Optimising polymer use in alum sludge conditioning: an *ad hoc* test

D. H. Bache and Y. Q. Zhao

ABSTRACT

The paper describes two test procedures for gaining insight into the optimum polymer dose for conditioning an alum sludge prior to dewatering. The first method centres on a development of theory used to estimate the specific resistance to filtration (SRF) using the standard Buchner funnel test. The second is an *ad hoc* test (termed the 'CML30 test') which provides a simple means for treatment works operatives to gauge the optimum dose; it focuses on a particular type of alum sludge and there are no claims concerning its wider utility. Standard filtration theory has been developed to take account of the hydraulic resistance of the filter medium arising from clogging phenomena in overdose conditions. The modified theory yields an adjusted filtration resistance, namely SRF_{adj}, which refers solely to the filtration behaviour of the cake. The CML30 test is based on 30 min settlement in a 100 ml measuring cylinder. With increasing dose, there exists a minimum interfacial height at a dose which coincides with the minimum in the SRF_{adj} trend; this is regarded as an optimum and shows proportionality with the solids concentration. Simplified theory is presented to explain the settling behaviour.

Key words | alum sludge, dewatering, dosage, polymer, settlement

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INTRODUCTION

Many test methods exist for identifying the optimum use of chemicals in sludge conditioning. The best known of these are the capillary suction time (CST) and the specific resistance to filtration (SRF). More recent approaches include streaming current analysis, rheological methods, and viscosity measurement (Dentel *et al.* 1998). Certain of these methods do not focus directly on the dewatering behaviour, but nevertheless serve as useful surrogates for following changes in the physicochemical character of the sludge in response to dose. At a fundamental level, the ability to measure polymer residuals (Keenan *et al.* 1998) and knowledge of the adsorption characteristics provides valuable supporting information for discerning the polymer interaction (Papavasilopoulos & Bache 1998). However, none of these tests can replicate the performance of the full-scale dewatering system and it is at this level that the 'technical optimum' must be judged. One of the difficulties with the laboratory scale test methods is that they demand specialised equipment and can be time-consuming in their execution. Experienced operatives often circumvent this problem by assessing the optimum dosage by 'the look' of a sludge. For example, as the dose is increased from zero, an alumino-humic sludge (gained from the coagulation of a low turbidity coloured water) transforms from the appearance of a fine-grained 'soup' to a coarse-grained solids structure which facilitates the rate of water release.

In this paper, the focus is on a simple sedimentation test as a rapid means of estimating the optimum dose of a polymer. Its purpose is to bridge the gap between the established approaches for identifying the optimum dose and the type of visual judgement exercised by operatives. In order to provide a datum against which to judge the efficacy of the approach, reference is made to the optima gained from a modified form of the SRF, hereafter referred to as the 'adjusted SRF' or SRF_{adj} which was described in
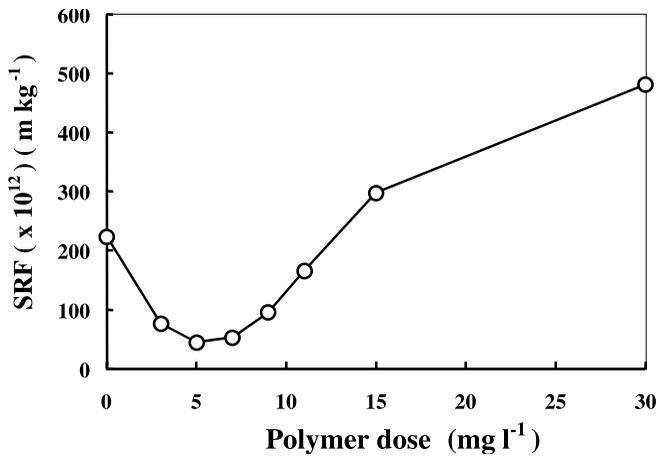
Figure 1 | Showing SRF trend (using Equation 7) for an alum sludge conditioned with Magnafloc LT25 (solids concentration: 3,100 mg l^{-1}) (from Zhao *et al.* 1998).

ADJUSTED SRF

Figure 1 illustrates a typical response of an alum sludge to increasing dosage of polymer. Salient features are the existence of the minimum and the increase in the SRF at high dosage. At high dosage the SRF can exceed that at zero dosage. This phenomenon was examined in Zhao *et al.*, the rising limb of the SRF being associated with polymer trapped on the filter medium. More specifically, it is caused by the blockages of the filter pores by agglomerated solids rather than by the polymer in isolation. In filtration theory (e.g. Coackley & Jones 1956), the hydraulic resistance offered by the filter medium should have no bearing on the evaluation of the SRF. SRF is a statement about the cake resistance and not of the filter medium. When severe clogging occurs, as in the case illustrated in Figure 1, the calculated SRF becomes distorted by the influence of the filter medium and conventional filtration theory needs to be modified in order

to distinguish between the separate components of resistance.

The Buchner funnel test (from which the SRF may be evaluated) rests on the analysis of the resistances and pressure differentials illustrated in Figure 2. From the Darcy equation, the rate of release of the filtrate volume (V) with time (t) can be written

$$\frac{1}{A}\frac{dV}{dt} = \frac{P_t}{\mu(R_c + R_m)} \tag{1}$$

in which A is the filtration area, P_t the total pressure drop and μ is the dynamic viscosity of the filtrate. Terms R_c and R_m refer to the total resistance per unit area. As is well known (see Figure 2), the total resistance can be subdivided into the cake resistance R_c and that of the support medium R_m. R_c is usually represented by rCV/A in which r is the specific resistance of the sludge cake (SRF) and C refers to the mass of dry suspended solids per unit volume of filtrate derived. This leads to the well-known form:

$$\frac{dV}{dt} = \frac{P_t A}{\mu(rCV/A + R_m)} \tag{2}$$

For the case in which there is no sludge cake, Equation 2 leads to the definition:

$$R_m = \frac{P_t A}{\mu\left(\dfrac{dV}{dt}\right)} \tag{3}$$

In Zhao *et al.* (1998), it was noted that R_m was well represented by the empirical form:

$$R_m = \alpha e^{\beta V} \tag{4}$$

in which α and β are treated as constants for a given set of initial conditions. Substituting Equation (4) in Equation (2), and integrating from initial condition $V = 0$ at $t = 0$ leads to:

$$\left[\frac{P_t A^2}{\mu}\frac{t}{V} - \frac{A\alpha}{\beta V}\left(e^{\beta V} - 1\right)\right] = \frac{1}{2}rCV \tag{5}$$
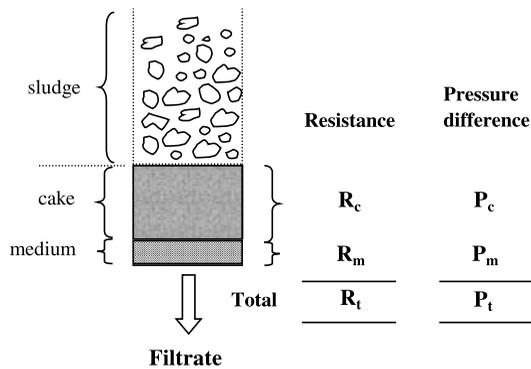
Figure 2 | Schematic representation of the basis of the SRF.

If the left hand side of Equation (5) is represented by the symbol Y (which does not involve the term r), this provides the simplified statement:

$$Y = \frac{1}{2} rCV \qquad (6)$$

Provided α and β are known, Y can be evaluated and the specific resistance r ($\equiv$SRF$_{adj}$) can be calculated from the slope of the Y versus V plot. It may be seen that when $\alpha = 0$, i.e. zero medium resistance, that Equation (5) reduces to the well-known form:

$$\frac{t}{V} = \frac{\mu rC}{2P_t A^2} V \qquad (7)$$

from which r ($\equiv$SRF) can be evaluated from the t/V versus V plot in the usual way. Following a practice advocated in Coackley & Jones (1956), parameter C can be represented by the initial solids concentration C_s, this providing an estimate of the dry solids per unit volume of liquid. Under this definition, one obtains the 'apparent' resistance to filtration rather than the 'true' specific resistance (see IWPC, 1981). The key to success in the SRF$_{adj}$ approach lies in the specification of R_m, as described in Zhao *et al.* (1998). Basically it involves separation of the supernatant following 30 min settlement and measuring the filtration of the supernatant through the same type of filter paper as used in the Buchner funnel test. Plots of the volume-time dependence may be fitted by the empirical equation

$$V = C_1 \ln t + C_2 \qquad (8)$$

with C_1 and C_2 as fitting coefficients; this provides a ready means of determining the gradient dV/dt ($= C_1/t$) which appears in the denominator of Equation (3). R_m may then be evaluated. At each point in time, V and R_m are known, thus allowing the construction of an R_m-V plot. In practice, the latter was fitted by Equation (4) using least squares analysis with α and β as fitting coefficients. This procedure must be applied at each dose. A more direct approach for evaluating α and β can be gained by differentiating Equation (8) (as indicated above) and substituting directly into Equation (4). By replacing the term 't' using Equation (8), one obtains the identities

$$\alpha = \frac{P_T A}{\mu C_1} \exp\left(-\frac{C_2}{C_1} \right) \qquad (9)$$

and

$$\beta = \frac{1}{C_1} \qquad (10)$$

EXPERIMENTAL MATERIALS AND METHODS

Tests were performed on waterworks sludge derived from the treatment of a low turbidity coloured water, using aluminium sulphate as the coagulant. The sludge was obtained from a sludge holding tank following primary clarification prior to conditioning and had a solids concentration in the range 3,090–9,230 mg l^{-1} and pH in the range 6.6–7.3. The sludge was conditioned with Magnafloc LT25 (Allied Colloids UK Ltd), which is an anionic organic polymer.

Polymer dosages in the range 3–30 mg l^{-1} were added to 200-ml sludge samples. Following polymer addition the sludge was subjected to 30 s of rapid mixing followed by 1 min slow mixing to promote flocculation.

Filtration tests were performed on 100 ml sludge samples in a Buchner funnel using standard procedures. In these tests, the pressure differential was maintained at 800 mm Hg and Whatman 1# filter paper was used as the filter medium. Further filtration tests were carried out on 100 ml samples of supernatant obtained after 30 min

**Box 1** │ Test procedure for CML30 settling test

1. Prepare polymer concentrate e.g. 100 mg l^{-1} and allow to stand for at least 24 h.
2. Measure sludge solids concentration.
3. Place 200 ml sludge sample in series of beakers (one per dose).
4. Add selected polymer dose to beaker with rapid stirring for 30 s for dispersion and 1 min slow stirring to support flocculation. *This part of the procedure can be varied as long as it is standardised across the samples and the polymer is dispersed.*
5. Repeat dosing and mixing for all beakers.
6. Allow suspension to settle and remove a volume of supernatant matching the volume of the polymer concentrate added during dosing. *This ensures that the sludge solids concentration is maintained at a fixed value; it is unlikely to distort the existing pattern of polymer adsorption because the residual polymer concentration is unaltered.*
7. Transfer 100 ml of dosed sample into 100 ml measuring cylinder. *Plastic measuring cylinders were used by author's group, but glass cylinders may also be used.*
8. In rapid succession, cover measuring cylinder with palm of hand, upturn and reverse allowing trapped air bubble to pass through suspension. *The purpose is to distribute the sludge solids throughout the column. In the interests of hygiene and safety, the hand should be protected.*
9. Allow to settle for 30 min and record height of interface.
10. Remove supernatant with pipette and measure moisture content of remainder. *This step is optional and is included as a check on the moisture content.*
11. Repeat if necessary using remaining 100 ml sample starting at step 7.
12. From knowledge of solids content evaluate polymer dosage on basis of mass ratio.

Figure 3 │ State of settlement at 30 min in response to dose in a 100 ml cylinder. The solids concentration was 4,595 mg l^{-1}.
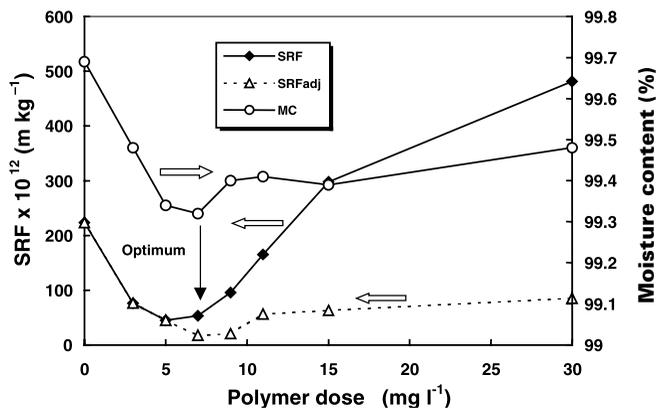

Figure 4 │ Trends of SRF, SRF$_{adj}$ and MC as a function of polymer dose at a solids concentration of 3,100 mg l^{-1}.

settlement in a measuring cylinder. As a general rule, all measurements were repeated three times and an average value is reported. Settlement tests (termed 'CML30') were performed on the remaining conditioned sample in a standard 100 ml plastic measuring cylinder. The settlement heights (at 30 min) were generally within ± 3% of the mean settlement in the underdose regime (see later text) and within ± 7% in the overdose regime In the interests of clarity, the procedures which were followed are summarised in Box 1.

The viscosity of the supernatant was measured using an Oswald viscometer (PSL Ltd, Wickford, UK) held in a constant temperature bath at 20°C.

RESULTS

Figure 3 illustrates the state of settlement in response to dose after 30 min for a solids concentration of 4,595 mg l^{-1}. It shows that the height of the interface passes through minimum as the dose increases from zero. It is this behaviour that has been translated into moisture content (MC) values in the plots shown in Figure 4. In common with similar plots at other solids concentration, it is seen that the minimum moisture content coincides with the dose at which there is a minimum in the SRF$_{adj}$ trend. By way of explanation of the SRF trends, the trend labelled SRF is based on the standard t/V versus V plot represented by Equation (7). At high dose, the estimates of
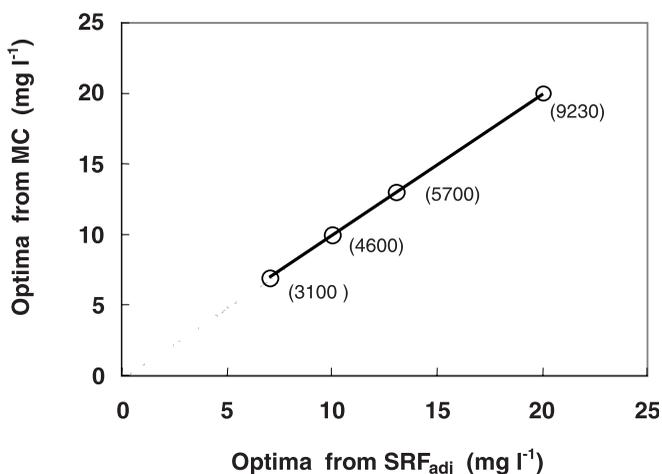
Figure 5 | Correlation between dosage optima for MC and SRF$_{adj}$; brackets show the solids concentration (mg l^{-1}).
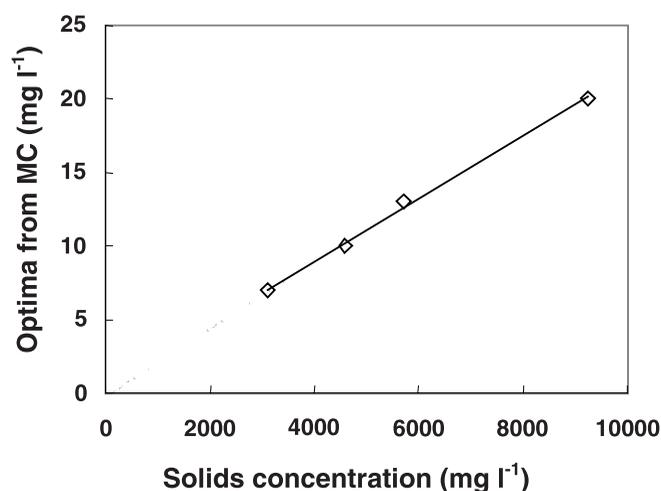
Figure 6 | Correlation between the MC optima and the solids concentration.

SRF become a little speculative in view of the increasingly non-linear behaviour in the t/V versus V plot as the dose is increased. The SRF$_{adj}$ is based on Equation (6) and takes account of the medium resistance gauged via the supernatant samples. The 'flatness' of the trends beyond the minimum SRF$_{adj}$ is indicative of significance of the medium resistance as reported in Zhao *et al.* (1998).

The correspondence between the minima identified in the settling test with those shown in the SRF$_{adj}$ plot (see Figure 5) suggests that the minimum after 30 min settlement in the 100 ml measuring cylinder might serve as a convenient surrogate for identifying the minimum in SRF$_{adj}$. Figure 6 indicates that the optima based on moisture content are proportional to the solids concentration. As with Figure 5, there is a high degree of correlation.

DISCUSSION

A crucial starting point in this analysis lies in the ability to define a datum against which to judge the utility of the sedimentation test. From the study reported in Papavasilopoulos & Bache (1998), it was evident that there were as many 'optima' as methods scrutinised, the SRF being representative and selected for this purpose.

However, as illustrated in Figure 1, the SRF value becomes distorted in the overdose range, this being a domain of interest in the settling test.

The semi-empirical theory outlined at the beginning of this paper provides a route for separating the medium and cake resistance in the domain of high polymer dosage. Compared with the conventional SRF value, the SRF$_{adj}$ is regarded as the more accurate representation of the cake resistance and its response to dose. At doses below the optimum, there is little difference between the SRF and SRF$_{adj}$ because there is no excess polymer. At doses in excess of the optimum or thereabouts, the polymer adsorption capacity of the solids is exceeded and the excess polymer remains in solution. It is this feature which drives the clogging of the filter medium (see Zhao *et al.* 1998); it also drives up the viscosity of the supernatant (see Figure 7; discussed later). As with the SRF, the SRF$_{adj}$ displays optimal features, but it is recognised that its existence may be an artefact of the method by which the SRF$_{adj}$ is estimated. In defence of the calculation of SRF$_{adj}$ (and thereby the associated trends), it must be pointed out that the evaluation of R_m was based on the observed resistance of the filter medium and can be regarded as a reasonably good estimate. This is indicated by the 'flatness' of the SRF$_{adj}$ plots at high dose when compared with the SRF in the same range.
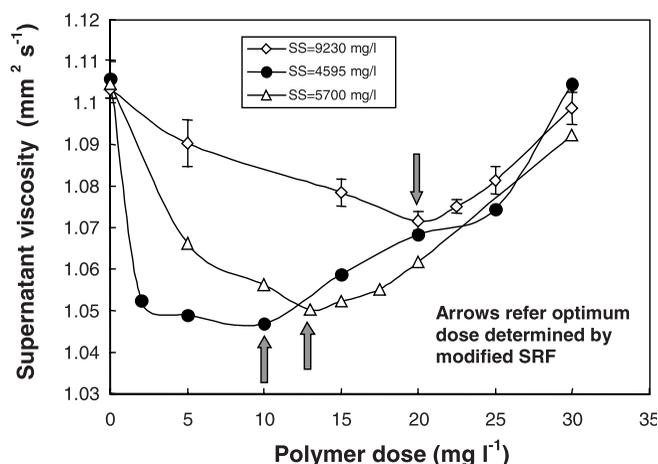
Figure 7 │ Response of supernatant viscosity (from settlement tests) to polymer dose at 20°C.

The plots shown in Figure 7 are of interest in their own right. Their main purpose was to demonstrate the presence of excess polymer, the latter causing increases in viscosity beyond the minima as the dose increases (Bache & Papavasilopoulos 2000). This is reinforced by the feature that the minima in the viscosity plots occur at the same dose as the minima in the corresponding SRF_{adj} plots. An explanation of the factors which control the shape of the plots is given in Bache & Papavasilopoulos (2000).

The correlation between the MC optima and the solids concentration shown in Figure 6 corresponds to a dosage of 2.1 kg tonne^{-1}. Measurements performed on sludges of similar type obtained from other treatment works display the same features; detail is provided in Table 1. Data

shown in the final column of Table 1 emphasise that the correlation between the settlement behaviour and the moisture release (as measured by SRF_{adj}) reflects a common behavioural trait among sludges of similar character. It is worth remarking that subsequent work (not reported in this paper) demonstrates that similar correlations exist between the CML30 data and minima in CST-dose plots. Thus a trend of the form shown by Figure 4 is not specific to SRF_{adj}, but reflects the underlying physicochemical condition of the sludge solids matrix with respect to water release.

Although trends in MC have been used as a reference parameter, these emanate from the settling heights as illustrated in Figure 3. For practical usage of the CML30 test, it is not necessary to measure the MC. Generally it is more convenient to identify the dose associated with the minimum settling height e.g. 10 mg l^{-1} in Figure 3. With knowledge of the dose and the solids concentration, an optimum dosage (i.e. kg t^{-1}) may be rapidly identified. This holds for a range of solids concentration. However, the height of the settled phase is bound to be influenced by the solids concentration on the grounds that the initial solids concentration affects the settlement velocity (see Kynch 1952). Figure 8 shows a plot in which five CML30 tests have been amalgamated for solids concentrations in the range 3,000–6,000 mg l^{-1}. The upper and lower limits refer to the mean ± SD. Best settlement occurs in the range 1.0–1.8 kg t^{-1}. The points refer to floc samples taken from the launder outlet in the thickener. These were associated with a variable solids concentration at fixed input of polymer; they show a greater sensitivity between the settled volume and the dosage than apparent in the
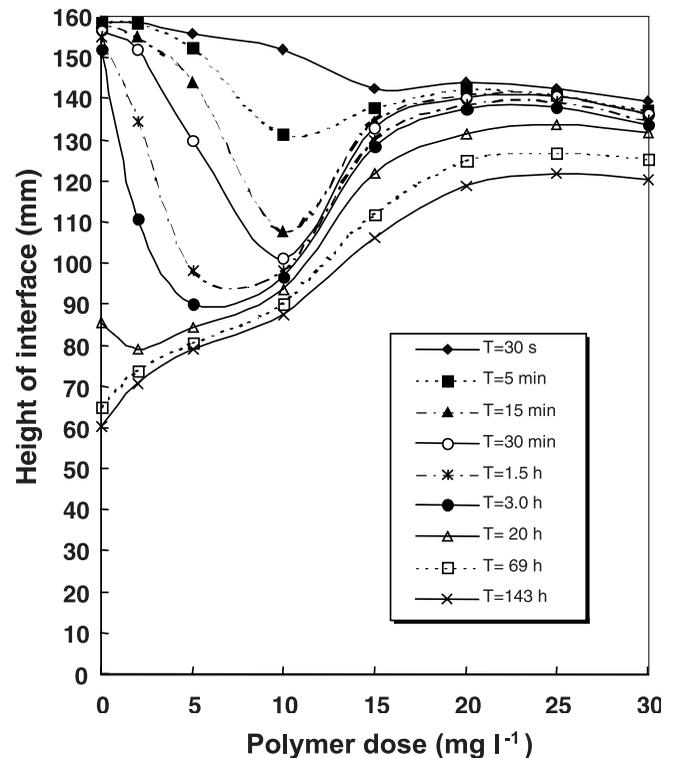
Table 1 │ Illustrating correspondence between optimised dose values based on MC and SRF_{adj} for sludges from different treatment works and the polymers which are in use

Works	Polymer	Solids concentration (mg l^{-1})	Optimum dose (kg t^{-1})	Dose ratio for MC_{min}: $SRF_{adj,min}$
A	Magnafloc LT25	3100–9200	2.1	1.00
B	Posifloc PW85	4100	2.4	0.95
C	Posifloc PW85	5700	1.8	0.91
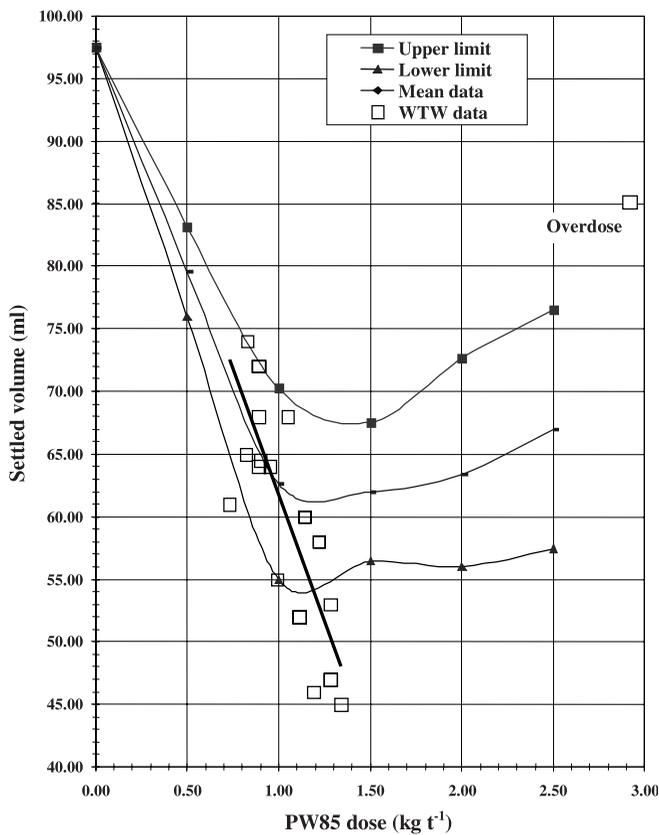
Figure 8 │ Trends represent the mean and range of settlement arising from standardised CML30 tests in response to dose for sludges with solids in the range 3,000–6,000 mg l^{-1}. The data points refer to the settlement of samples taken from a thickener.
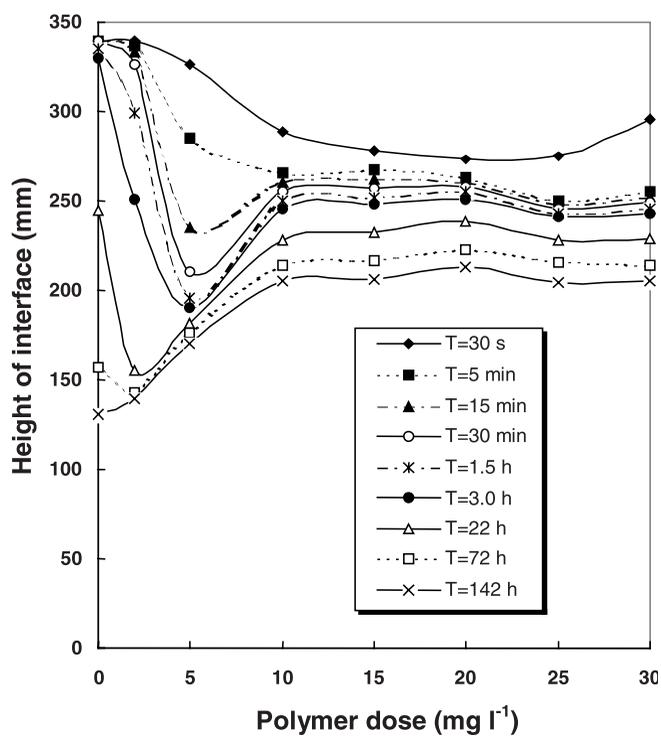

Figure 9 │ Showing response interface height to time and dose for a sludge of 4,440 mg l^{-1} solids concentration in a 100 ml measuring cylinder.

bulked data. Below 1.0 kg t^{-1}, there is good agreement between the trends in the bulked samples and the data points. However, at higher dosages (in the range 1.0–1.5 kg t^{-1}), the flocs drawn from the thickening tanks settled to a lower level than indicated by the bulked data. Such differences probably arise from scale effects (affecting the hydrodynamics) and differences in floc character moulded by their previous shear history; the shear history is known to affect the CST and thereby the character of the sludge (IWPC 1981). In the CML30 test the shear history was 'standardised' by use of the procedures summarised in Box 1, whereas at full-scale, the shear history is moulded by local factors. In the case of CST testing, techniques have evolved to simulate the on site shear conditions prior to CST measurement. The same philosophy may apply to

the sludge preparation techniques used in this study, but this aspect has not been tested. The overall message to be drawn from Figure 8 is that the CML30 test provides useful insight into the behaviour of the thickener as the dose is varied. It demonstrates very clearly that underdosing inhibits settlement. It is also capable of showing the symptoms of overdosing (see Figure 8): the sludge shows poor settlement in the CML30 test and indeed in the thickener itself in one case of high dosage.

In order to gain a deeper understanding of the mechanisms leading to the pattern of behaviour shown in Figure 3, further settling tests were carried out at a fixed solids concentration in 100, 500 and 1,000 ml measuring cylinders from very early times up to 143 h of settlement. Trends in the interfacial height for the 100 ml and 1,000 ml measuring cylinders are illustrated in Figures 9 and 10. In Figure 9 one can observe the development of the minimum in the interface at 10 mg l^{-1} in the time interval 5–30 min. At longer times the position of the

Figure 10 | Showing response interface height to time and dose for a sludge of 4,440 mg l^{-1} solids concentration in a 1,000 ml measuring cylinder.

minimum occurs at lower doses. Similar features are evident in Figure 10. However, it is also evident that there is significant difference in the position of the minimum; this is attributed to pronounced wall effects in the 100 ml cylinder. A schematic explanation of the trends shown in Figures 9 and 10 is provided in the Appendix. With regard to this explanation (which concerns the interaction of the settling phase with the settled layer), it is emphasised that the principles which are introduced are essentially correct, but the equations used to express the dynamics are inadequate in view of the complexity of the process.

The coincidence between the maximum MC (i.e. the minimum interface height) and the minimum in the SRF$_{adj}$ as illustrated in Figure 5 appears to be unique to the 100 ml cylinder for times in the range 5–30 min (see Figure 9). As such, it is a physicochemical quirk that the coincidence occurs. For this reason, the test is described as 'ad hoc'. However, it has been called the 'CML30 test'

in recognition of the fact that it applies to a 100 ml measuring cylinder and 30 min settlement. On the assumption that the SRF$_{adj}$ is a valid indicator of the optimum dose, the CML30 test appears to provide treatment works operatives with a simple method for checking dosages for conditioning.

The methodology outlined in Box 1 provides a record of the procedures adopted by the authors and has not been subjected to detailed scrutiny. In addition, it is emphasised that the test method has been designed for ease of use, and use of simple equipment. The test focuses on a particular type of alum sludge and there are no claims concerning its wider utility.

CONCLUSIONS

- There is good agreement between the *ad hoc* test, namely the 'CML30 test' and SRF$_{adj}$ data in terms of specifying an optimum dose for alum sludges gained from the treatment of low alkalinity and low turbidity coloured waters.
- The *ad hoc* test appears to be specific to the settlement in a 100 ml measuring cylinder in the time interval 5–30 min.
- The CML30 test provides operatives at a treatment works with a simple test using basic equipment for checking the optimum polymer dose for sludge conditioning.
- Schematic theory suggests that the appearance of the minimum in the settlement behaviour in response to dose and time is controlled by the response of the free settling phase to dose, coupled with the structural features of the settled phase.
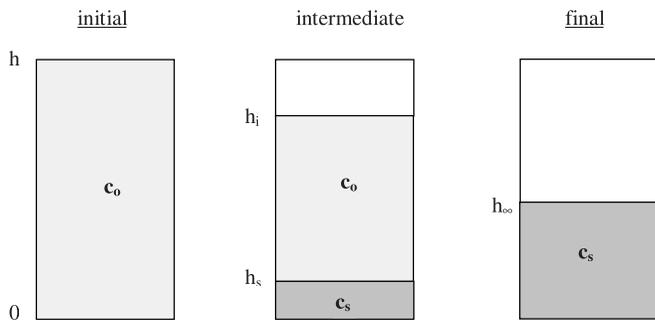
ACKNOWLEDGEMENTS

Figure 11 | Schematic view of settling behaviour.

APPENDIX. EXPLANATION OF SETTLING BEHAVIOUR

In this section a schematic analysis of the phenomena leading to the trends shown in Figures 9 and 10 is described. At the outset it is recognised that the explanation is over-simplified. Nevertheless, it addresses the principal processes which are taking place. Referring to Figure 11, it is assumed that the concentration within the settling phase (c_o) and the settled phase (c_s) are quasi-uniform. At any instant prior to completion of the settlement, the loss of mass is balanced by the gain in mass in the settled layer, that is:

$$(h - h_i)c_o = h_s(c_s - c_o) \qquad (11)$$

At completion of settlement, it is useful to express this in the form:

$$h_\infty c_s = h_o c_o \qquad (12)$$

Substituting Equation (12) into Equation (11) leads to

$$h_s = (h - h_i)/(h/h_\infty - 1) \qquad (13)$$

Inspection of Figure 9 shows that when the dose exceeds about 20 mg l^{-1}, the height is insensitive to dose. This is because the solids have been observed to have a finite adsorption capacity for the polymer (Papavasilopoulos & Bache 1998). Once the saturation capacity is exceeded, excess polymer remains in the liquid phase increasing its viscosity (see Figure 8) and the solids are characterised by

a single state commensurate with polymer saturation. In this state the 'flocs' are relatively large and form a fairly open network structure. Through time, compressive settlement takes place and supernatant is released. At the extreme of zero dose and after an extensive period of settlement, h_∞ occurs at a much lower level. This indicates that the flocs (which are much smaller) are able to pack together more efficiently and form a network with a higher solids concentration. So it is seen that h_∞ is a function of dose (up to the point of saturation adsorption) and can be roughly modelled by

$$h_\infty = 75 + 2.5D \quad \text{mm} \quad 0{\leq}D{\leq}20 \text{ mg l}^{-1} \qquad (14)$$

The second major feature of the settlement process is the sensitivity of the 'free settlement phase' to dose. In this context 'free settlement' is interpreted as settlement which is unaffected by the behaviour of the settled phase. At low doses (say $<$10 mg l^{-1}) and short settlement times (say $<$30 min) it is seen that the speed of the interface increases with increasing dose. However, at later times (as $h_i{\rightarrow}h_\infty$), the rate of settlement slows, presumably because the solids become increasingly networked as they approach the gel point (represented by h_∞). At early times, plots show that the interface velocity is slow to begin with and then speeds up (presumed to be caused by flocculation during settlement). Overall, it suffices to state that the settlement process is extremely complex. A very rough representation of the average interface settlement speed in the 'free settlement' phase can be gained from

$$v_i = 3.5 + 10D \text{ cm h}^{-1} \quad 0{\leq}D{\leq}15 \text{ mg l}^{-1} \qquad (15)$$

and

$$v_i{\approx}230 \text{ cm h}^{-1} \quad D{>}15 \text{ mg l}^{-1} \qquad (16)$$

What has been described can be viewed as a downward propagating wavefront (associated with h_i) and an upward propagating wavefront (associated with h_s). If one ignores the effects of compressive settlement, settlement ceases when the wavefronts meet, i.e. $h_i = h_s = h_\infty$. At earlier times, the upper interface is defined by:

$$h_i = h - v_i t \quad t \leq h_\infty / v_i \tag{17}$$

In principle, this set of equations can be used to map the response of the h_i to dose and are capable of generating the type of behaviour shown in Figure 9. The greatest difficulty lies in the specification of the interfacial velocity, particularly in the domain $h_i \rightarrow h_\infty$. A more refined theory would include features to describe compressive settlement, this aspect having been specifically excluded in order to focus on the principal mechanisms leading to the formation of the minimum.

For any time (exceeding 30 s), it is apparent that the minimum arises from the pattern of settlement in response to dose. It lies in a transition region between 'free settlement' at low doses and 'completed' settlement (including wall effects) at higher doses. As time progresses, the dose at which the minimum occurs drifts towards zero dose, because longer settling times allow the settlement to go to completion at a lower dose. In Figure 9, it is of interest to note that the time of 30 min represents the greatest time for which a minimum exists at 10 mg l^{-1}, settlement (or $h_i \approx h_\infty$) being virtually complete at this dose and time.

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