of sludge.
- Sludge recovery is on-going as part of a decommissioning programme with settlement in a corral prior to removal for ultimate encapsulation.

$$V_{\text{rms}} = \frac{k_s k_t}{\psi r} C^{\frac{1}{2}} e^{-2r(\alpha_w + \alpha_s)}$$

k_s particle species backscatter co-efficient

k_t transducer constant

ψ near field correction factor

r distance from transducer face

C mass concentration

α_w attenuation of water

α_s attenuation of suspended particles

$$G = \ln(\psi r V_{\text{rms}}) = \ln(k_{sh} k_t) + \frac{1}{2} \ln C - 2r(\alpha_w + \alpha_{sh})$$

k_{sh}	particle species backscatter co-efficient (under homogeneous conditions)
k_t	transducer constant
ψ	near field correction factor
r	distance from transducer face
C	mass concentration
α_w	attenuation of water
α_{sh}	attenuation of suspended particles (under homogeneous conditions)

$$\frac{\partial G}{\partial r} = -2(\alpha_w + \alpha_s)$$

$$\alpha_w = 0.05641 f^2 e^{\left(-\frac{T}{27}\right)} \quad \alpha_s = \xi_h C$$

~~k_s — particle species backscatter co-efficient~~

~~k_t — transducer constant~~

ψ — near field correction factor

r — distance from transducer face

C — mass concentration

α_w — attenuation of water

α_s — attenuation of suspended particles

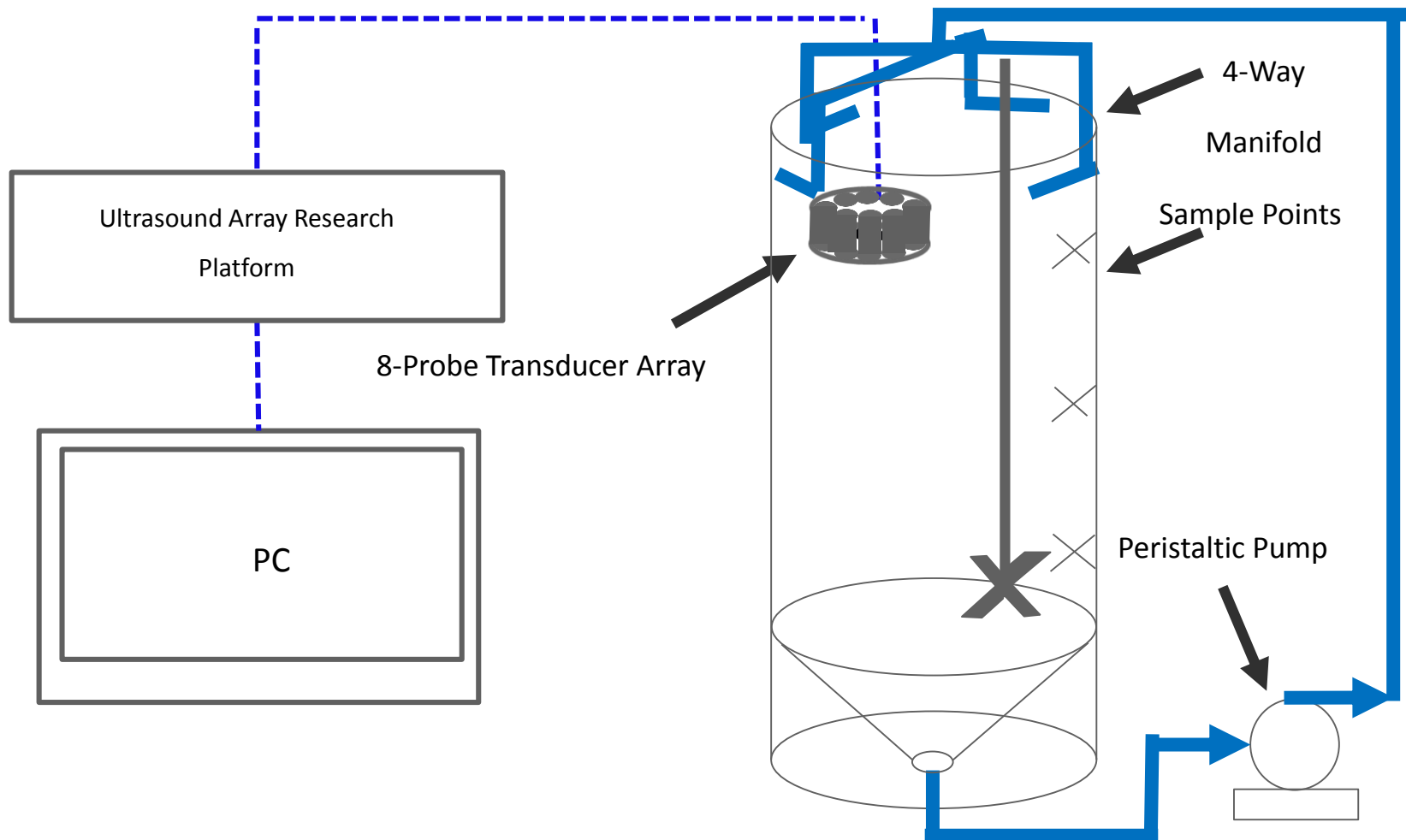
Acoustics Calibration

Instrument in prototype enclosure



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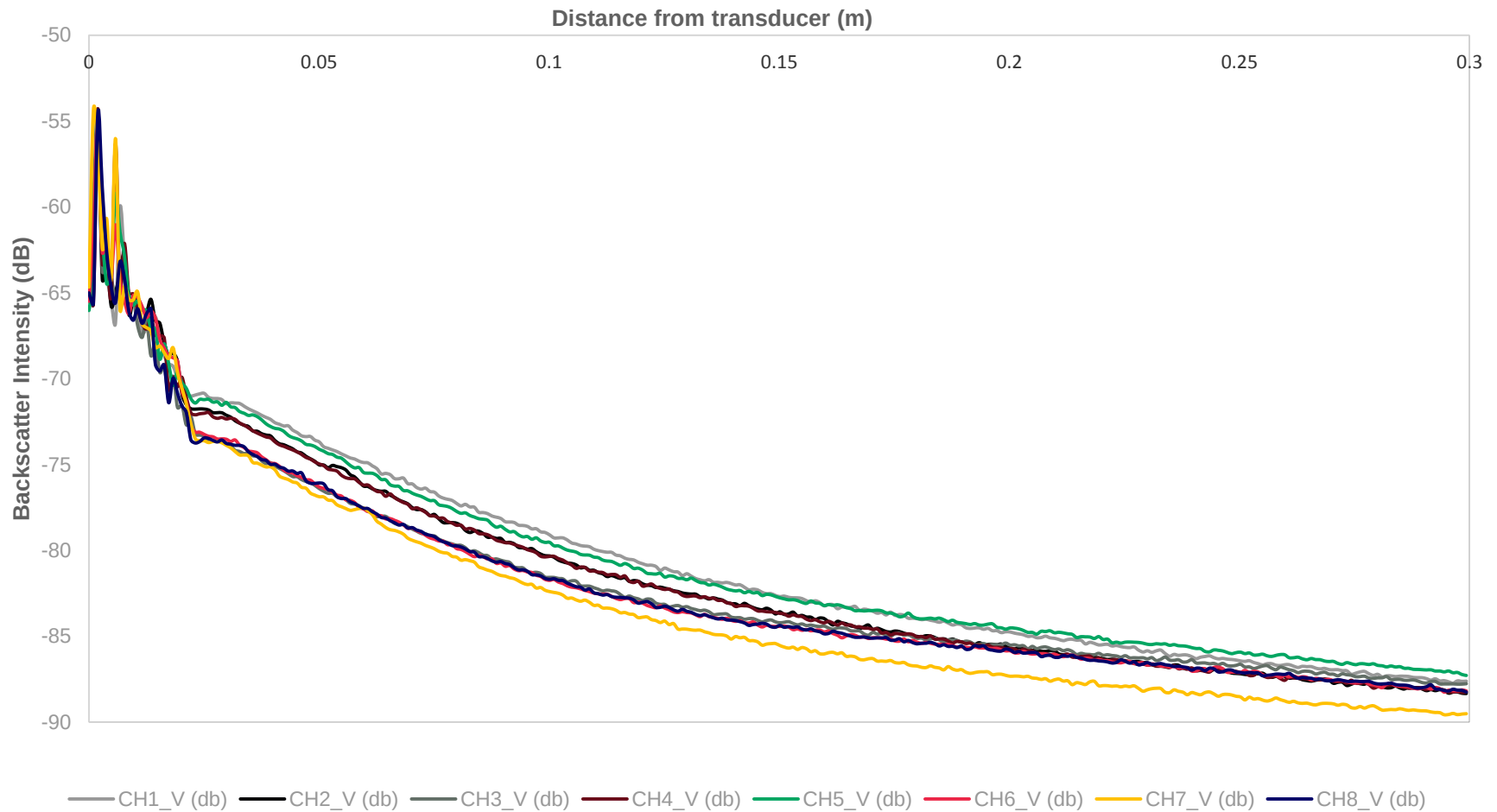




Calibration Data



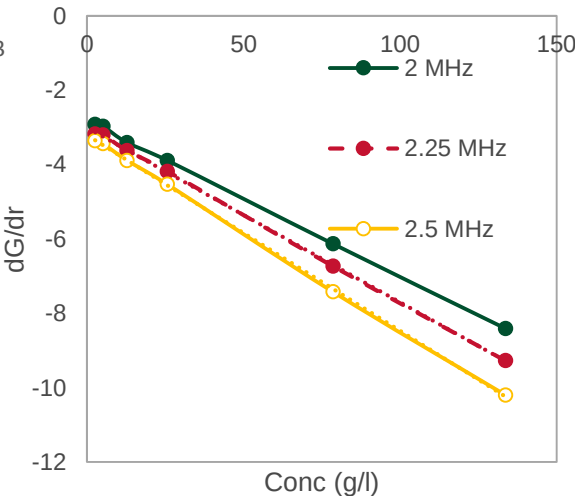
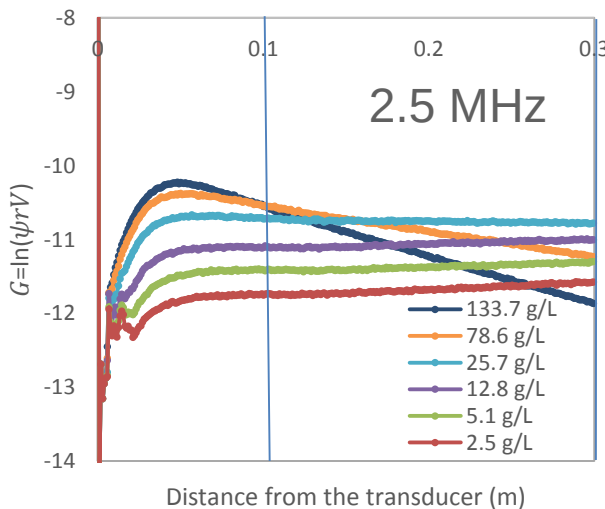
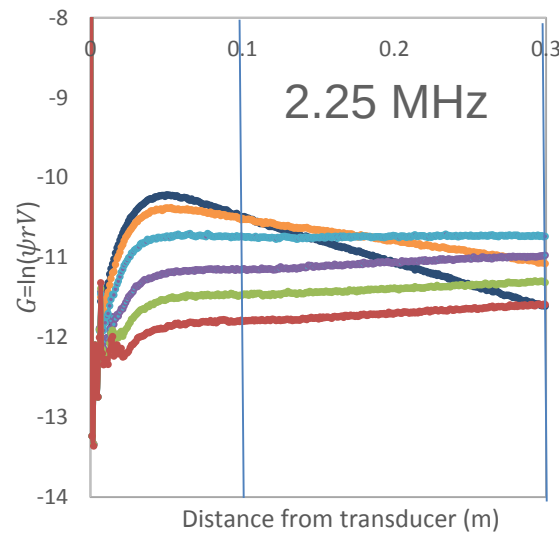
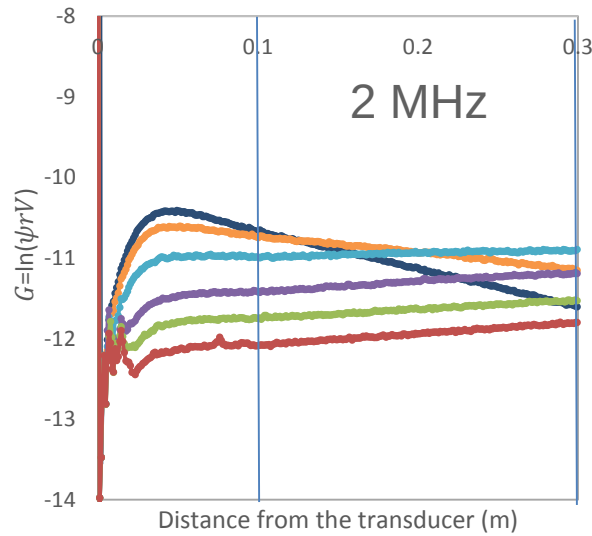
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Calibration Data



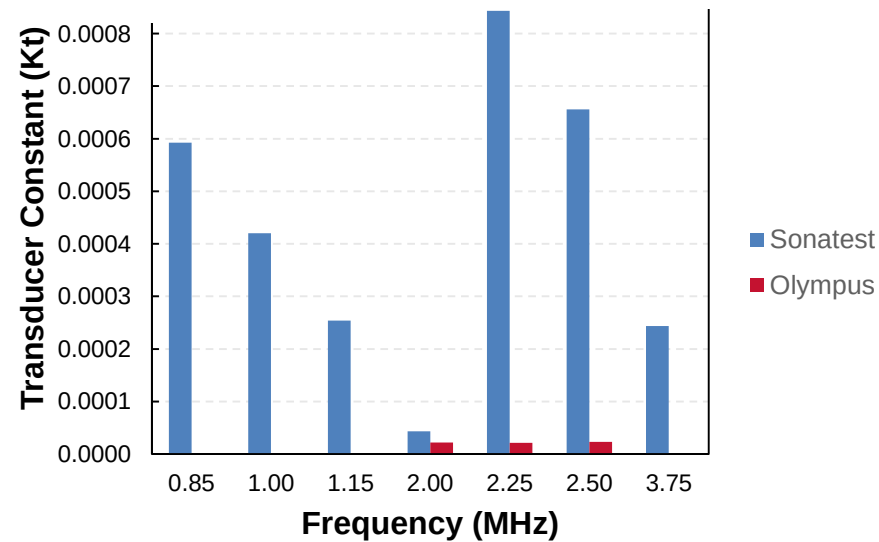
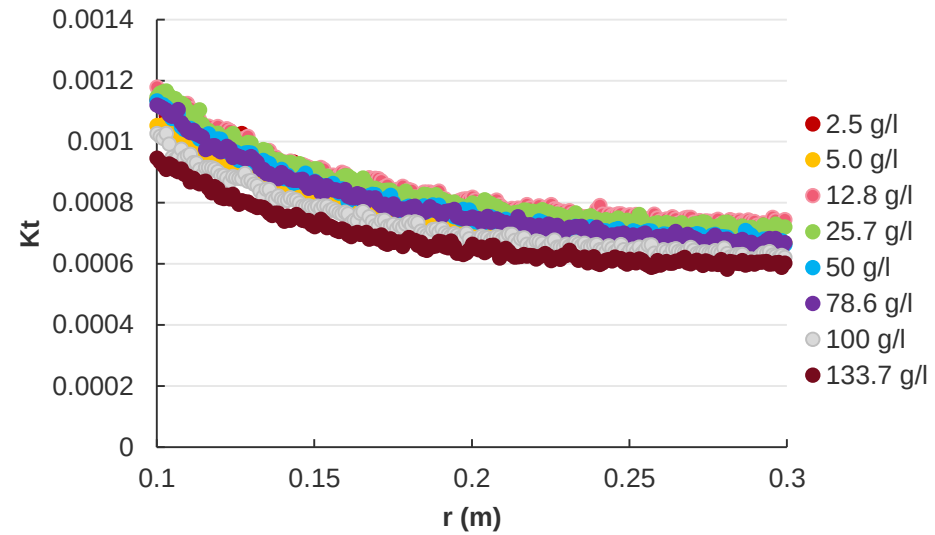
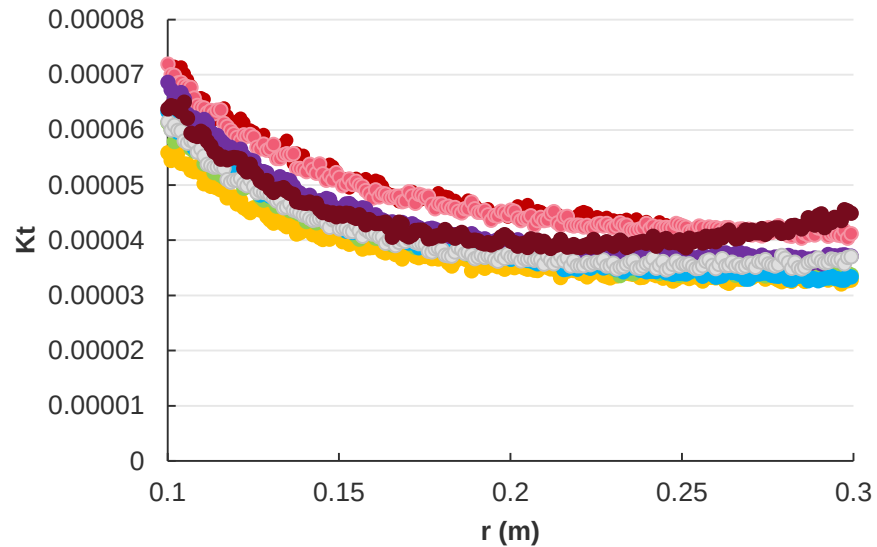
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$$\frac{\partial G}{\partial r} = -2(\alpha_w + \alpha_s)$$

$$\frac{\partial G}{\partial r} = -2(\alpha_w + \xi_h C)$$

$$\xi_h = -\frac{1}{2} \left(\frac{\partial G}{\partial r} \right) \frac{1}{\partial C}$$



$$k = \frac{2\pi}{\lambda}$$

$$\chi = \frac{4 a_s \rho_s \xi}{3}$$

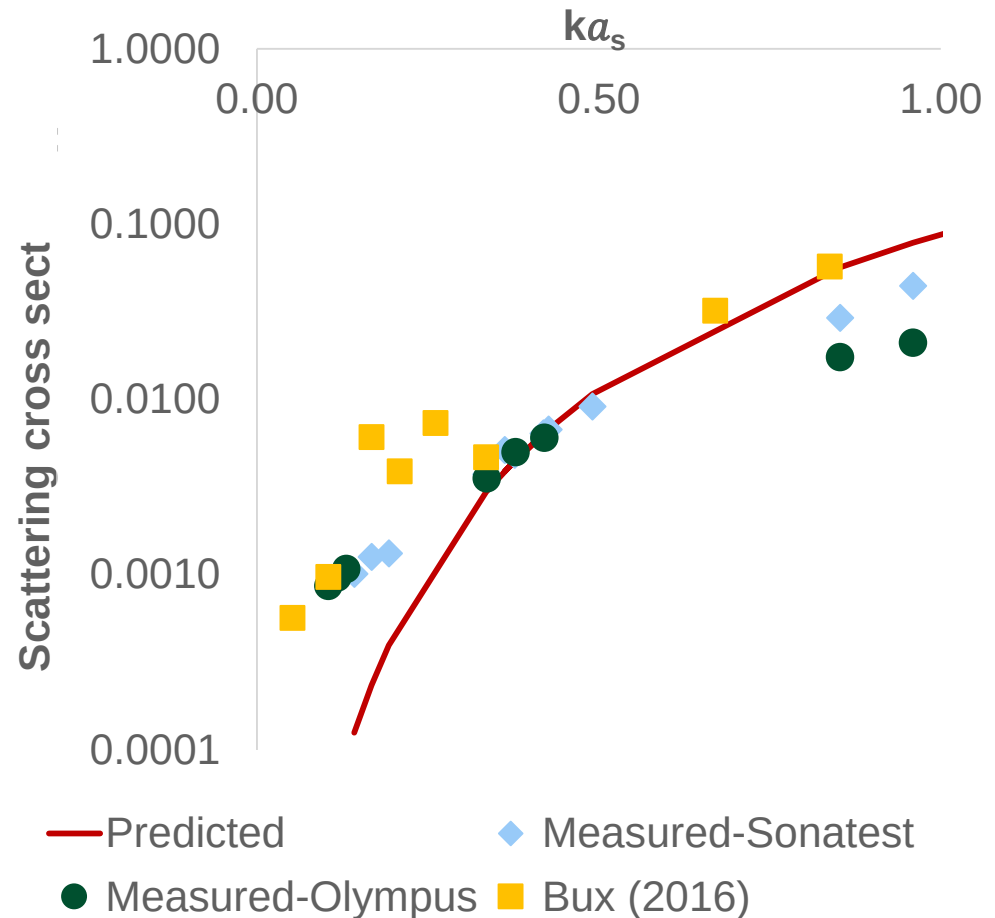
k = Wavenumber (m^{-1})

λ = Wavelength (m)

χ = Scattering cross section (m^2)

ρ_s = Sediment particle density (kgm^{-3})

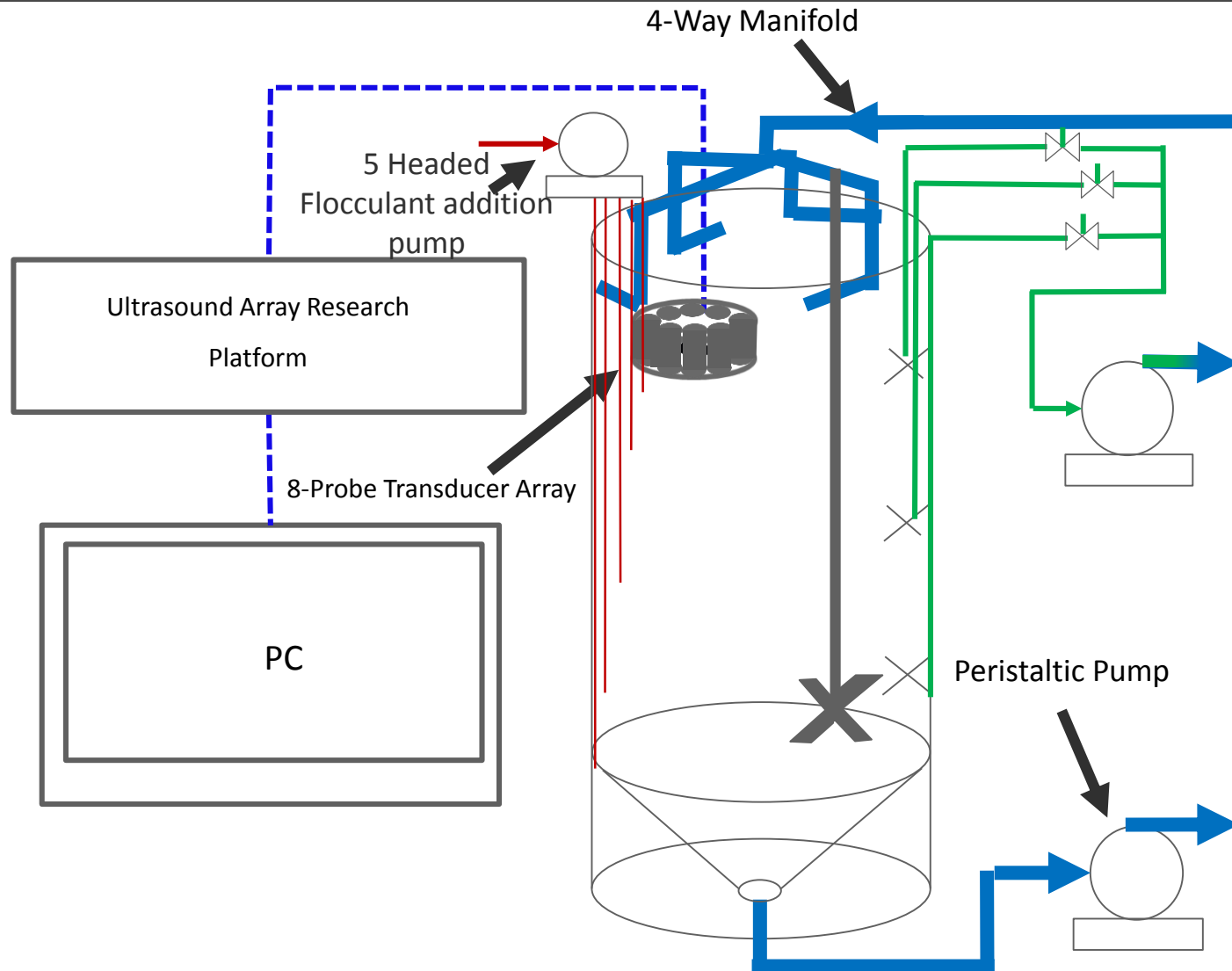
a_s = Particle diameter (m)



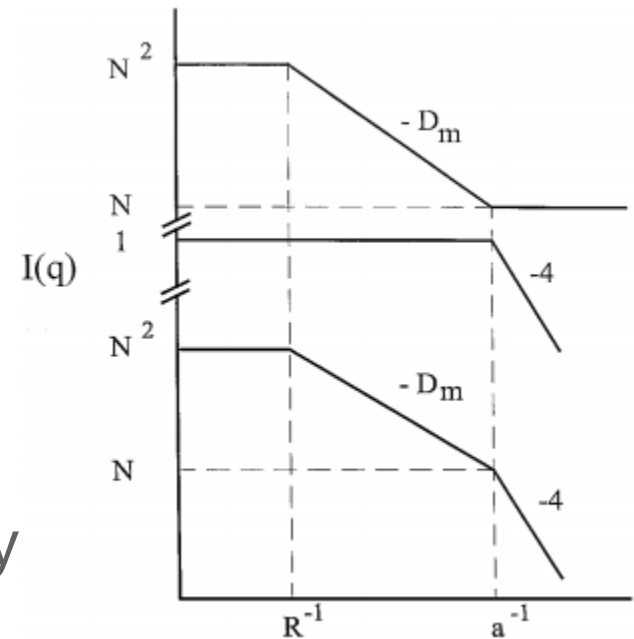
New Sampling System



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- Collect large angle light scattering (LALS) for flocs using Saturn Digisizer
- Set up new sampling/ flocculant delivery system



- Synthesise latex particles using XME rig and collect acoustic and size data (FBRM, mastersizer, digisizer) to compare to data collected by Bux (2016)
- Repeat previous flocculation experiments and document size change over time using FBRM so that flocs may be characterised acoustically according to size and structure