# The effect of alum dose on the consolidation behaviour of coagulated clay dispersions

David R. Dixon, Robert J. Eldridge, N. P. Le and Peter J. Scales

## ABSTRACT

Model sludges were prepared by coagulating clay dispersions (with or without added humate) with alum and/or a cationic polymer. The sludges were consolidated by centrifuging and their compressive yield stresses calculated as a function of solids content. Both the initial solids content and the final value after dewatering decreased with increasing alum:clay ratio. The adverse effect of alum dose on dewatering was evident even at the optimum dose for effective clarification and was more pronounced for humate-containing sludges. Poor dewatering was attributed to the low density of alum sludges at the gel point and to the high yield stress of networks formed at alum doses close to the charge neutralisation point. Dispersions coagulated with cationic polyacrylamide alone dewatered to higher solids than did alum sludges, but when added after alum, the polymer had no beneficial effect on ultimate solids content. The data confirms that the addition of polymer to alum sludges is beneficial to improving the rate of dewatering only.

**Key words** | alum, clay, coagulation, consolidation, dewatering, water treatment

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## INTRODUCTION

Inorganic coagulants remain the workhorse reagents for removing particulate impurities from raw water for potable utilisation, despite a number of disadvantages. Chief among these disadvantages are the low solids content and difficult dewaterability of the resulting sludge, especially in the case of alum coagulation (Bratby 1980). Coagulant demand can be quite low (a few milligrams per litre) if the raw water is low in colour, but increases strongly with increasing organic content. It is generally accepted that the problem of low sludge solids is worse at higher coagulant dose, but this effect has apparently not been quantified. In particular, it is unclear whether a slight overdose of coagulant has a significant effect on the volume of sludge and its properties. Therefore, the consequence of moving from charge neutralisation to sweep coagulation to enhanced coagulation on the achievable extent of dewatering requires more analysis.

The extent of dewatering or consolidation of sludges can be represented by the volume fraction ($\varphi$) of solid constituents. The rate of dewatering and achievable extent of dewatering are controlled by two sludge properties, respectively, the permeability and compressive yield stress, both of which are functions of the volume fraction of solids (Landman and White 1994; Scales *et al.* 1995; Johnson *et al.* 2000; Harbour *et al.* 2004). As inputs to models of dewatering, these parameters are now seeing wide industrial utilisation as quantitative measures of dewaterability (Landman and White 1997). This should be compared with the parameters that are indicative of sludge dewaterability (e.g. capillary suction time and specific resistance to filtration). These parameters generally extract information that relates to the rate rather than the equilibrium extent of dewaterability and are not typically measured as a function of the volume or weight fraction of solids.

The achievable extent of dewatering as a function of the applied pressure in filtration is relatively straight forward to measure but the experimental time is often lengthy. Indeed, times of the order of weeks are not uncommon at low pressures, although a new technique provides automated methods for pressures in excess of a few kPa (DeKretser *et al.* 2001). The output of the

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automated technique is material property information useful for the prediction of filtration dewatering experiments but even at a few kPa, the pressures are too high to characterise dewatering in sedimentation and thickening processes. Centrifuge techniques are more applicable for gravity driven consolidation processes.

The relationship between the yield stress of the sludge in shear and compression and commonly measured rheological parameters such as the viscosity has been measured by a number of workers, but usually for model particulate systems only (Zhou *et al.* 2001). The data shows that, for coagulated suspensions up to moderate solids concentrations (around 40 volume percent solids for ceramic materials), the relationship between shear viscosity (at a fixed shear rate), the shear yield stress and the compressive yield stress follows the same trends. At high solids fractions, the compressive yielding behaviour of particulate suspensions then deviates from the other two parameters. The other important outcome of these model studies is that the compressive yield stress is a material property that is independent of the method of measurement and, therefore, an ideal basis on which to compare the extent of dewatering of different sludges, materials and treatment options.

Measurements of the solids content before and after centrifugal dewatering, of model alum sludges as a function of alum dose is reported. Model sludges were prepared by coagulating clay dispersions with alum or, for comparison, a cationic polymer or alum-polymer combinations. For each of the sludges produced, we measured the extent of volume contraction on centrifuging to equilibrium at progressively increasing speeds was measured. At each centrifuge speed the sludge consolidates until its yield stress is equal to the compressive force acting on a unit cross-sectional area, after which the volume remains constant. Drying the solids after centrifuging and measuring the density then allowed the solids concentration to be calculated from the steady sludge volume reached at each centrifuge speed (Buscall 1982).

## MATERIALS AND METHODS

Sludges were prepared from dispersions of ball clay (which consists mainly of disordered kaolinite), with and without added humic acid, by coagulating with alum (aluminium sulfate) and/or cationic polyacrylamide. In all cases an initial clay concentration of 500 mg $l^{-1}$ was used, to provide a convenient volume of sludge. The ionic strength was adjusted to 0.001 M with NaCl. The clay (surface area approx 250 $m^2$ $g^{-1}$) was dispersed in high-purity water containing the calculated amount of NaCl by means of a Polytron PT10-35 ultrasonic mixer (Kinematica, Switzerland) and diluted to 500 mg $l^{-1}$.

When required, sodium humate (Aldrich) was added at 50 mg $l^{-1}$. One-litre aliquots were measured into square jars and coagulated with alum and/or Betz polymer 1158 at pH $5.8 \pm 0.2$ under standardised jar test conditions. After 2 minutes rapid mixing and 20 minutes slow mixing (7.5 cm × 2.5 cm rectangular paddles) the resulting flocs were allowed to settle.

Turbidities were measured with a Hach Ratio/XR turbidimeter and absorbances with a Shimadzu UV-160 spectrophotometer. Sludges were transferred to graduated flat-bottomed glass vials (14 mm or 23.5 mm internal diameter) and allowed to stand until a constant bed height was reached (the gel point). The samples were then centrifuged in a Sigma model 3-1 benchtop swing-bucket centrifuge (effective radius approx 13.5 cm) and the steady bed height noted at each speed. The time taken to reach equilibrium decreased from 1–2 weeks at low speed to 6–8 hours at 2,000 rpm. Angular speeds were measured with a tachometer. The solids were oven dried and weighed. The densities of the dry solids were measured by the density bottle method. The volume fraction, $\varphi$, of solids at each centrifuge speed was calculated from the mass, density and apparent volume data. The corresponding yield stress was computed from the compressive force at that speed using the method proposed by Buscall and & White (1987) and exemplified by Green *et al.* (1996).

For measurement of zeta potentials, larger sludge samples were made by upscaling the above procedure: 20 g of clay was dispersed in a few litres of water and the suspension diluted to 40 l (with the addition of NaCl and humate) in a 70 l plastic rubbish bin. The bin had ribbed walls that provided a measure of turbulence during coagulation. To provide sufficient sludge at low alum doses the procedure was repeated several times and the sediments pooled. A portion of each sludge was centrifuged as

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**Table 1** | Jar test results for clay/humate/alum systems

| $Al_2(SO_4)_3$ (mg $l^{-1}$) | alum/(clay+alum) (%) | $\Delta\tau$* (%) | $\Delta A_{254}$ (%) |
|---|---|---|---|
| 0** | 0 | 44, 44 | |
| 20 | 3.85 | 7, 5 | |
| 30 | 5.67 | 89, 43, 12, 12 | |
| 40 | 7.41 | 96, 96 | 93 |
| 50 | 9.09 | 96, 96 | 97 |
| 60 | 10.7 | 97, 98 | 98 |
| 80 | 13.8 | 98 | 99 |
| 100 | 16.7 | 98 | 97 |
| 120 | 19.4 | 99 | 99 |
| 300 | 37.5 | 99 | 99 |
| 400 | 44.4 | | |

*The first $\Delta\tau$ value at alum doses of 20–60 mg $l^{-1}$ was obtained in a 1-l jar test; all other turbidity and absorbance values are from 40-l experiments.
**6 mg $l^{-1}$ Betz 1158.

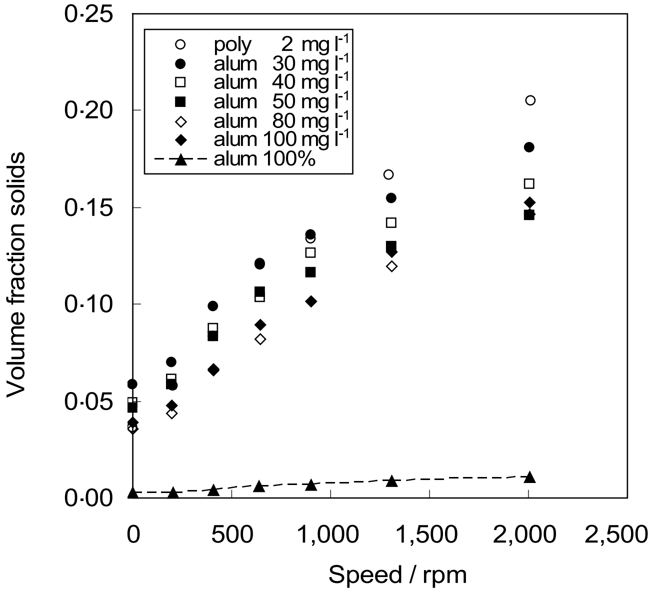


**Figure 1** | Centrifugal consolidation of clay dispersions coagulated with alum compared with clay coagulated with polymer and hydrolysed alum coagulated with polymer.

previously. Zeta potentials were determined on the remainder (200 ml samples, diluted where necessary) with an AcoustoSizer® (Colloidal Dynamics).

## RESULTS

### Jar test results

Coagulation of clay-humate dispersions was effective at alum doses above 40 mg $l^{-1}$, as gauged by the reduction in the turbidity $\tau$ and absorbance at 254 nm $A_{254}$ (Table 1). (Doses are expressed as anhydrous $Al_2(SO_4)_3$.) The percentage change in turbidity ($\Delta\tau$) was small at 20 mg $l^{-1}$ alum and erratic at 30 mg $l^{-1}$. However, the yield of sediment was high at all alum doses. This suggests that the residual turbidity was due mainly to colloidal $Al(OH)_3$ and/or Al-humate complexes rather than clay. In the absence of humate the clay dispersions were effectively coagulated at all alum doses investigated (30–100 mg $l^{-1}$).

### Effect of alum on solids content

Centrifuge results for humate-free sludges are shown in Figure 1. The 100% alum sample was produced by hydrolysing $Al_2(SO_4)_3$ in the absence of clay and flocculating the resultant $Al(OH)_3$ with cationic polyacrylamide. A zero-alum sample (clay flocculated with cationic polyacrylamide) is also included for comparison. The solids content at the gel point, $\varphi_{gel}$, decreases significantly as the alum dose increases in the 30–80 mg $l^{-1}$ range, but is still greater by an order of magnitude than for $Al(OH)_3$ flocs. The solids content at 2,000 rpm, $\varphi_\infty$, is also lower at high alum dose, but is always significantly higher than $\varphi_{gel}$. The polymer-only sludge was low in solids at the gel point, but reached the highest $\varphi$ after centrifuging.

The yield stresses shown in Figure 2 indicate that the clay sludges become exponentially more difficult to compress as the volume fraction approaches 20%. Hydrolysed alum has a very low solids content at the gel

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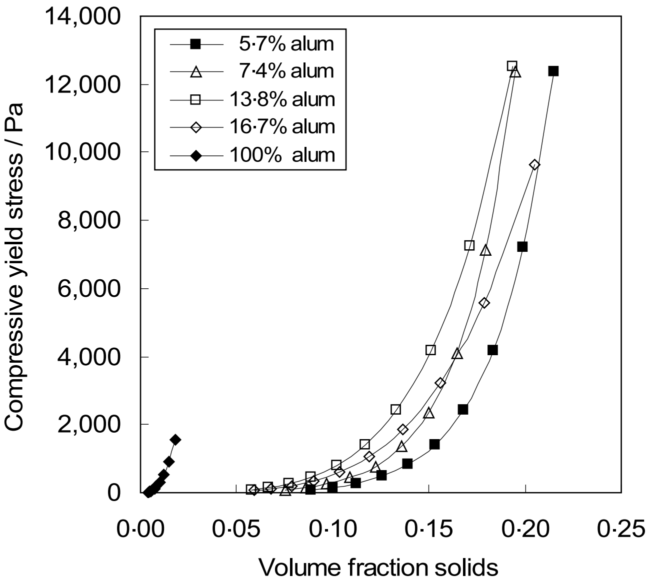


**Figure 2** | Compressive yield stress of coagulated clay dispersions as a function of volume fraction solids for different alum contents.

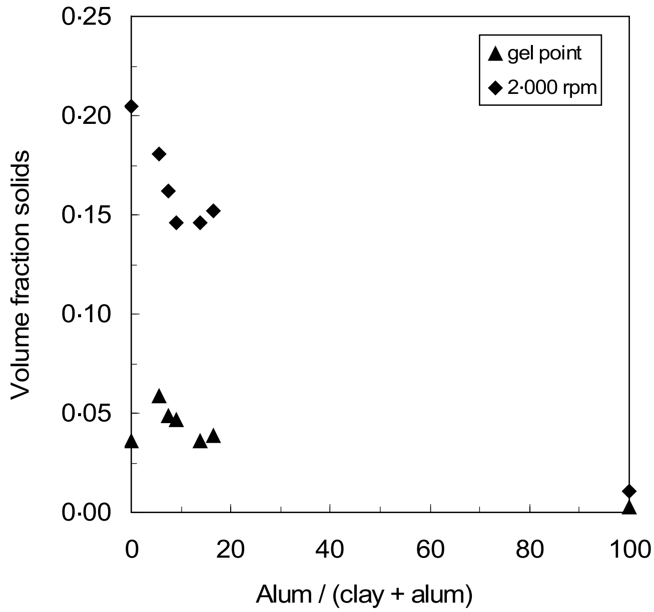


**Figure 3** | Effect of alum dose on solids content of the sludges in Figure 1 at the gel point and after centrifuging.

point ($\varphi_{gel} = 0.003$) and does not reach values greater than about 2% under the centrifugal conditions employed here. The failure to reach high compressive forces is due to its low density and the size of the precipitated particulate. Yield stress data for model suspensions show that the yield stress has an inverse squared dependence on the particle size (Zhou *et al*. 2001). A higher solids content would result at higher applied pressures (ie if a conventional pressure dewatering device was utilised) although this was not considered here (Harbour *et al*. 2004).

Figure 3 is a plot of $\varphi_{gel}$ and $\varphi_{\infty}$ against alum content, expressed as alum/(clay + alum) to enable the 0% and 100% points to be included. The zero-alum point is sensitive to the polymer dose; 2 mg $l^{-1}$ was chosen arbitrarily. $Al(OH)_3$ is essentially unaffected by polymer. The results clearly show that alum doses greater than about 6% (i.e. 6 g anhydrous $Al_2(SO_4)_3$ per 94 g clay) adversely affect sludge properties and dewatering behaviour.

## Effect of humate

Figures 4 and 5 show centrifuge results for clay-humate dispersions. For a given alum dose the humate-containing sludge is more voluminous than the clay-only sludge ($\varphi$ is lower), both at the gel point and after centrifuging. Yield stresses are higher, except at the highest volume fractions. It is also clear that dewatering becomes much more difficult as the alum dose increases, especially in the range 30–100 mg $l^{-1}$ (5.7–16.7% alum). In other words, the adverse effect of alum on sludge behaviour is already evident at the minimum dose needed for effective clarification. As the dose increases $\varphi_{gel}$ decreases and the yield stress for a given $\varphi$ increases.

## Effect of cationic polymer

The 40 mg $l^{-1}$ alum system was repeated with the addition of Betz 1158 at several doses. The polymer was added after the alum and before the settling step. There was no significant effect on $\varphi_{gel}$ at polymer doses up to 4 mg $l^{-1}$, while the effect on dewatering was if anything marginally disadvantageous, although polymer alone yields a more compact sludge than 40 mg $l^{-1}$ alum alone (Figure 6). (At least 6 mg $l^{-1}$ Betz 1158 was needed to generate a zero-alum sludge from clay-humate

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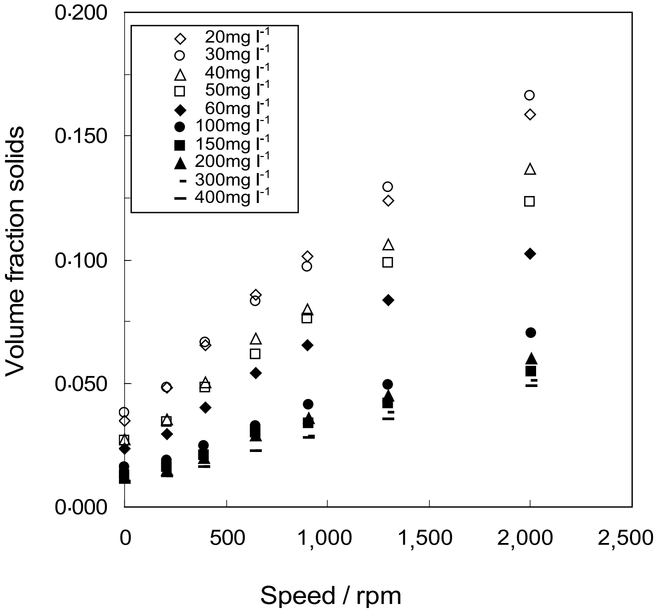


**Figure 4** | Centrifugal consolidation of clay-humate dispersions coagulated with alum. Addition rates are shown in the figure in mg $l^{-1}$.

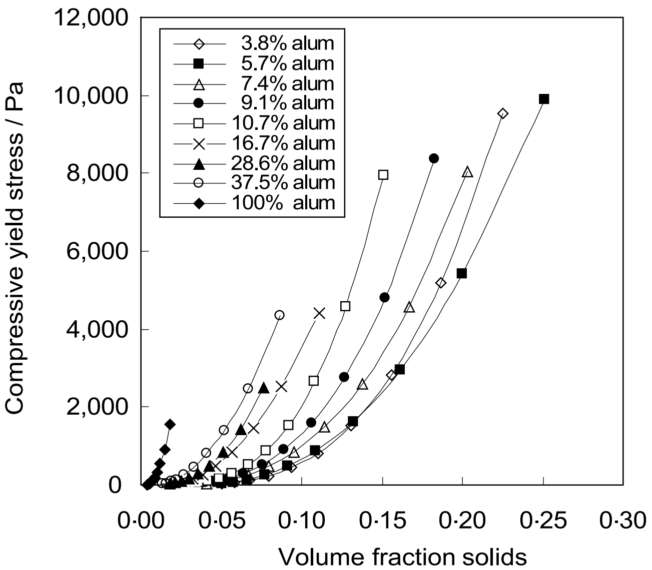


**Figure 5** | Compressive yield stress of coagulated clay-humate dispersions as a function of volume fraction for different alum contents.

dispersions. Even at 6 mg $l^{-1}$ the yield (recovery of clay to the sludge) was only 42%, increasing to 68% at 14 mg $l^{-1}$.)

Volume fractions for clay-humate-alum sludges at the gel point and 2,000 rpm are plotted against alum content

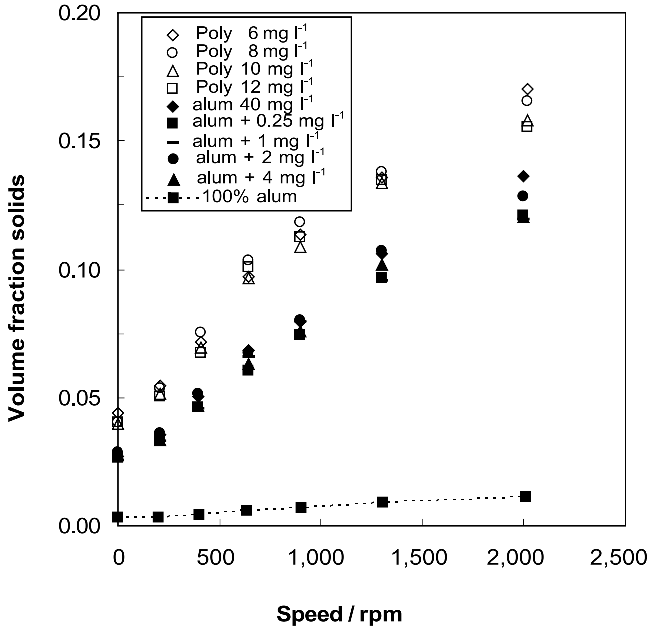


**Figure 6** | Centrifugal consolidation of clay-humate dispersions coagulated with 40 mg $l^{-1}$ alum plus 0–4 mg $l^{-1}$ polymer (closed symbols), compared with clay-humate coagulated with polymer alone (open symbols) and hydrolysed alum coagulated with polymer (dashed line).

in Figure 7, together with selected Betz 1158 results. This plot brings out the steep decline in final solids content with increasing alum dose. A dose of 40 mg $l^{-1}$ is needed for good turbidity and colour removal; the figure shows that dewatering has just begun to suffer at this dose (alum content = 7.4%). The critical dose range is the same as in the organics-free case (Figure 3), but the decline is to a much lower solids content. This suggests that Al(OH)3 complexed with humics is more troublesome than $Al(OH)_3$ alone. Again polymer-only sludges are seen to dewater to higher solids than alum sludges.

## Role of surface charge

Sludges from 40 l experiments were centrifuged and zeta potentials (approximate Smoluchowski values) at the natural pH measured in the AcoustoSizer®. Figure 8 shows (negative) zeta potentials as a function of alum content, overlaid on final volume fractions for sludges from both small-scale and 40 l jar tests. The $\varphi_\infty$ values for

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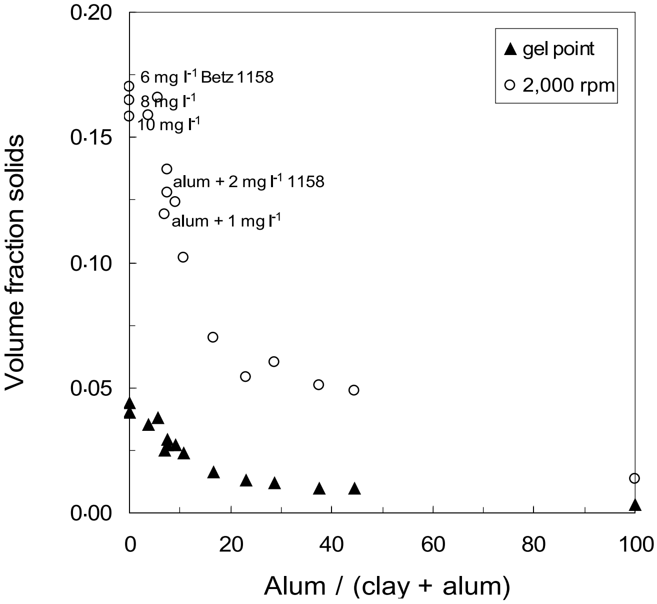


**Figure 7** | Effect of alum dose on solids content of clay-humate sludges at the gel point and after centrifuging.

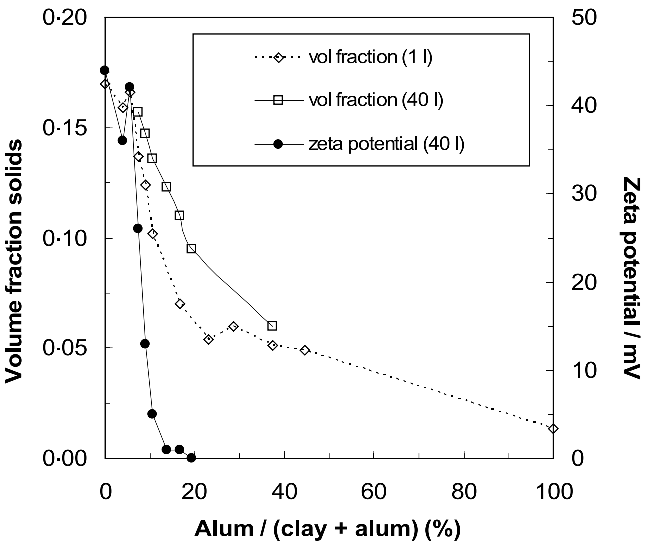


**Figure 8** | Dependence of surface potential and final solids volume fraction on alum content of clay-humate sludges.

the two series of experiments are in approximate agreement and show the same trend with alum dose. Zeta potentials are strongly negative up to quite high alum contents. Since $Al(OH)_3$ is positively charged at near-neutral pH this finding underlines the effect of organics on sludge properties. The zeta potential increases from about −40 mV to about −5 mV in the composition range where dewaterability declines severely, but the decline continues after neutrality is reached. (The erratic results at low alum dose can be attributed to incomplete removal of clay and, especially, organics in this range, making the sludge composition uncertain.)

## DISCUSSION

Inorganic coagulants clarify colloidal dispersions by two mechanisms, depending on the dose. Clarification at low dose is attributed to charge neutralisation (although minimum turbidity is often observed at a slightly lower dose than that corresponding to full neutralisation) and at high dose to enmeshment in metal hydroxide flocs ('sweep flocculation') (Gregory and Duan 2001). The solids content $\varphi_{gel}$ of the resulting sludge is low even at low dose (no more than 0.06 for the model sludges investigated here, usually closer to 0.005–0.01 for water plant sludges) and its consequent low density is one reason for the ineffectiveness of gravity or centrifugal dewatering. The higher coagulant doses typical of sweep flocculation lead to lower $\varphi_{gel}$ and lower density, exacerbating this effect. On the other hand, the yield stress of the sludge is proportional to the attractive force between particles, which is at a maximum in the absence of electrostatic repulsion (Scales *et al*. 1995; Johnson *et al*. 1998; Scales *et al*. 1998; Franks *et al*. 1998). Hence sludges produced under charge neutralisation conditions are inherently resistant to compressive dewatering. As discussed earlier, other factors such as the precipitate particle size also dominates yield stress behaviour but this is assumed to be a non-variable in this instance (Zhou *et al*. 2001). Water quality parameters such as hardness would also be expected to affect the sludge dewaterability but is not expected to dominate the observed trends.

Increasing alum dose adversely affects the initial solids content and centrifugal dewatering behaviour of model sludges (Figures 1–5). This effect can be attributed in part to the yield stress reaching high values at relatively low solids content (Figures 2, 5). The compressive yield stress

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of hydrolysed alum itself is relatively low; this material fails to consolidate because of its low density and small particulate dimensions. In other words, its dewatering is inhibited by its high initial water content. The increase in yield stress of coagulated clay with increasing alum dose is consistent with the findings of Harbour *et al.* (2002) on the yield behaviour of alumina dispersions dosed with $FeCl_3$. They found the *shear* yield stress to increase with $FeCl_3$:$Al_2O_3$ ratio (above a threshold value) and attributed this behaviour to an increase in fine particles, which strengthens the networked solids by increasing both the total surface area of the system and the number of inter-particle bonds in the network.

The effect of high alum dose on coagulated clay is greater for humate-containing sludges (compare Figures 3 and 7). Part of this effect occurs over the dose range in which the zeta potential changes from strongly negative to near-zero values (Figure 8), allowing van der Waals attraction between particles to create a semi-rigid network. Harbour *et al*. (2002) found the addition of humic matter to shift the isoelectric point (iep) of their dispersions, and consequently the maximum in a plot of yield stress against pH. The yield stress increased at any pH close to the new iep and decreased near the original iep because of electrostatic repulsion weakening the network. Consistent with this, our results show that, as an increase in alum:humate ratio in coagulated dispersions reduces the negative charge on the sludge particles at pH 5.8, the yield stress increases and the sludge becomes less compressible. The continuing decline in dewaterability with further increase in alum dose can be attributed to the decrease in $\varphi_{gel}$ and hence in density as the sludge composition approaches the limiting value represented by $Al(OH)_3$ (Figures 4, 5).

Dosing with a cationic polyelectrolyte after alum had no significant effect on $\varphi_{gel}$ and little or none on $\varphi_\infty$ (Figure 6). High molecular weight polymers have previously been shown to have no measurable effect on the compressive yield stress of flocculated dispersions (Johnson *et al.* 2000). Although a wide range of flocculants and dose rates were not employed here, the trends with increasing dose are clear. We conclude that the beneficial effect of cationic polymers in sludge conditioning is to improve permeability, and hence dewatering *rate*, rather than yield stress. On the other hand, clay or clay-humate dispersions coagulated with polymer alone consolidated to higher $\varphi_\infty$ than alum sludges. However, limited literature data on the dewatering of sludges produced through polymer coagulation (no alum or ferric added) indicates that the improvement in compressibility is matched by a decrease in suspension permeability. Therefore, coagulation with polymer alone does not lead to more tractable sludges (Harbour *et al*. 2004).

In practice, coagulant doses will always be selected to optimise clarification, not dewatering. In at least some cases the use of a cationic polymer in combination with alum allows a reduction in alum dose with no loss in clarification performance (Bolto *et al.* 1999). This appears to be an attractive option since the volume of sludge will be lower and the present results suggest that a reduced $Al(OH)_3$ content will result in the potential to dewater to higher solids.

## CONCLUSIONS

Alum sludges are low in solids at the gel point. The consequent low density and low particle size of the precipitate contributes to their poor dewaterability. With increasing alum dose dewaterability deteriorates further, both because $\varphi_{gel}$ decreases and because the compressive yield stress at a given $\varphi$ increases.

The deleterious effect of alum on the dewatering of model clay-humate sludges sets in at a dose of $Al_2(SO_4)_3$ equal to about 6–8% of their clay content, where the surface charge is becoming less negative. Dewaterability is already slightly below optimum at the alum dose giving optimum clarification, and deteriorates rapidly with overdosing as charge neutralisation leads to a stronger network.

Clay (with or without humate) coagulated with a cationic polyacrylamide dewaters to a higher ultimate solids content than clay coagulated with alum, but adding polymer after alum has little or no effect on $\varphi_{gel}$ or $\varphi_\infty$. Observations for the compressibility of ferric sludges are that this behaviour is typical of a range of metal oxide precipitates (Harbour *et al*. 2004) and that generically, once the method of coagulation has been chosen, the compressibility of the sludge has also been chosen.

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