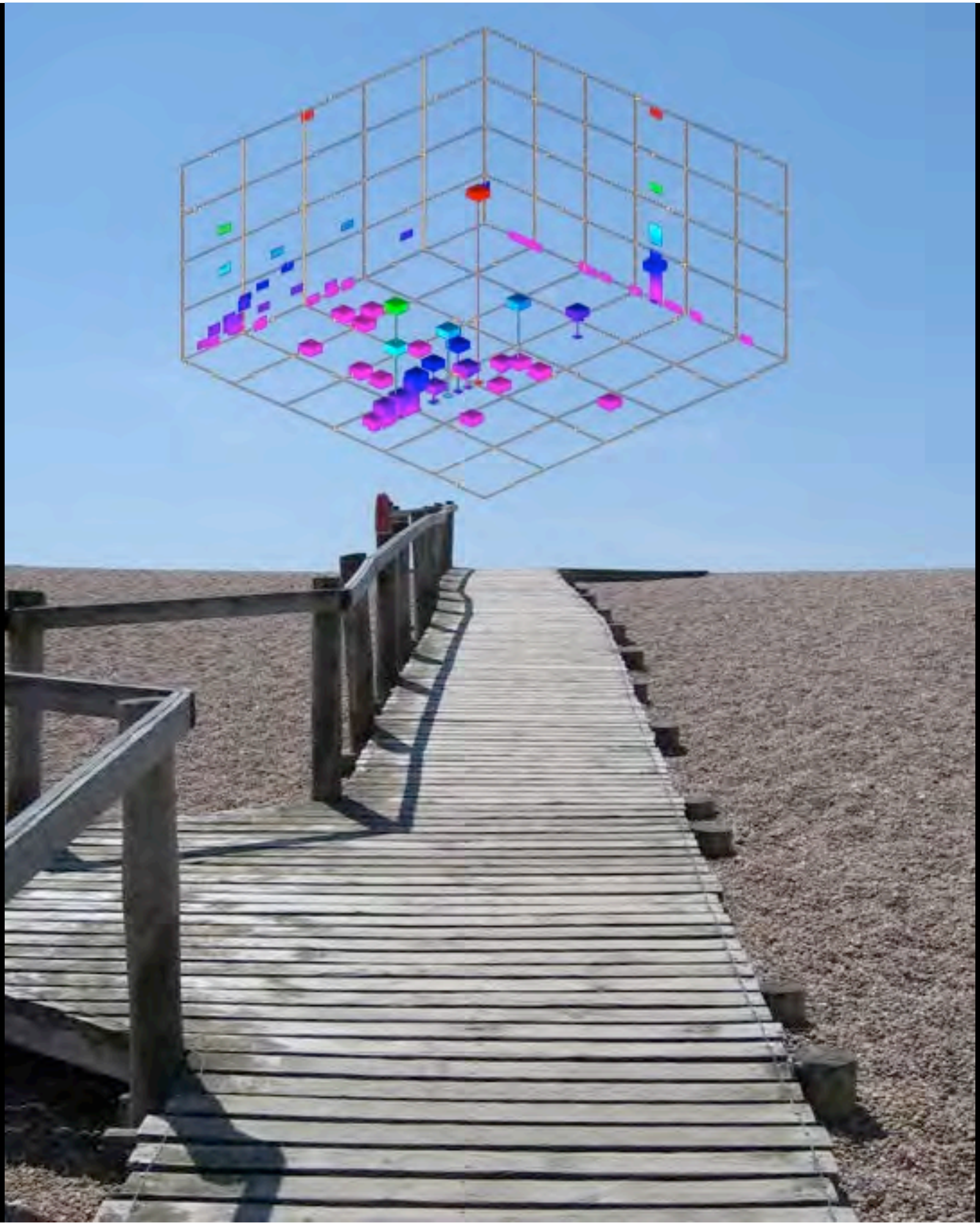


“It was gravity which pulled us down…”

Richard Buscall

MSACT Research & Consulting

Some of today’s speakers work on sedimentation & creaming.

“It was gravity which pulled us down and destiny which broke us apart”.

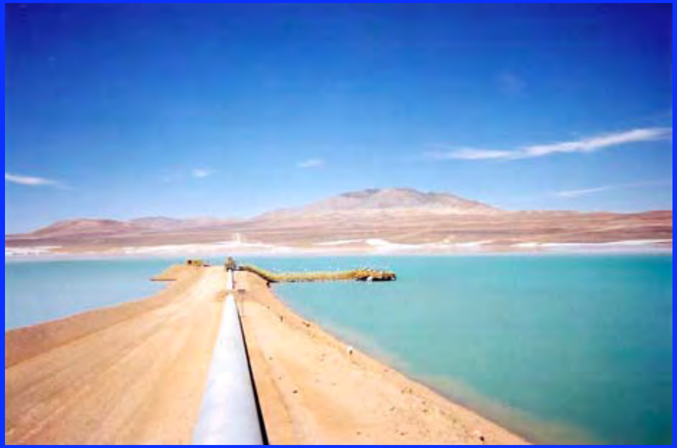
(from “Idiot Wind” by Bob Dylan).

Some of them have worked for ICI.

Sedimentation of concentrated suspensions

Oil Sands tailings pond - Alberta

Water recovery ponds at Escondida in the Atacama desert, Chile



Fabric softener

So-called salad dressing

Sedimentation of concentrated dispersions

Possibilities

- Discrete particles (colloidally stable)
    - monodisperse.
    - polydisperse.
    - aspherical.
- Aggregated particles ("gelled" systems)
    - Strongly aggregated ("frozen")
    - Weakly aggregated (susceptible to fluctuations).

Sedimentation of concentrated dispersions

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- Discrete particles (colloidally stable)
  - monodisperse.
  - polydisperse.
  - aspherical.
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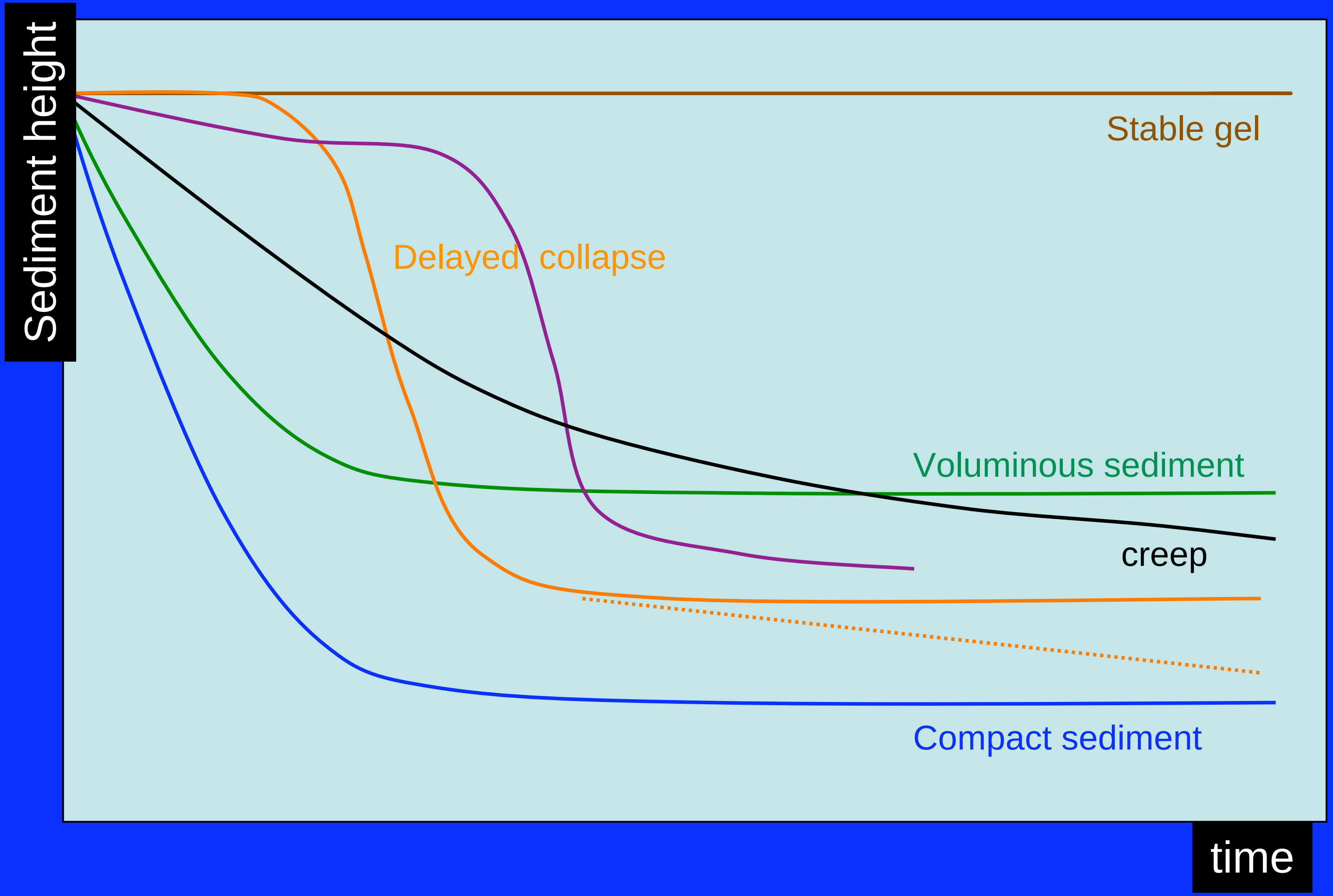
  - Strongly aggregated (“frozen”)
  - Weakly aggregated (susceptible to fluctuations).

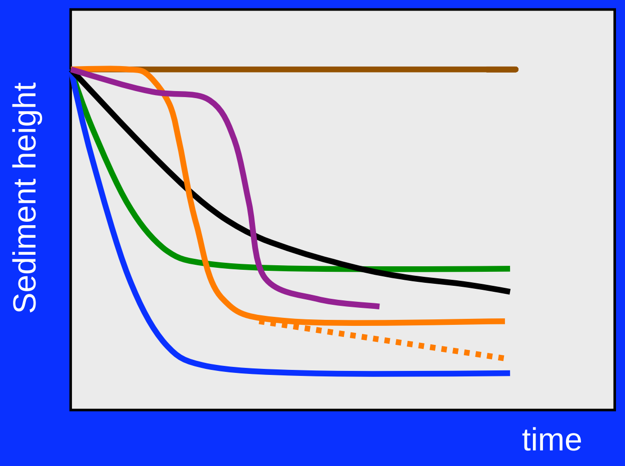
Sedimentation curves (schematic)

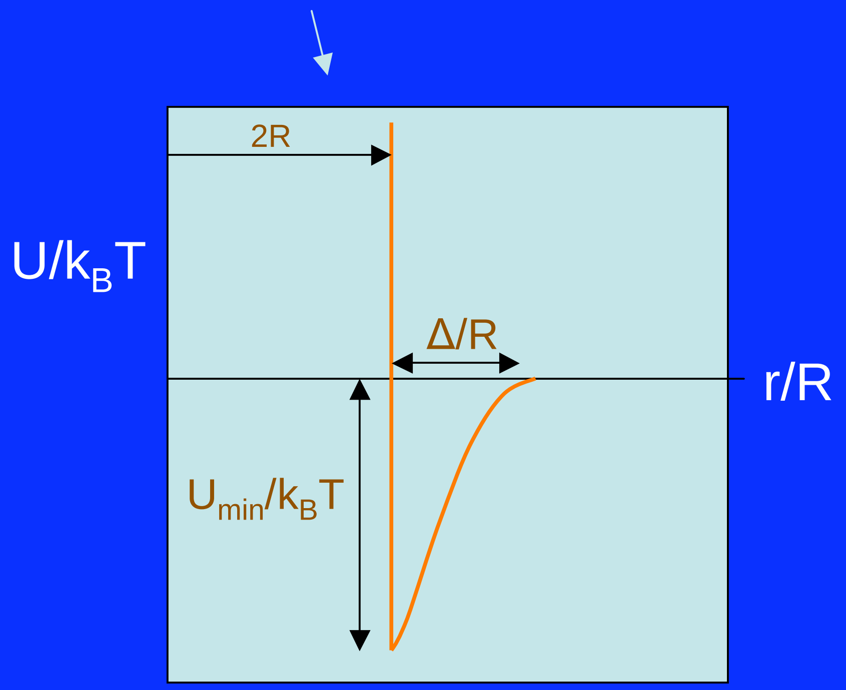
A diverse range of behaviours seen.



Variables

- Particle concentration - φ
- Mean particle size - R
- Particle and liquid density - Δρ
- Liquid viscosity - μ
- Column height and width - H & D
- Interaction potential - depth & width crudely





U_{min}/k_BT >> 20

Strong poroelastic gels

U_{min}/k_BT < 20

Weak gels

Creep,
Coarsening &
Collapse

Strong gels

(Strong) gel formation - conceptual

H Tanaka

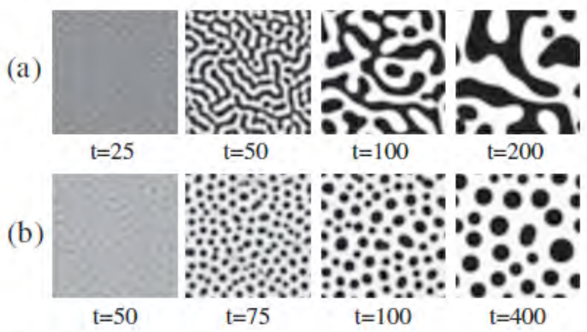


Figure 1. Typical phase-separation processes for (a) bicontinuous and (b) droplet spinodal decomposition in a two-dimensional fluid mixture. They are simulated by numerically solving the basic equations of model H. Time is scaled by the characteristic time τ.

The system would like to phase separate via spinodal decomposition, except that this is arrested by gel formation. The usual condition for the spinodal line, $\frac{d\Pi_{os}}{d\ln\varphi}=0$ is replaced by,

$$\frac{d\Pi_{os}}{d\ln\varphi} + K(\varphi) = 0$$

where $K(\varphi)$ is the compressional modulus of the network or gel.

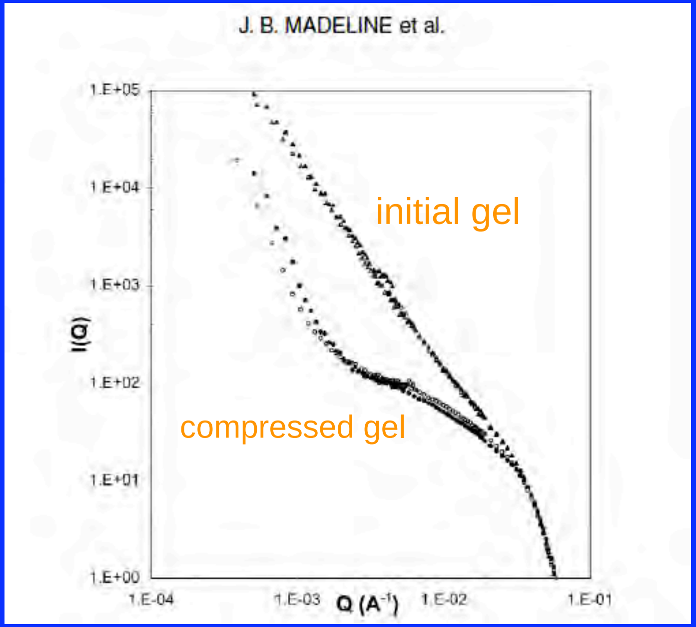
Coarsening by diffusive growth (particle hopping) still admissible, although rate will be negligible for strong gels.

H. Tanaka, Viscoelastic model of phase separation in colloidal suspensions and emulsions, Physical Review E Vol. 59, No. 6, pp. 6842-6852 (1999).

H.Tanaka, Viscoelastic phase separation, Journal of Physics: Condensed Matter Vol. 12, No. 15, pp. R207-R264 (2000).

Strong gels - structural

- e.g. Cabane et al. Neutron scattering & TEM



From colloidal dispersions to colloidal pastes through solid/liquid separation processes J.B. Madeline, M. Meireles, J. Persello, C. Martin, R. Botet, R. Schweins, B. Cabane Pure and Applied Chemistry 77 (2005) 1369.
Restructuring of colloidal cakes during dewatering Madeline, J.B. and Meireles, Martine and Bourgerette, C and Botet, Robert and Schweins, R and Cabane, Bernard (2007). Langmuir, vol. 23 (n− 4). pp. 1645-1658. ISSN 0743-7463

Sedimentation of strong gels overview

- Understood at phenomenological level
  - fluid drag function $\Gamma(\varphi)$
  - compressive strength $p_y(\varphi)$
  - adhesive shear strength $\tau_w(\varphi)$

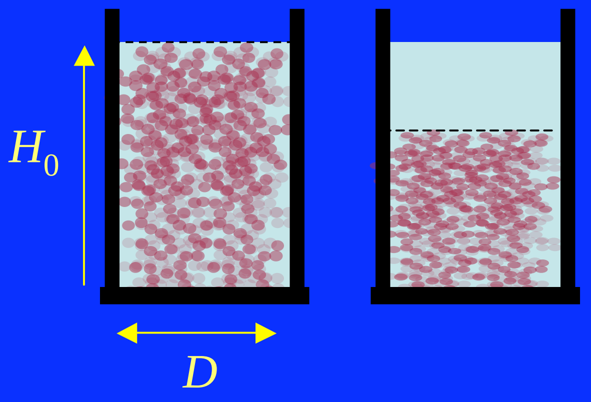
A.S. Michaels, J.C. Bolger, Ind. Eng. Chem. Fund., **1**(3) 24-33 (1962).
D.C. Dixon, Sep. Sci., **12**, 171-191 (1971).
Tiller, F.M., Khatib, Z.,.J. Colloid Interface Sci., 100, 55– 67 (1984).
R. Buscall & L.R. White, J. Chem. Soc. Faraday Trans. 1, (1987), 83 873.
Howells, I., Landman, K.A., Panjkov, A., Sirakoff, C., White, L.R., Appl. Math.Modelling, **14**:77-86 (1990).
S.P. Usher, P.J .Scales and L.R. White, AIChE Journal, **52**(3): 986-993 (2006).

Strong gels in this talk

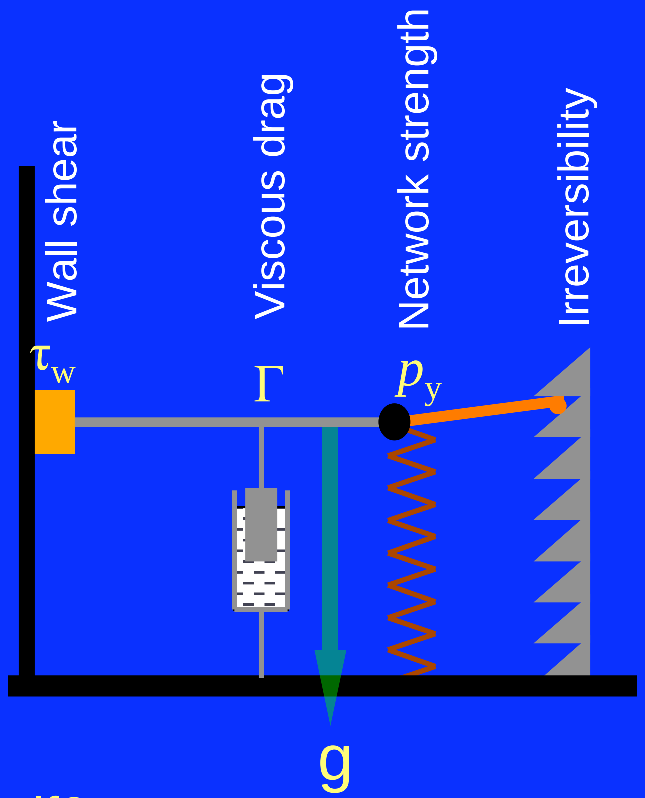
- Compressive versus adhesive shear strength
  - Is the gel held up by the walls or by itself?

Consensus phenomenological model of a strong gel

Following Michaels & Bolger… Ind. Eng. Chem. Fund., **1**(3) 24-33 (1962).



(N.b. all material coeff's increase with -ve volumetric strain)



$$\frac{p_y}{\Delta\rho g \varphi_0 H_0}\left(1+4S\frac{H_0}{D}\right)$$

Is the gel held up by the walls or by itself?

Compressive strength p_y versus adhesive shear strength τ_w (n.b. $S = \tau_w / p_y$).

Consensus phenomenological model of a strong gel ?

Accessible review?

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30) The role of solid friction in the sedimentation of strongly attractive colloidal gels, Jean-Michel Condre, Christian Ligoure and Luca Cipelletti J. Stat. Mech. (2007) P02010 doi: 10.1088/1742-5468/2007/02/P02010
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Poroelastic constitutive eqn

Colloids Surf., 5(4) (1982) 269-283., arXiv:0903.0970v1 [cond-mat.soft] (2009); J. non-Newtonian Fluid Mech. (submitted).

Compressive strength is given by integrating the bulk modulus $K(\varphi)$ over the total volumetric strain from *gel point* upwards.

$$p_y(\varphi) = \int_{\varphi_g}^{\varphi} K(x)\, \mathrm{d}\ln x$$

Strong networks are brittle in shear though, i.e. the critical strain γ_c is usually small**,

$$\tau_w(\varphi) = \int_{0}^{\gamma_c} G(x)\, \mathrm{d}\ln x$$

whence the ratio of shear to compressive strength is expected to decrease with increasing volume-fraction.

** e.g. Uhlherr, P.H.T., Guo, J., Tiu, C., Zhang, X.M., Zhou, J.Z.Q., Fang, T.N. , Journal of Non-Newtonian Fluid Mechanics, 125 (2), 101-119, (2005); A.L. Tindley, The effect of electrolytes on the properties of titanium dioxide dispersions, Ph.D. Thesis, Engineering Faculty, University of Leeds, (2007).

Poroelastic constitutive eqn

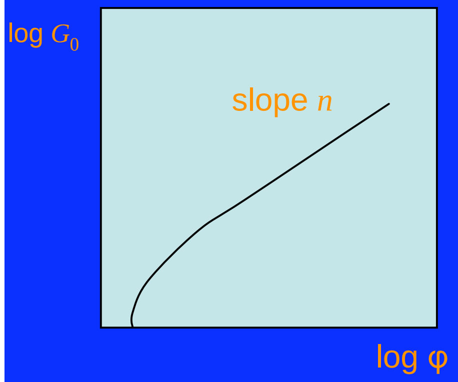
$$\tau_w(\varphi) = \int_0^{\gamma_c} G(x)\,\mathrm{d}\ln x \;\le\; \gamma_c G_0 \qquad p_y(\varphi) = \int_{\varphi_g}^{\varphi} K(x)\,\mathrm{d}\ln x = \frac{2}{3}\left(\frac{1-\nu}{1-2\nu}\right)\int_{\varphi_g}^{\varphi} G_0(x)\,\mathrm{d}\ln x$$

Whence,

$$1/S \;\ge\; \frac{2}{3\gamma_c}\left(\frac{1-\nu}{1-2\nu}\right)\int_{\varphi_g}^{\varphi}\frac{G_0(x)}{G_0(\varphi_0)}\,\mathrm{d}\ln x$$

e.g. if

$$G_0 \sim k\left(\left[\frac{\varphi}{\varphi_g}\right]^n - 1\right)$$



High concentration

$$1/S \ge \frac{p_y}{\tau_y} = \frac{2\left(1-\left[\dfrac{\varphi_g}{\varphi}\right]^n\right)\left(\dfrac{1-\nu}{1-2\nu}\right)}{3n\gamma_c} \simeq \frac{1}{n\gamma_c}$$

arXiv:0903.0970v1 [cond-mat.soft] (2009); J. non-Newtonian Fluid Mech. (submitted).

Some explicit predictions

The relationship between the compressional and shear strengths of poroelastic colloidal gels, R. Buscall, arXiv:0903.0970v1 [cond-mat.soft] (2009); J. non-Newtonian Fluid Mech. (submitted)

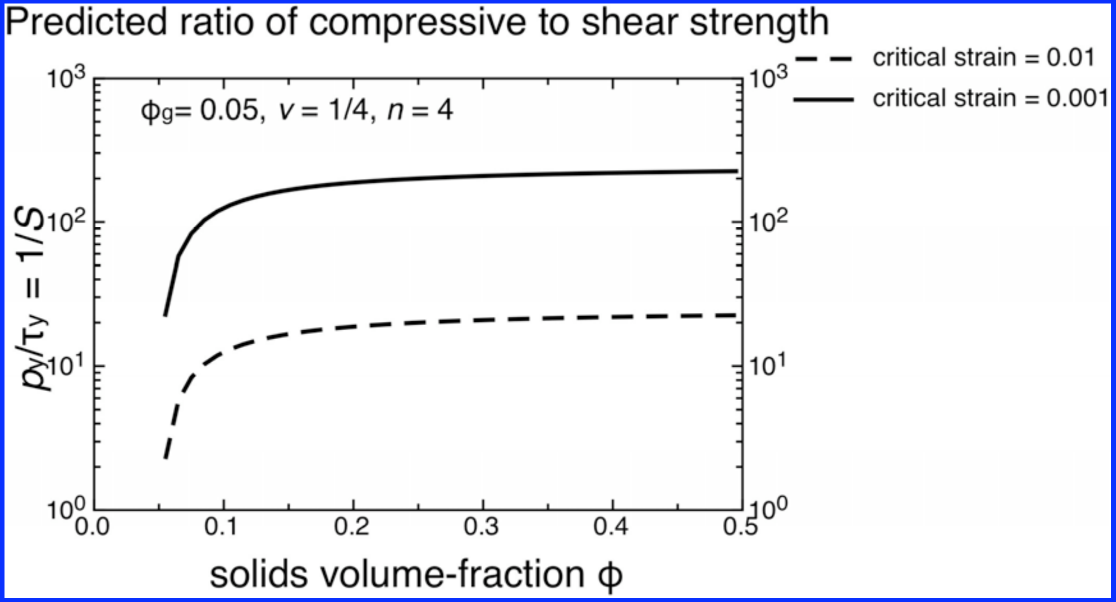


Fig. 6 Some predicted values for the strength ratio for two values of the critical shear strain. Otherwise, the gel-point, the poisson's ratio and the concentrationpower-law index have the values shown.

Data of Channell & Zukoski - α-alumina particles

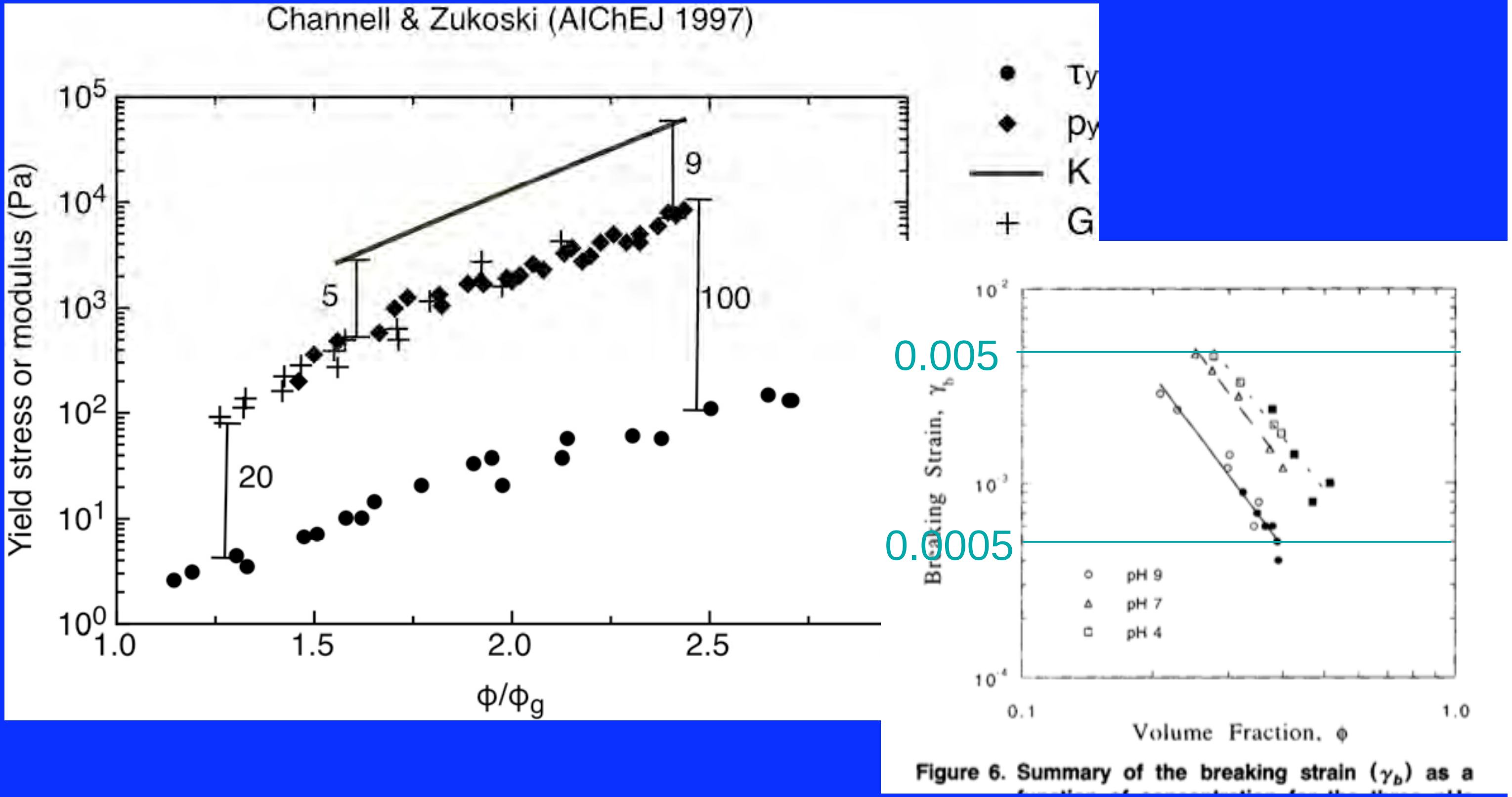


Figure 6. Summary of the breaking strain (γ_b) as a

Fig. 1 Plot of shear strength, τ_y, compressional strength, p_y, shear modulus, *G*, and compressional modulus, *K*, versus volume-fraction, normalised by that at the gel-point, for flocculated α-alumina particles – data taken from G. M. Channell and C. F. Zukoski, “Shear and Compressive Rheology of Aggregated Alumina Suspensions,” AIChE J., 43 (7), 1700-1708 (1997).

Data of Aziz et al. and Zhou et al. α-alumina particles

University of Melbourne

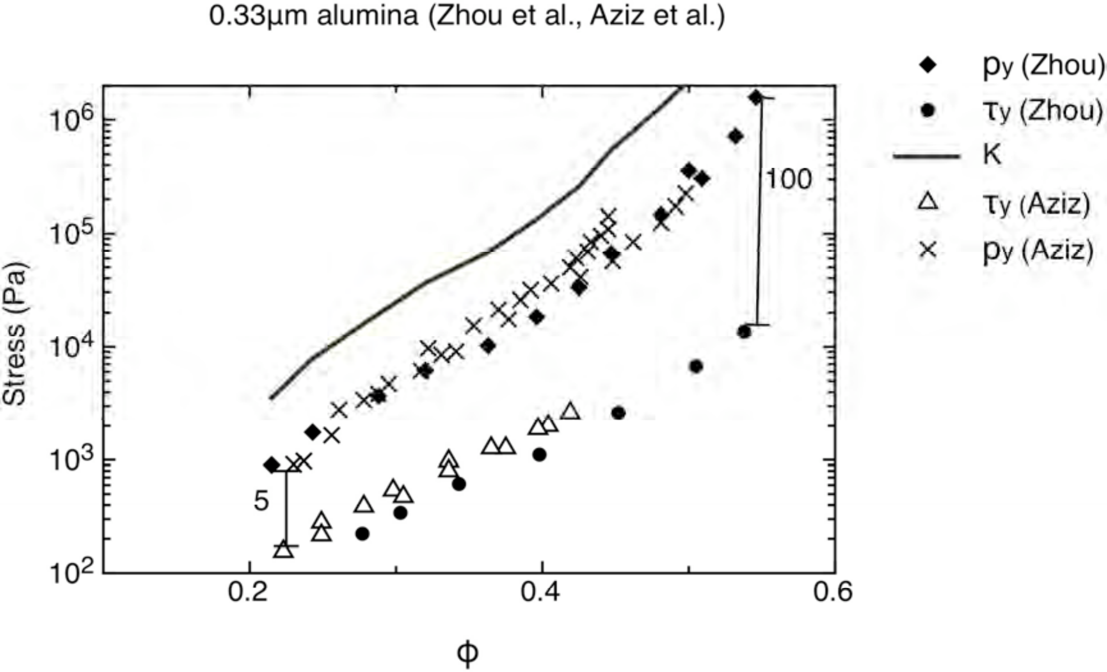


Fig. 2 Plot of shear strength, τ_y, compressional strength, p_y, and compressional modulus, K, versus volume-fraction, normalised by that at the gel-point, for flocculated α-alumina particles. Data from the Melbourne group [42-44]. Note the good agreement between two separate investigations. 42] Z Zhou, PJ Scales and DV Boger, "Chemical and physical control of the rheology of concentrated metal oxide suspensions," Chemical Engineering Science, 56: 2901-2920 (2001).

43] PJ Harbour, AAA Aziz, PJ Scales and DR Dixon, "Prediction of the dewatering of selected inorganic sludges," Water Science and Technology, 44 (10), 191, (2001).

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Data of Buscall et al. for coagulated PSL

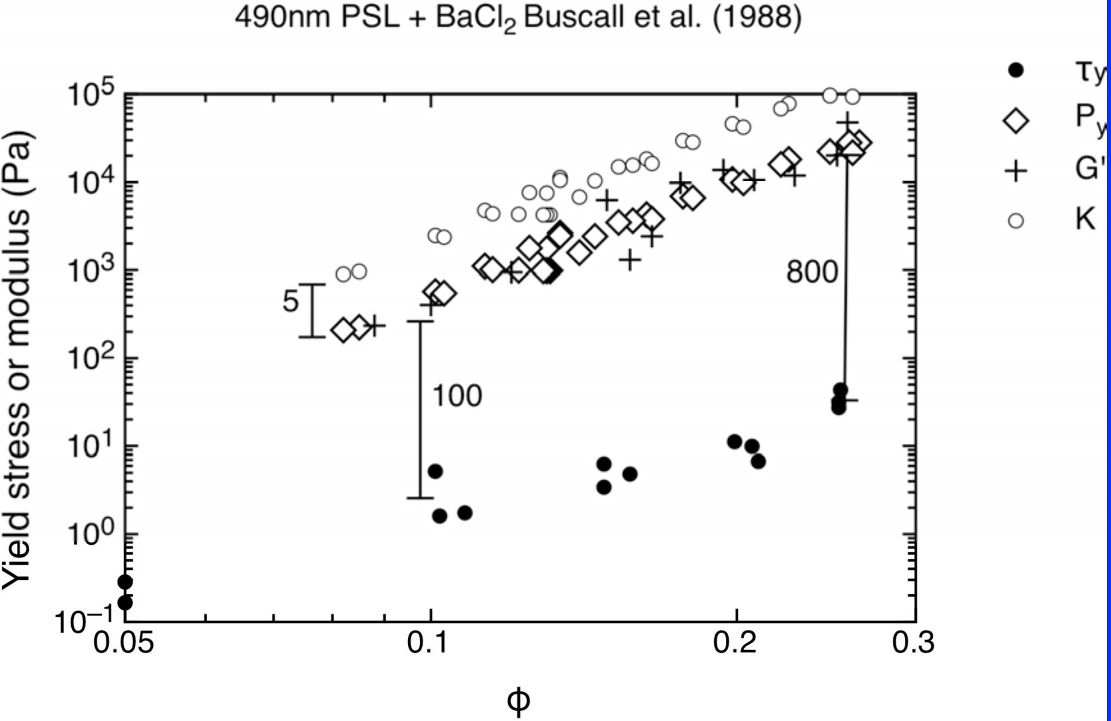


Fig. 3 Plot of shear strength, τ_y, compressional strength, p_y, shear modulus, *G*, and compressional modulus, *K*, versus volume-fraction for flocculated PSL particles – data taken from Buscall et al. [37, 40-41]. 37] R. Buscall, I.J. McGowan, P.D.A. Mills, R.F. Stewart, D. Sutton, L.R. White, G.E. Yates, The rheology of strongly-flocculated suspensions, J. Non-Newtonian Fluid Mech., 24(2) (1987) 183-202. 40] R. Buscall, P.D.A. Mills, G.E. Yates, Viscoelastic properties of strongly flocculated polystyrene latex dispersions, Colloids Surf., 18(2-4) (1986) 341-358. 41] R. Buscall, P.D.A. Mills, J.W. Goodwin, D.W. Lawson, Scaling behavior of the rheology of aggregate networks formed from colloidal particles, J. Chem. Soc. Faraday Trans. I, 84(12) (1988) 4249-4260.

n.b. critical shear strain is very small for PSL

Nano-silica at very low volume-fraction

$0.0025 < \varphi < 0.01$ - wall adhesion dominant

J-M. Condre, C. Ligoure and L. Cipelletti J. Stat. Mech. (2007) P02010 doi: 10.1088/1742-5468/2007/02/P02010.

$0.01 - < \varphi < 0.08$ - compression becoming important

S. Manley, J.M. Skotheim, L. Mahadevan, D.A. Weitz, Phys. Rev. Lett. 94, 218302 (2005).

See also The relationship between the compressional and shear strengths of poroelastic colloidal gels, Richard Buscall, arXiv:0903.0970v1 [cond-mat.soft] (2009).

Summary thus far

- Assumption of poro-elasticity leads directly to the conclusion that wall adhesion is only important very near the gel point (or for very narrow tubes).

- The data available for strong gels are consistent with this idea.

- More data near the gel point would be welcome.

Why be interested in wall (shear) versus compression?

1. If compressional and shear strengths were always similar, the world would be quite different.
2. The delayed collapse of weak gels does show wall effects…
3. A warm-up exercise for the tensorial rheology of particulate networks.

Weak gels

(The effect of fluctuations)

Weak gel formation - conceptual

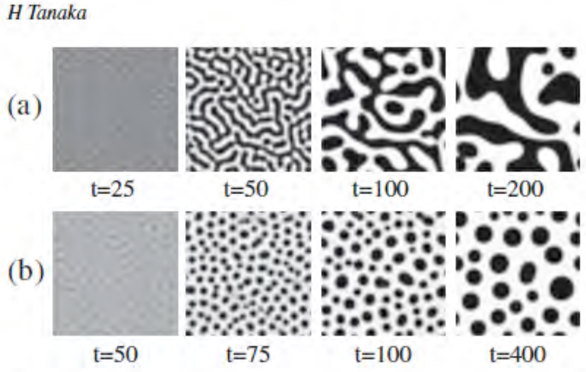


Figure 1. Typical phase-separation processes for (a) bicontinuous and (b) droplet spinodal decomposition in a two-dimensional fluid mixture. They are simulated by numerically solving the basic equations of model H. Time is scaled by the characteristic time τ.

The system would like to phase separate via spinodal decomposition, except that this is arrested by gel formation. The usual condition for the spinodal line, $\frac{d\Pi_{os}}{d\ln\varphi}=0$ is replaced by,

$$\frac{d\Pi_{os}}{d\ln\varphi}+K(\varphi,t)=0$$

where $K(\varphi, t)$ is the compressional modulus of the network or gel.

Coarsening by diffusive growth (particle hopping) is still possible and might be expected take place at a rate determined by the well-depth, as would creep due to self-weight.

H. Tanaka, Viscoelastic model of phase separation in colloidal suspensions and emulsions, Physical Review E Vol. 59, No. 6, pp. 6842-6852 (1999).

H.Tanaka, Viscoelastic phase separation, Journal of Physics: Condensed Matter Vol. 12, No. 15, pp. R207-R264 (2000).

Weak gels overview

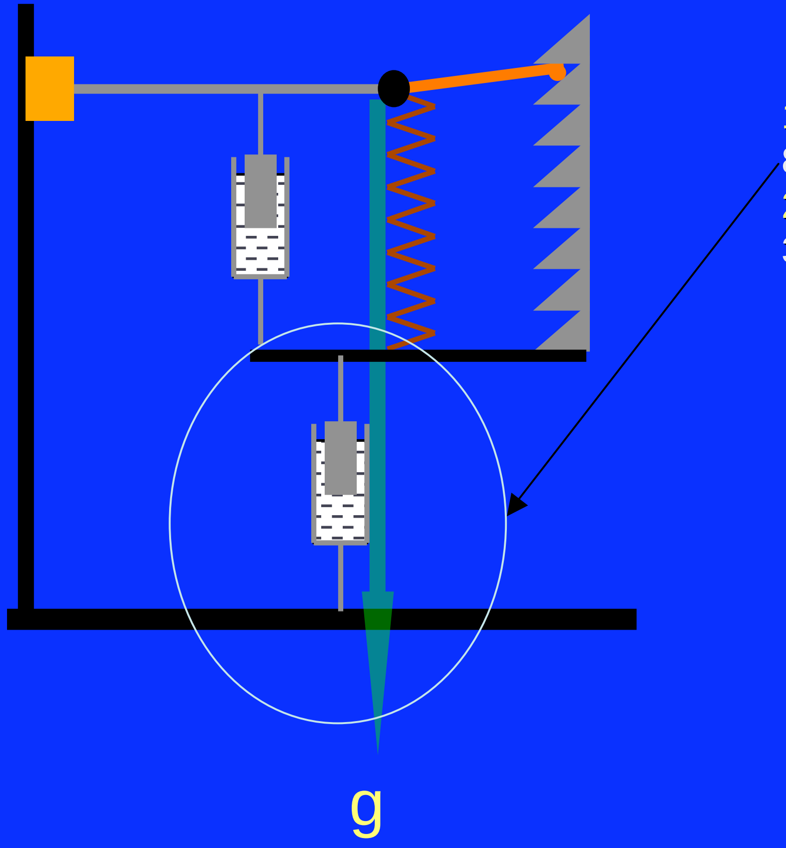
- Still have
  - fluid drag function $\Gamma(\varphi, t)$
  - compressive strength $p_y(\varphi, t)$
  - adhesive shear strength $\tau_w(\varphi, t)$
- Except that now these are going to change with time as a result of coarsening of the gel.
- And in addition there will be creep[1,2] and perhaps stress concentration and energy landscape effects.
  - creep can be modelled by adding a bulk viscosity[3]

---

1. L. Bergström, J. Chem. Soc. Faraday Trans. 1, 1992, 88, 3201;
2. Auzerais, F., R. Jackson, W. Russel and W. Murphy, J. Fluid Mech., 221, 613-639, 1990.
3. A.A. Potanin & W.B. Russel, Phys. Rev. E, 1996, 53 3702.

Poro-**visco**-elasticity

(N.b. all material coeff’s increase with -ve volumetric strain)



1. L. Bergström, J. Chem. Soc. Faraday Trans. 1, 1992, 88, 3201;
2. A.A. Potanin & W.B. Russel, Phys. Rev. E, 1996, 53 3702.

- Incorporating a “structural” viscosity accounts for creep.
- Not delayed collapse though.

Weak gels here

- Creep or delayed collapse?
  - either, or, or both?
- Comparison of the effect of well-depth and particle concentration two different dynamical properties
  - Viscosity versus delay time for delayed collapse

Delayed collapse

- Partridge et al. (Bristol) - 1982-85

S.J. Partridge, Ph.D. dissertation, School of Chemistry, University of Bristol, 1985.
A. Parker, P.A. Gunning, K. Ng & M.M. Robins, Food Hydrocolloids, 1999, 9, 333.
W. C. K. Poon, L. Starrs, S. P. Meeker, A. Moussaid, R. M. L. Evans, P. N. Pusey & M. M. Robins, Faraday Discuss., 1999, 112, 143; L. Starrs. W.C.K. Poon, D.J. Hibbert & M.M. Robins, J. Phys.: Condensed Matter, 2002, 14, 2485.
V. Gopalakrishnan, K.S. Schweizer and C.F. Zukoski, J. Phys.: Condens. Matter, 2006, 18, 11531.
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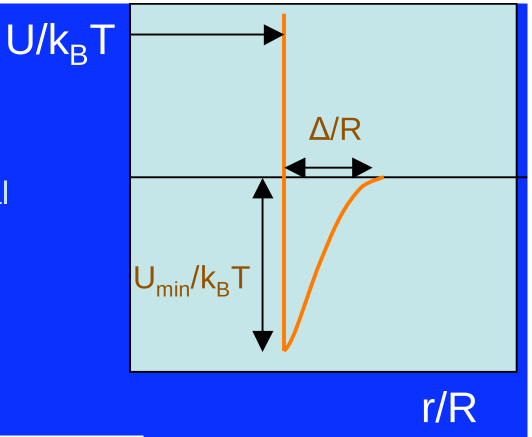
M.A. Faers, Advances in Colloid and Interface Science, 2003,106, 23-54.

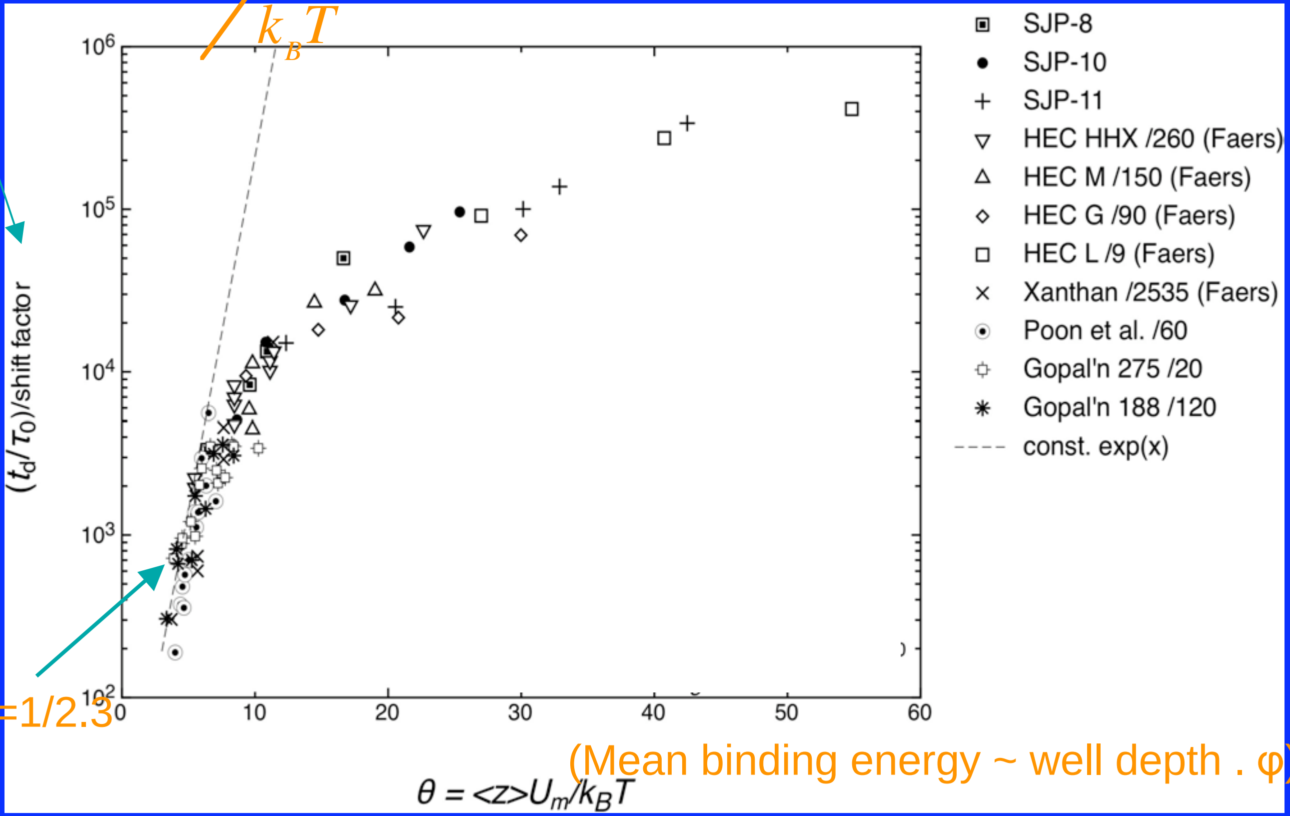
Key - Secondary minimum flocculation Depletion flocculation

Collapse is an activated process

Towards rationalising collapse times for the delayed sedimentation of weakly-aggregated colloidal gels, Richard Buscall, Tahsin H. Choudhury, Malcolm A. Faers, James W. Goodwin, Paul A. Luckham & Susan J. Partridge, Soft Matter (2009); DOI:10.1039/b805807e



$$\propto t_{delay} \cdot \frac{6\pi R\Delta^2\mu}{k_B T}$$

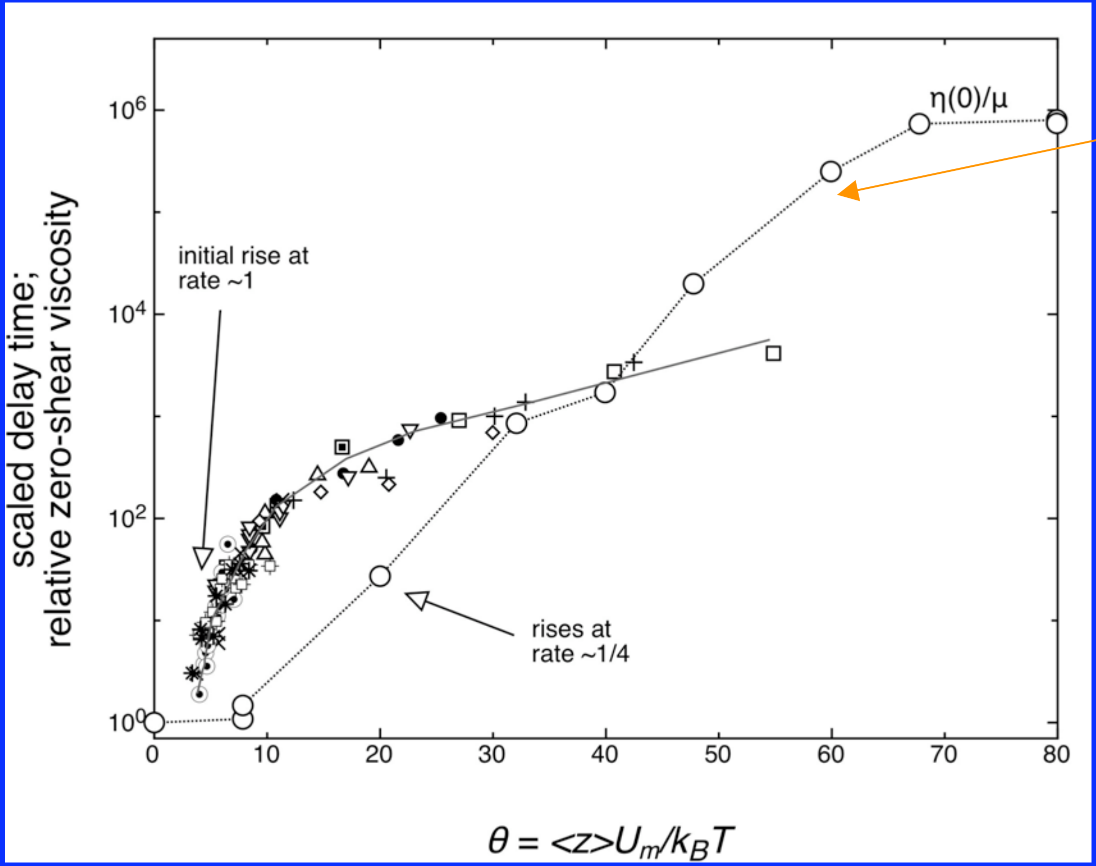


Initial slope =1/2.3

(Mean binding energy ~ well depth . φ)

Comparison of two rheological properties

The delay time and the ~~terminal relaxation time~~ zero shear viscosity



RLSV versus well-depth at constant $\varphi = 0.4$ (this constant <z>

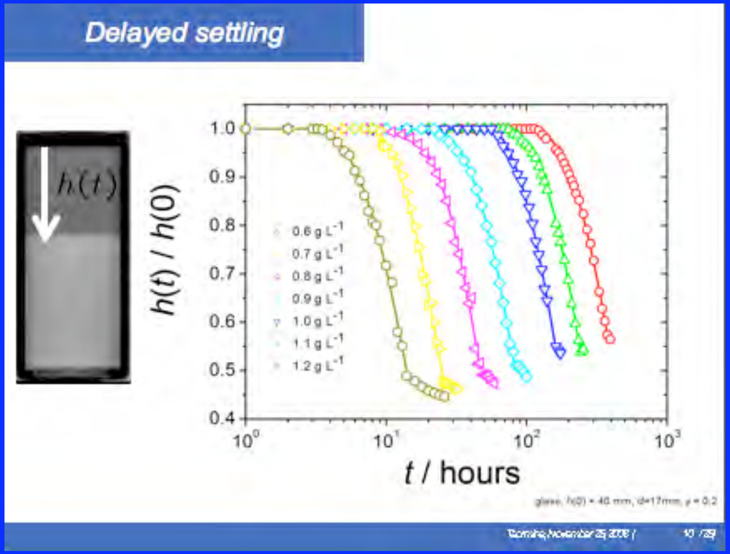
- Viscoelastic relaxation dominated by particles in very low z environments, whereas coarsening is somewhat more democratic
- Mean-field theory dead in the water (or dead in the suspension).

Collapse curves

Collapse curves

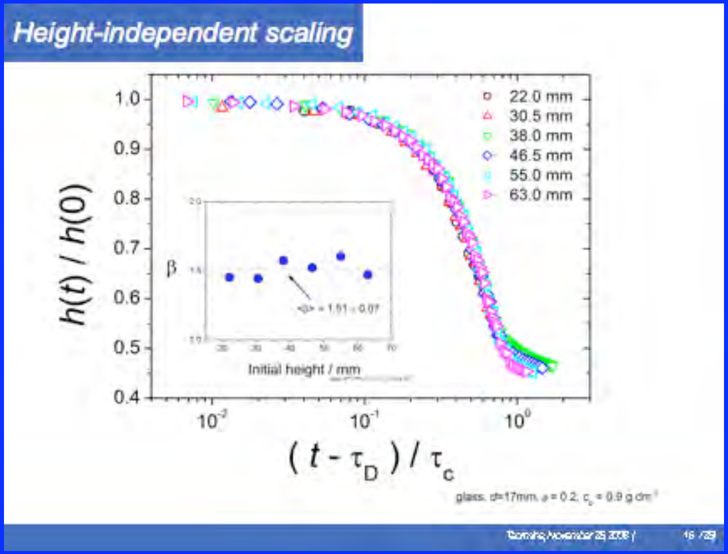
From Bristol

Teece, Faers & Bartlett 2008 - unpublished (vide infra).



Delayed settling

glass, $h(0) = 40$ mm, $d=17$mm, $\varphi = 0.2$



Height-independent scaling

glass, $d=17$mm, $\varphi = 0.2$; $c_c = 0.9$ g dm^{-3}

How do these compare with Partridge, Goodwin & Buscall 1985 - unpublished?

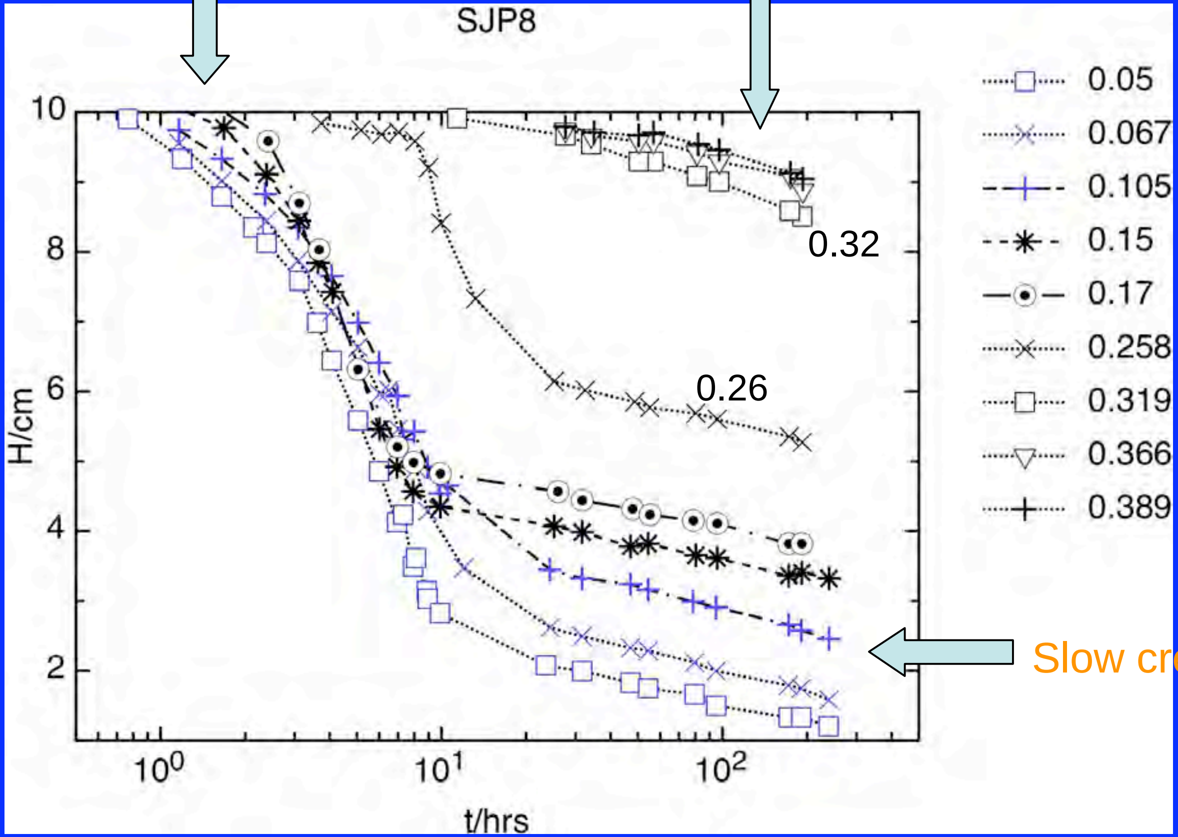
Sedimentation curves for SM flocc'd latex SJP8

R = 490 nm

$U_m/kT = 6$

Relative collapse rate becomes faster in the gel

Creep at constant rate

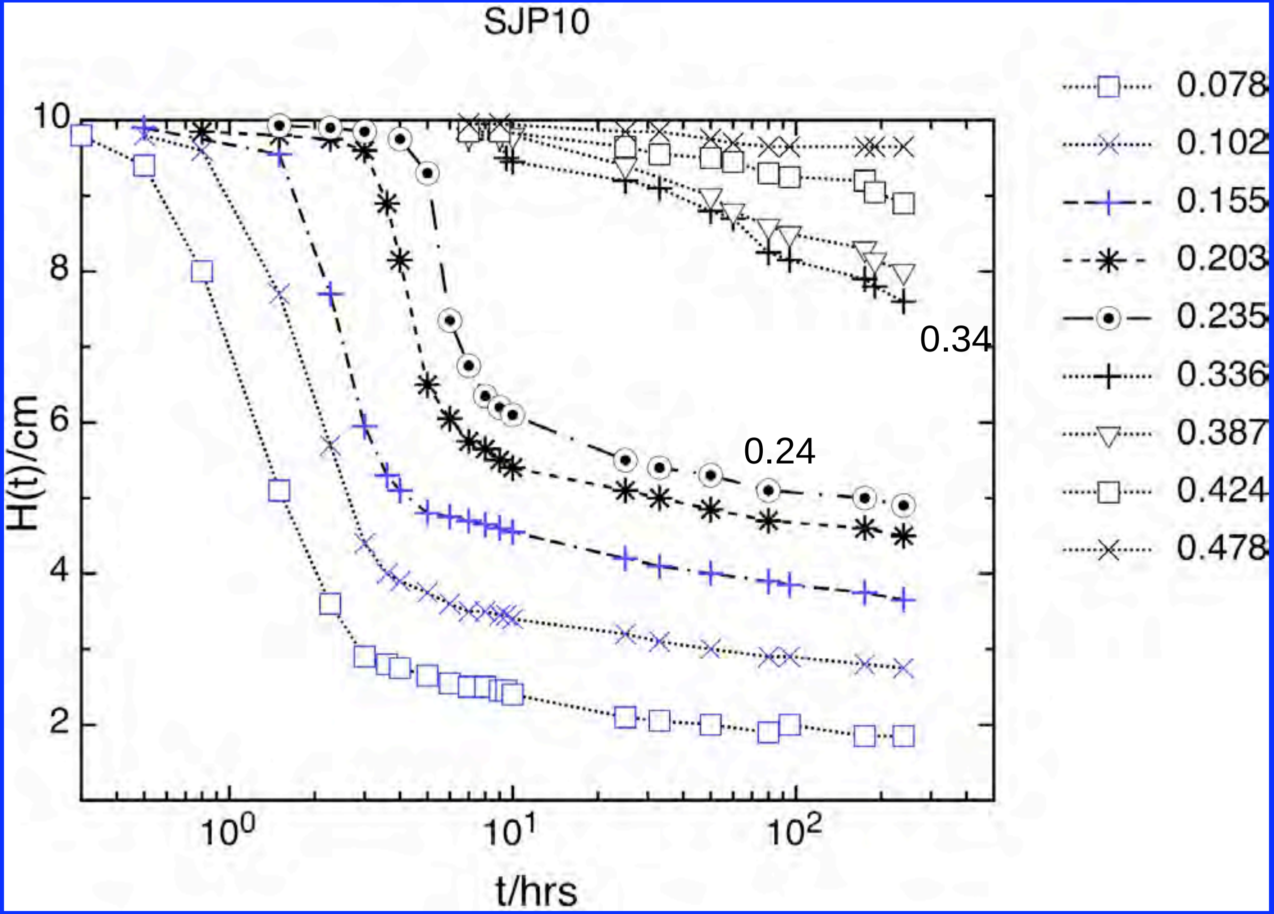


Slow creep at long times

Sedimentation curves for SM flocc’d latex SJP10

R = 710 nm

U_m/kT = 11

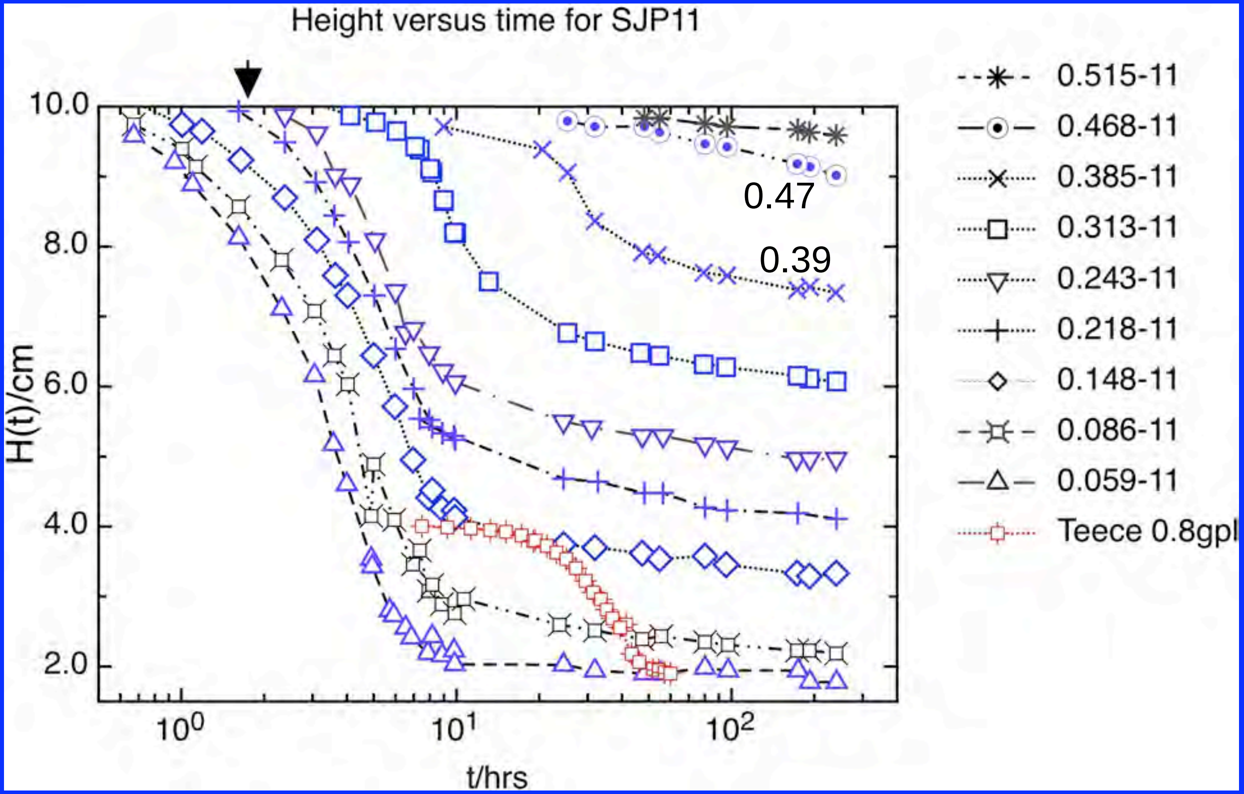


Similar except that the earlier curves are now spread because of the larger well-depth

Sedimentation curves for SM flocc'd latex SJP11

R = 960nm

U_m/kT = 14



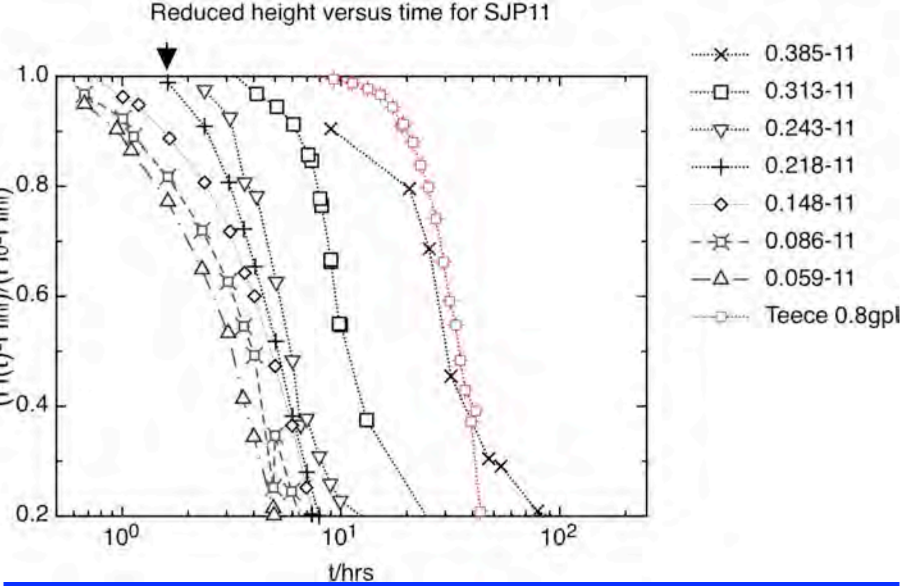
Similar except that the transition from enhanced collapse to simple creep is at higher volume-fraction

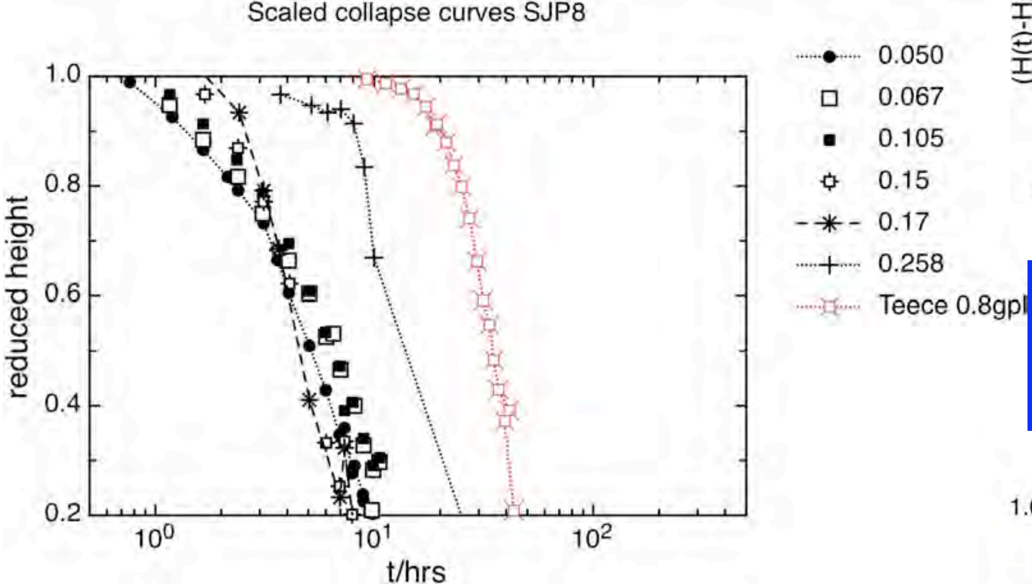
In order to compare the shape of the collapse curves we have to scale the heights fully

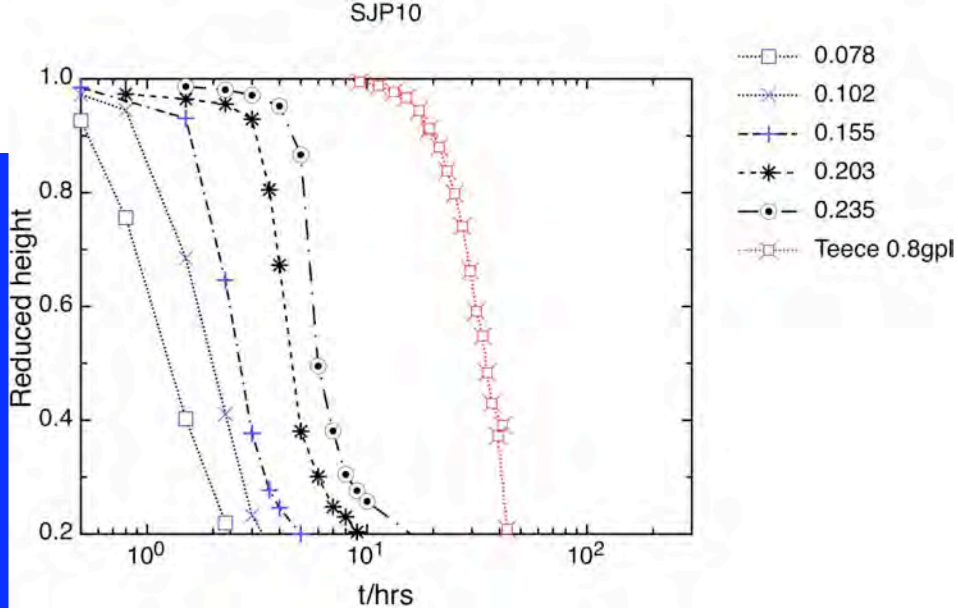
$$H_{reduced} = \frac{H(t) - H_{\infty}}{H_0 - H_{\infty}}$$

H_{∞} is estimated and the guess affects the long-time shape, not shown in the following.

Comparison of collapse curves of Partridge et al. (Bristol) with those of Bartlett & Teece (Bristol).


Reduced height versus time for SJP11


Scaled collapse curves SJP8


SJP10

Collapse curves summary

- Collapse time scales on delay time at intermediate φ in agreement with results of Bartlett & Teece.

- Creep is seen at high φ and post-collapse.

- Creep is generic, collapse is not.

Further detail

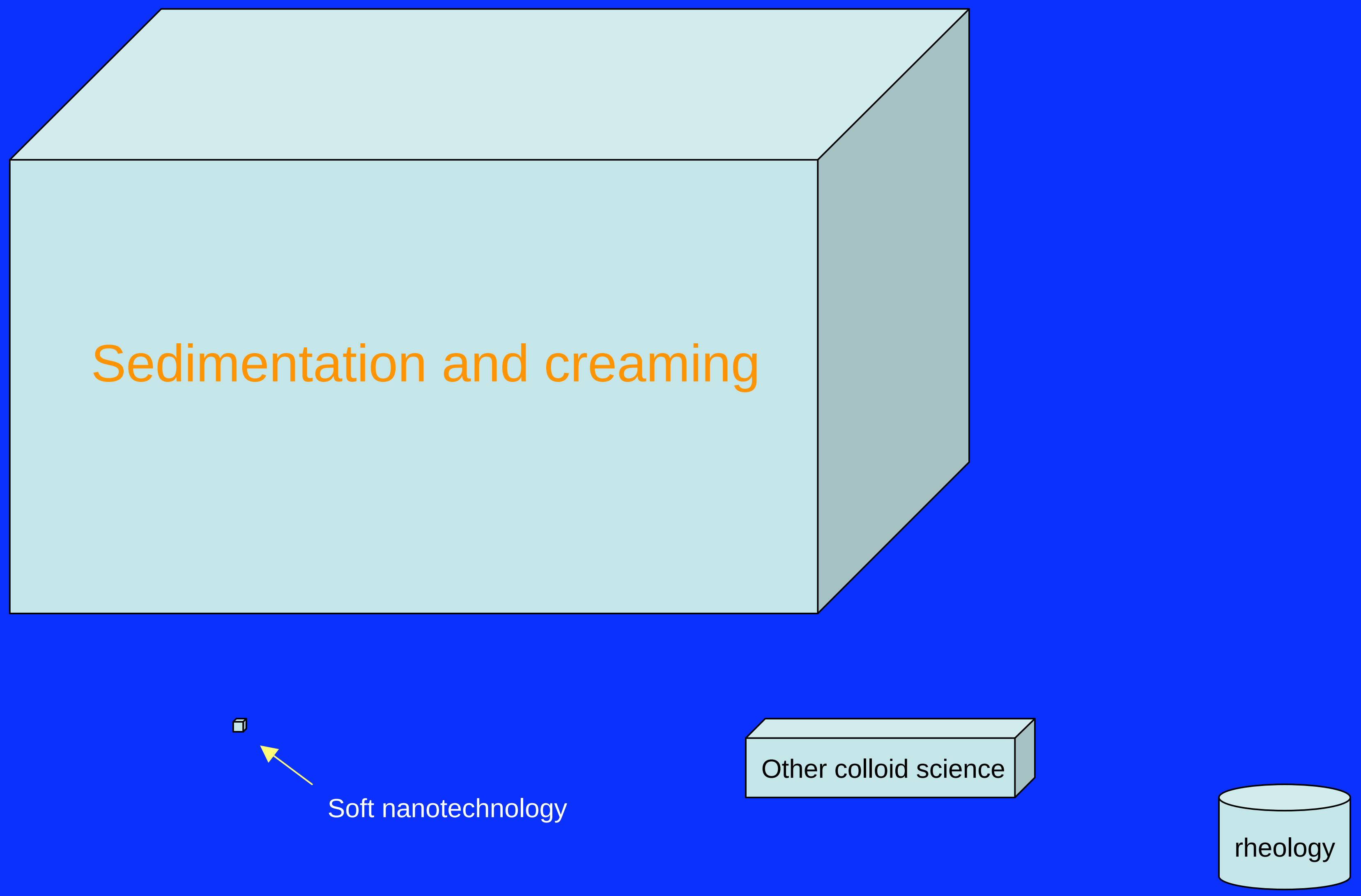
Towards rationalising collapse times for the delayed sedimentation of weakly-aggregated colloidal gels, Richard Buscall, Tahsin H. Choudhury, Malcom A. Faers, James W. Goodwin, Paul A. Luckham & Susan J. Partridge, Soft Matter (2009); **5**, 1345 - 1349 DOI:10.1039/b805807e

Whither compressional rheology?, Anthony D Stickland & Richard Buscall, J. non-Newtonian Fluid Mech., (2009) 157, 151–157.

The relationship between the compressional and shear strengths of poroelastic colloidal gels, Richard Buscall, arXiv:0903.0970v1 [cond-mat.soft] (2009).

Wall slip in dispersion rheometry, Richard Buscall, arXiv:0903.0265v1 [cond-mat.soft] (2009).

An objective view of research priorities



Sedimentation and creaming

Soft nanotechnology

Other colloid science

rheology

Acknowledgements

Variously for collaborations, data & discussions

Lee White, Jim Goodwin, Sue Partridge, Malcom Faers, Tahsin Choudhury, Peter Scales, Paul Bartlett, Lisa Teece, Paul Mills, Matt Lynch, Alan Parker, Rammile Ettelaie, Mike Evans …

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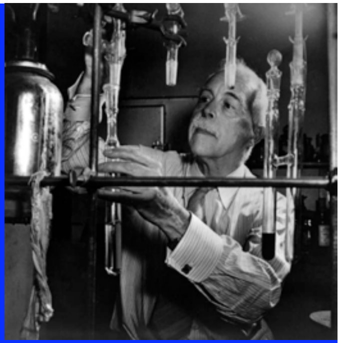
RB was funded by RB

H.E. Lawrence of the SCI

> “The fact is the physical chemists never use their eyes and are most lacking in chemical culture. It is essential to cast out from our midst, root and branch, this physical element and return to our laboratories.”

H.E. Lawrence, Chem. Ind. 1936, **14** 917.

More of H.E Lawrence of the SCI



Ten years before he had accused the great G.N. Lewis, whom he referred to as

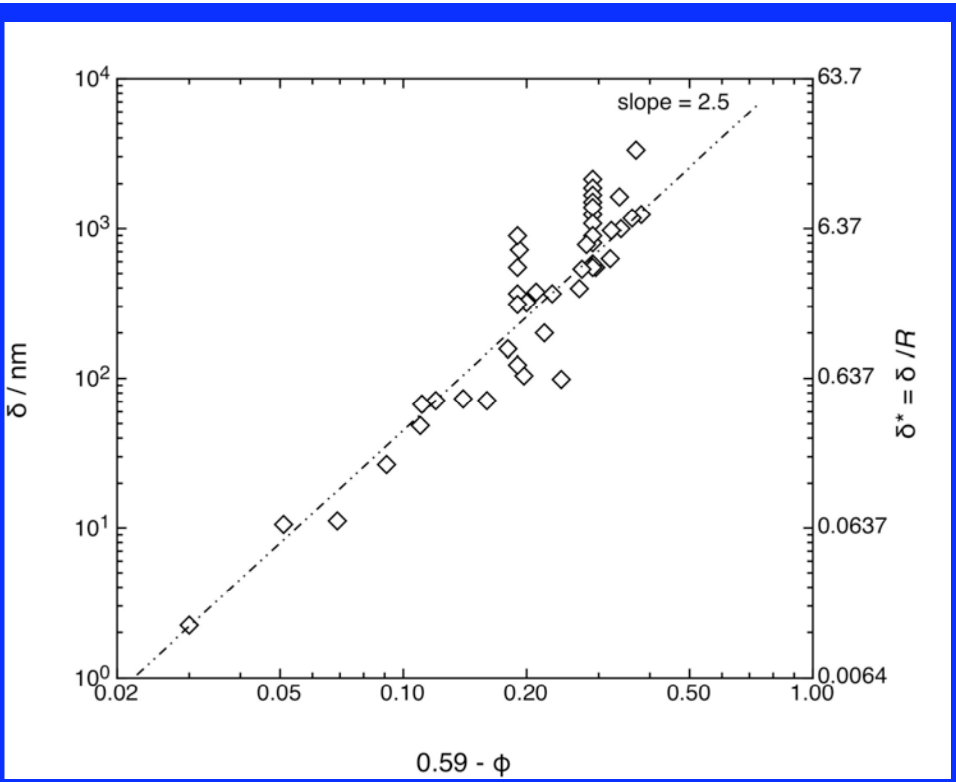
> “a Californian Thermodynamiter”,

of

> “…premature speculation upon the role of the electron as the cause of valency”.

Elasticity and wall slip

Slip layer thickness for weakly-flocculated suspensions versus volume fraction



Buscall, R., J. I. McGowan, and A. J. Morton-Jones, ''The rheology of concentrated dispersions of weakly-attracting colloidal particles with and without wall slip,'' J. Rheol. **37**, 621–641 (1993).

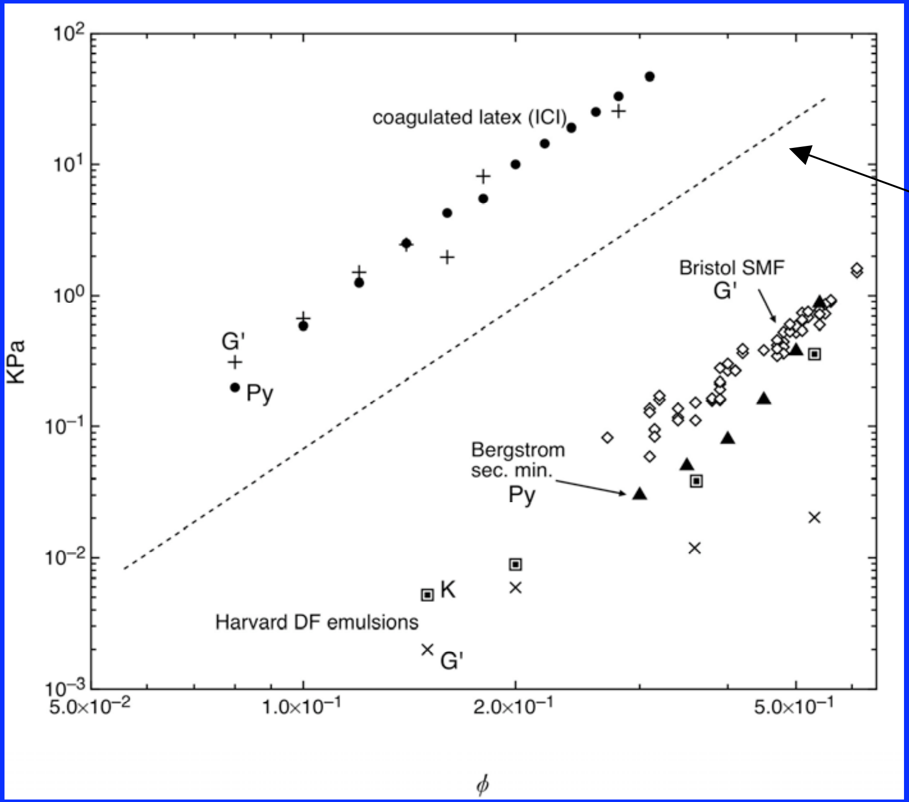
According to Seth et al. (2008), the δ is predicted to vary with the shear modulus of the particulate gel G like G-2/3, everything else being equal.

Note that in their model, Seth et al., used 1/G as a proxy for the compressibility of the particulate phase.

The exponent of 2.5 from fig. 1 then, should correspond to concentration exponents for the modulus and compressive strength of ca. 3.7

Seth, J. R., M. Cloitre and R.T. Bonnecaze, "Influence of short-range forces on wall-slip in microgel pastes", J. Rheol. **52**, 1241-1268 (2008).

Concentration dependence of plateau shear modulus



Modulus data are not available for the same suspensions, however a slope of 3.5 to 4.5 is typical of both weakly and strongly flocculated suspensions

The drawn line has a slope of 3.7, the slope implied by the slip layer data.