Validation of a New Filtration Technique for Dewaterability Characterization

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Characterization of the compressibility and permeability of flocculated suspensions is a time-consuming experimental process that often takes days to perform. A new pressure filtration method developed characterizes a sample only in hours. Using stepped pressure filtration, the compressive yield stress $P_y(\phi)$ and a hindered settling function $R(\phi)$ of flocculated suspensions are determined as a function of the solids volume fraction ϕ. Traditional pressure filtration experiments involve the pressure filtration of a particulate suspension in which the time of filtration t is monitored as a function of piston height from which the specific volume of filtrate V is determined. $P_y(\phi)$ is determined from the equilibrium solids volume fraction. The gradient $d(t/V)/dV$ is traditionally used to determine $R(\phi)$ using a suspension filtration theory developed by Landman and White. The new method uses only one stepped pressure compressibility filtration test and one stepped pressure permeability filtration test to determine $P_y(\phi)$ and $R(\phi)$ for multiple solids volume fractions and substantially decreases the time required for sample characterization. To eliminate the influence of a stepped pressure on calculated permeabilities, the analysis of experimental results was modified using the gradient dt/dV^2. Results are presented for a zirconia suspension comparing both single and stepped pressure filtration.

Introduction

The mineral, pigment, water and wastewater industries produce millions of tons of wet particulate suspensions as process intermediates, wastes, and final products. It is usual to increase the solids concentration of these suspensions at some point in the process through methods including gravity settling, vacuum filtration, centrifugation, and pressure filtration. Flocculants are added to encourage floc formation and thus enhance settling and compression rates. Flocculant selection and dose is often based on measurement of settling rates, while neglecting to characterize the dewaterability of the sediment.

A mathematical theory of the dewatering of flocculated suspensions has been developed by Buscall and White (1987) using the concept of a flocculated particle network structure. They established compressibility and permeability as physical properties that determine the dewaterability of flocculated suspensions. Compressibility is defined as a compressive yield stress $P_y(\phi)$, which is the minimum compressive stress required for the network structure to yield and compress irreversibly for a given solids volume fraction ϕ. Permeability is determined from the hindered settling function $R(\phi) = (\lambda/V_p)r(\phi)$, with units $Pa \cdot s \cdot m^{-2}$. λ/V_p represents the Stokes drag coefficient, with units $Pa \cdot s \cdot m$, divided by the average particle or floc volume. The hindered settling factor $r(\phi)$ is a dimensionless quantity which accounts for the influence of hydrodynamic interactions between particles on the rate of dewatering relative to the Stokes settling velocity for a single settling particle or floc. More specifically, $R(\phi)$ is defined as the hydrodynamic resistance to flow through the suspension network structure as a function of solids volume fraction ϕ. $R(\phi)$ is related to the traditional permeability $k(\phi)$ with units

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m^2, according to the following equation (Green, 1997)

$$k(\phi) = \frac{\eta}{R(\phi)} \frac{1-\phi}{\phi} \tag{1}$$

These material properties can be used to predict dewatering behavior in batch settling (Howells et al., 1990; Landman and White, 1994), continuous flow gravity thickening (Green, 1997; Landman et al., 1988) and pressure filtration (Landman, Sirakoff, and White, 1991; Landman and White, 1994, 1997; Landman, White, and Eberl, 1995).

A limited number of methods have been proposed to characterize $P_y(\phi)$ and $R(\phi)$ as a function of solids volume fraction ϕ. $P_y(\phi)$ can be determined using a centrifuge, as shown by Green (1997), but the measurements take from days to weeks to perform. Determination of $R(\phi)$ from the nonhindered settling rate is possible at low solids concentrations below the gel point ϕ_g, which is the concentration at which a flocculated suspension of particles first becomes fully networked. A pressure filtration rig can be used to determine both $P_y(\phi)$ and $R(\phi)$ at concentrations above the gel point (Green, 1997; Landman et al., 1999; Landman and White, 1994; Landman et al., 1995).

The problem with characterizing the compressibility and permeability of particulate suspensions has historically been that the characterization time is too high. Standard constant pressure filtration requires five or more individual filtration tests to characterize a suspension across a range of solids concentrations. This is a laborious process that can take many days to analyze a large number of samples. A new stepped pressure filtration technique has thus been developed where a sample may be characterized from just one complete stepped pressure compressibility filtration test and one truncated stepped pressure permeability filtration test using the fundamental theory of Landman et al. (1999). Full treatment of the technique and its application to characterizing dewatering behavior is presented separately (de Kretser et al., 2001). This article uses the assumption of a uniform cake porosity to enable a simple physical understanding of why stepped pressure filtration is used. The validity of the stepped pressure filtration method is then backed up by experimental results.

Pressure Filtration

The filtration rig used in this study is shown in Figure 1. The apparatus includes a cylinder filled with a flocculated suspension of particles at an initial volume fraction ϕ_0. A pressure is applied to the top of the suspension via a downward moving piston with a pressure transducer mounted flush with the piston face. The pressure at the piston face P is used in a feedback control loop with a computer and pneumatic cylinder to maintain a constant applied pressure. When the cylinder diameter is small, the influence of wall friction can be significant, but the work of Green (1997) suggests that a cylinder diameter of 26.5 mm should be sufficient to minimize this effect. In the experiments described in this article, the cylinder diameter is 40 mm and wall friction has been ignored. The experimental setup used also has the advantage that the controlled pressure is the pressure measured at the

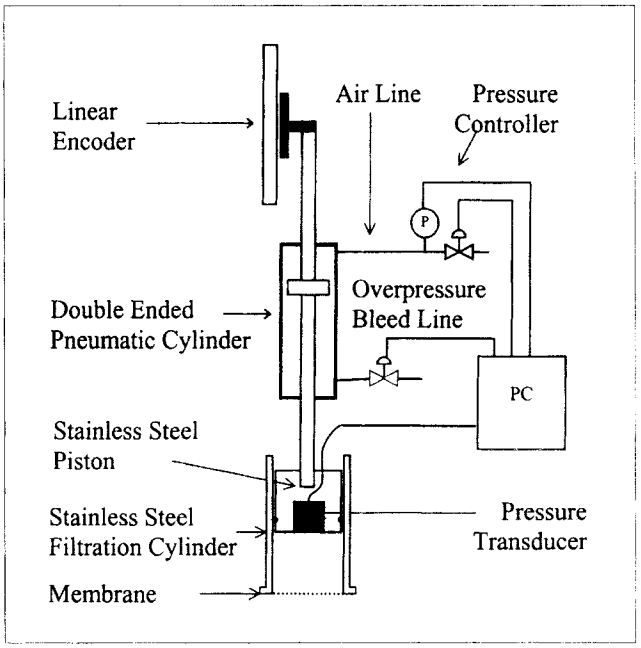

Figure 1. Pressure filtration rig.

piston face, as measured by a pressure transducer in contact with the suspension. A permeable membrane at the base of the cylinder allows liquid to pass through but retains the solids, gradually building up a filter cake. Filtration progress is measured over time t as a function of specific filtrate volume V which is calculated from a measured piston displacement using a linear encoder.

Single pressure filtration

Standard pressure filtration involves the application of a single constant pressure and enables determination of one compressibility and one permeability value for each test. A combination of individual tests can be completed to establish a compressibility and permeability profile for a sample. In a standard single pressure filtration test, a constant pressure P is applied until the filter cake stops compressing. The compressive yield stress $P_y(\phi)$ at the equilibrium solids volume fraction ϕ is equal to the applied pressure, P. The gradient $d(t/V)/dV$ is relatively constant during the middle stages of a filtration test. A single value of the hindered settling function $R(\phi)$ is calculated for each gradient as demonstrated by Green et al. (1998). Landman and White (1997) have shown that this is sufficient to fully characterize, predict and optimize a filtration process. This single pressure method is adequate, but requires significant time with five or more individual filtration tests required to effectively characterize a sample.

Stepped pressure filtration

To reduce characterization time, a new stepped pressure filtration technique has been developed. Stepped pressure filtration provides the option to fully characterize a sample from only one compressibility filtration test and one trun-

cated permeability filtration test. The first test determines the compressibility at a range of pressures and another truncated permeability test determines the hindered settling function over the same range of pressures. The step technique makes efficient use of time in terms of equipment use and labor.

In the compressibility test, the lowest pressure is applied until the filter cake stops exuding liquid and then the pressure is stepped and so on. The data obtained give the compressive yield stress $P_y(\phi)$ for a number of solids volume fractions. Time is saved in that the cake formation time is avoided for all but the initial pressure.

In the permeability test, the first pressure is applied until a specified gradient is stable, then the pressure is incremented. The hindered settling function $R(\phi)$ is traditionally calculated from the gradient of a t/V vs. V plot for a single applied pressure. It will be shown that this method is not mathematically justified when a number of pressures are applied in the one test. Experimentally, the t/V vs. V method produces erroneous gradients and subsequently incorrect $R(\phi)$ values when the pressure is stepped. It will be shown that the problem becomes progressively worse with the number of pressure steps applied. Thus, the t/V vs. V method is not applicable to the stepped pressure technique and another calculation method using t vs. V^2 is proposed.

Mathematical Modeling

The experimental observation that a plot of t/V vs. V is approximately linear, in the initial stages of filtration, suggests that the behavior is similar in form to Darcy's law based on the Hagen-Poisseuille equation (Dahlstrohm et al., 1997) given by

$$\frac{dV}{dt} = \frac{P}{\eta(\alpha_m w V + r)} \tag{2}$$

where η is the filtrate viscosity, α_m is the specific mass filter cake resistance, w is the weight of cake solids per unit volume of filtrate, and r is the membrane resistance.

A modified differential equation has been developed to model the filtration behavior of a compressible networked suspension. The specific mass filter cake resistance has been transformed to a specific volume filter cake resistance A which is a function of the applied pressure P. The specific filtrate volume V has been related to a physical filter cake height B, and membrane resistance redefined as α to minimize confusion with the hindered settling factor. This gives

$$\frac{dV}{dt} = \frac{P}{\eta(AB + \alpha)}. \tag{3}$$

Neglecting membrane resistance, Eq. 3 becomes

$$\frac{dV}{dt} = \frac{P}{\eta AB}. \tag{4}$$

The application of these concepts to the modeling of stepped pressure filtration involves three stages as defined below.

Initial filter cake formation. Filter cake development upwards from the base of the suspension by compression of the initial suspension.

Filter cake compaction. After the pressure is stepped, a new filter cake at higher solids volume fraction develops upwards by compression of the filter cake already present.

Filter cake growth. Once the previous filter cake has been completely compressed to the new filter cake solids volume fraction, the new filter cake develops upwards by compression of the initial suspension.

Initial filter cake formation

A pressure $P_1 = P_y(\phi_1)$, is applied to a suspension at solids volume fraction ϕ_0 and the suspension is assumed to compress to ϕ_1 from the base of the cylinder. This filter cake with constant volume fraction ϕ_1 grows as the pressure P_1 is maintained and the filtrate passes through the membrane on the bottom plate of the cylinder. Given the filter cake resistance A_1, corresponding to the application of pressure P_1, Eq. 4 becomes

$$\frac{dV}{dt} = \frac{P_1}{\eta A_1 B}. \tag{5}$$

A solids mass balance relating the ϕ_1 filter cake thickness B to the specific filtrate volume V gives

$$B = \frac{\phi_0}{\phi_1 - \phi_0} V. \tag{6}$$

Thus, substituting Eq. 6 into Eq. 5 and then integrating from time t_0 to t, and specific filtrate volume V_0 to V gives

$$t - t_0 = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} \left(V^2 - V_0^2\right). \tag{7}$$

Assuming $(t_0, V_0) = (0,0)$ corresponds to the start of the filtration test gives

$$t = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} V^2. \tag{8}$$

Consequently, it is predicted that during filter cake formation

$$\frac{d(t/V)}{dV} = \frac{dt}{dV^2} = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0}. \tag{9}$$

If however, (t_0, V_0) does not correspond to the start of a filtration test, then dt/dV^2 remains unchanged but

$$\frac{d(t/V)}{dV} = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} \left(1 + \frac{V_0^2}{V^2}\right) - \frac{t_0}{V^2}. \tag{10}$$

Filter cake compaction

At time $t = t_1$ and specific filtrate volume $V = V_1$, the ϕ_1 filter cake thickness B_1, derived from Eq. 6, is given by

$$B_1 = \frac{\phi_0}{\phi_1 - \phi_0} V_1. \tag{11}$$

At this time, the pressure is stepped from P_1 to $P_2 = P_s(\phi_2)$ and filter cake compaction begins. A new filter cake with thickness B' and solids volume fraction ϕ_2 begins to form. The filter cake develops from the base of the cylinder by compression of the ϕ_1 filter cake. With the application of pressure P_2 and the corresponding filter cake resistance A_2, Eq. 4 becomes

$$\frac{dV}{dt} = \frac{P_2}{\eta A_2 B'}. \tag{12}$$

A solids mass balance relating the ϕ_2 filter cake thickness B' to the specific filtrate volume V gives

$$B' = \frac{\phi_1}{\phi_2 - \phi_1}(V - V_1). \tag{13}$$

Thus, substituting Eq. 13 into Eq. 12 and then integrating from time t_1 to t and specific filtrate volume V_1 to V, gives

$$t = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}(V - V_1)^2 + t_1. \tag{14}$$

Thus, it is predicted that during filter cake compaction

$$\frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}\left(1 - \frac{V_1^2}{V^2}\right) - \frac{t_1}{V^2}, \tag{15}$$

and

$$\frac{dt}{dV^2} = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}\left(1 - \frac{V_1}{V}\right). \tag{16}$$

Filter cake growth

Soon, at time $t = (t_1 + t_2)$ and specific filtrate volume $V = (V_1 + V_2)$, the ϕ_2 filter cake reaches the top of the ϕ_1 filter cake. At this point, Eq. 14 simplifies to

$$t_2 = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}V_2^2. \tag{17}$$

Also, at this time, the ϕ_2 filter cake thickness B_2, is given by

$$B_2 = \frac{\phi_1}{\phi_2 - \phi_1}V_2. \tag{18}$$

Equating the solids in each filter cake at $V = V_1$ and $V = (V_1 + V_2)$ yields

$$\phi_2 B_2 = \phi_1 B_1. \tag{19}$$

Substituting Eq. 11 for B_1, Eq. 18 for B_2 and then rearranging gives

$$V_2 = \left(\frac{\phi_0}{\phi_2}\right)\left(\frac{\phi_2 - \phi_1}{\phi_1 - \phi_0}\right)V_1. \tag{20}$$

Equations 17–20 describe the material balance to calculate V_2 in terms of V_1 for use in the filter cake growth Eqs. 21–25.

In the filter cake growth stage, the application of pressure P_2 continues and the ϕ_2 filter cake height B'' continues to grow by compression of the original suspension at solids volume fraction ϕ_0. Thus, in the filter cake growth stage, Eq. 4 becomes

$$\frac{dV}{dt} = \frac{P_2}{\eta A_2 B''}. \tag{21}$$

A solids mass balance relating the ϕ_2 filter cake thickness B'' to the specific filtrate volume V during filter cake growth gives

$$B'' = \frac{\phi_0}{\phi_2 - \phi_0}V. \tag{22}$$

Thus, substituting, Eq. 22 into Eq. 21 and then integrating from time $(t_1 + t_2)$ to t and specific filtrate volume $(V_1 + V_2)$ to V gives

$$t = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0}\left(V^2 - \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)}V_1^2\right) + t_1. \tag{23}$$

Consequently, it is predicted that during filter cake growth

$$\frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0}\left(1 + \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)}\frac{V_1^2}{V^2}\right) - \frac{t_1^2}{V^2} \tag{24}$$

and

$$\frac{dt}{dV^2} = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0}. \tag{25}$$

The gradient dt/dV^2 in the filter cake growth stage given by Eq. 25 is of the same form as that in the initial filter cake formation stage given by Eq. 9. Therefore, the gradient dt/dV^2 in the filter cake growth stage could be used in the calculation of permeability in stepped pressure filtration.

Membrane resistance

Membrane resistance contributes an error to the modeled filtration behavior. The filtration behavior with membrane resistance α is modeled using Eq. 3 with applied pressure P_1 and corresponding specific filter cake resistance A_1 to give

$$\frac{dV}{dt} = \frac{P_1}{\eta(A_1 B + \alpha)}. \tag{26}$$

As calculated for the filter cake formation stage, integrating from time t_0 to t and specific filtrate volume V_0 to V predicts t vs. V behavior of the form

$$t - t_0 = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0}\left(V^2 - V_0^2\right) + \frac{\eta\alpha}{P}(V - V_0). \quad (27)$$

The gradients are then calculated to be

$$\frac{d(t/V)}{dV} = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0}\left(1 + \frac{V_0^2}{V^2}\right) + \frac{\eta\alpha}{2P_1}\frac{V_0}{V^2} - \frac{t_0}{V^2} \quad (28)$$

and

$$\frac{dt}{dV^2} = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0} + \frac{\eta\alpha}{2P_1}\frac{1}{V}. \quad (29)$$

The error term ($\eta\alpha/2P_1V$) in Eq. 29 diminishes with increasing extent of filtration due to the inverse V dependence and becomes insignificant at high extents of filtration.

Modeling summary

The derived filtration Eqs. 8, 14 and 23 are summarized in Table 1. Inspection of the filtration equations for the initial filter cake formation and new filter cake growth reveals that they are identical in form with the exception of a constant. This constant can be explained as a time correction. When the pressure is increased during a filtration test, the filter cake at solids volume fraction ϕ_1 that has already formed is gradually converted to a filter cake entirely at ϕ_2. This filter cake took time t to form, but would have taken a different amount of time if the second pressure had been applied from the beginning. Thus, modeling predicts that the normal t/V vs. V analysis to determine the hindered settling function would be in error for subsequent pressures due to the incorrect definition of zero time.

The predicted gradients $d(t/V)/dV$ for each filtration equation are also tabulated in Table 1. The predicted $1/V^2$ dependence of $d(t/V)/dV$ in the filter cake growth stage shows how the gradients in stepped pressure filtration differ from those in single pressure filtration. For single pressure filtration (that is the filter cake formation stage), Eq. 9 predicts that dt/dV^2 is equal to $d(t/V)/dV$. Fortunately, in stepped pressure filtration, the predicted value for dt/dV^2 is also constant in the filter cake growth stage, after filter cake compaction. Use of the gradient dt/dV^2 eliminates the influence of the time correction. This suggests that the gradient dt/dV^2 be used in the calculation of $R(\phi)$ from stepped pressure filtration.

After the pressure is stepped in a permeability test, the V at which compaction finishes can be calculated from Eq. 20 using values of ϕ_0, ϕ_1 and ϕ_2 from a compressibility filtration test. However, this condition is easily satisfied without such a calculation by waiting until dt/dV^2 is stable. To detect stability, the variation in the gradient is monitored until the rate of change is below a certain level. In principle, this is the only control measure required because the gradient will not be stable during filter cake compaction.

Table 1. Key Filtration Equations

Initial Filter Cake Formation

$$P = P_1 = P_y(\phi_1); \quad 0 \le V \le V_1$$

$$t = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0}V^2 \quad (8)$$

$$\frac{d(t/V)}{dV} = \frac{dt}{dV^2} = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0} \quad (9)$$

With membrane resistance

$$\frac{dt}{dV^2} = \frac{\eta A_1}{2P_1}\frac{\phi_0}{\phi_1 - \phi_0} + \frac{\eta\alpha}{2P_1}\frac{1}{V} \quad (29)$$

Filter Cake Compaction

$$P = P_2 = P_y(\phi_2); \quad V_1 \le V \le V_1\frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)}$$

$$t = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}(V - V_1)^2 + t_1 \quad (14)$$

$$\frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}\left(1 - \frac{V_1^2}{V^2}\right) - \frac{t_1}{V^2} \quad (15)$$

$$\frac{dt}{dV^2} = \frac{\eta A_2}{2P_2}\frac{\phi_1}{\phi_2 - \phi_1}\left(1 - \frac{V_1}{V}\right) \quad (16)$$

Filter Cake Growth

$$P = P_2 = P_y(\phi_2); \quad V_1\frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} < V$$

$$t = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0}\left(V^2 - \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)}V_1^2\right) + t_1 \quad (23)$$

$$\frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0}\left(1 + \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)}\frac{V_1^2}{V^2}\right) - \frac{t_1^2}{V^2} \quad (24)$$

$$\frac{dt}{dV^2} = \frac{\eta A_2}{2P_2}\frac{\phi_0}{\phi_2 - \phi_0} \quad (25)$$

Materials and Methods

To test the new method and modeling predictions, a coagulated zirconia suspension was characterized. 600 mL of zirconia (ZrO_2) suspension was prepared at a solids volume fraction of (0.0991 ± 0.0006). The zirconia was obtained from ICI Australia Operations Pty. Ltd., Rockingham, Western Australia. This zirconia (ELECTRO-FINE 0.5 zirconia) has been characterized in the work of Green (1997). The average particle size by volume was determined as 0.47 μm using a Leeds and Northrup Microtrac Particle-Size Analyzer. The solids density was taken as 5,720 $kg \cdot m^{-3}$. A 0.01 M KNO_3 solution, made up from distilled and Milli-Q filtered water, was used as the suspending fluid. The suspension was initially adjusted to pH < 3 with concentrated nitric acid (HNO_3) and dispersed with a high speed mixer. The pH was then neutralized with concentrated potassium hydroxide solution (KOH) and allowed to stabilize over three months. The experimental pH was (6.63 ± 0.07). The gel point ϕ_g is defined as the lowest solids volume fraction at which a suspension forms a continuous network structure. At the test pH of 6.63, the gel point was calculated to be 0.06 using the equilibrium batch settling method proposed by Green (1997).

The compressive yield stress and hindered settling function of the zirconia suspension were determined using single and stepped pressure filtration over a range of five pressures. The

initial solids volume fraction ϕ_0 was determined by weight loss on drying of a small sub sample.

Single pressure filtration

Five single pressure filtration tests, with initial suspension heights of around 25 mm, were performed at pressures ranging from 50 kPa to 200 kPa. The equilibrium solids volume fraction ϕ_f for the applied pressure in each filtration test was calculated from the initial solids volume fraction ϕ_0, initial suspension height h_0 and equilibrium suspension height h_f using

$$\phi_f = \phi_0 \frac{h_0}{h_f}. \tag{30}$$

The gradient $d(t/V)/dV$ was determined using the middle 50% of data points in an effort to eliminate errors. Errors are due to the influence of membrane resistance, the time for the set pressure to be reached at the beginning of the test, and the compaction of the filter cake against the piston face at the end of the test. The term β^2 was determined from the gradient using

$$\beta^2 = \frac{1}{\left(\frac{d(t/V)}{dV}\right)}. \tag{31}$$

The β^2 vs. P relationship was fitted to a power law curve and the hindered settling function $R(\phi)$ was then determined for each measured ϕ_f using the following equation derived by Landman et al. (1999)

$$R(\phi) = \frac{\lambda}{V_p} r(\phi) = \frac{2}{\left(\frac{d\beta^2}{dP}\right)} \left(\frac{1}{\phi_0} - \frac{1}{\phi_f}\right)(1-\phi_f)^2. \tag{32}$$

Stepped pressure filtration

Stepped pressure filtration compressibility and permeability tests were performed over the same pressures as the single pressure filtration tests. The stepped pressure tests were conducted twice, both before and after the single pressure tests. The compressibility tests also with initial suspension heights of around 25 mm, gave the equilibrium solids volume fraction for each pressure. The permeability tests used an initial suspension height of about 50 mm. During the test, each data point was detected by a pulse sent from the linear encoder and corresponded to an increment in V of 1.27×10^{-5} m. The gradient dt/dV^2 was calculated from linear least-squares fits of the previous 30 (V,t) data points. At each pressure, the calculated gradient over the previous 120 data points was monitored until it was stable to within a 1.75% tolerance. The average calculated gradient over these 120 data points was recorded and the pressure stepped. Selection of these stability criteria are a balance between gaining accurate results and gaining results at enough pressures since waiting longer may give more accurate results, but reduce the number of pressures that can be tested. For each set pressure, β^2 was determined from dt/dV^2 using

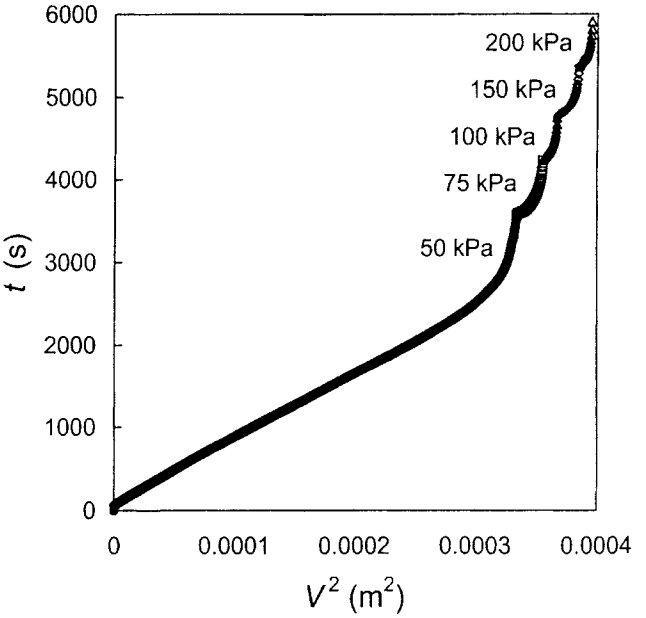

Figure 2. t vs. V^2 for a stepped pressure filtration compressibility test.

$$\beta^2 = \frac{1}{\left(\frac{dt}{dV^2}\right)}. \tag{33}$$

For each pressure, $R(\phi)$ was then calculated using Eq. 32 as it was for single pressure filtration.

Results and Discussion

Compressive yield stress determination

Data for a stepped pressure filtration compressibility test of a zirconia suspension at pH 6.67 are shown in Figure 2 using a plot of t vs. V^2. For the first pressure of 50 kPa, the data shows the initial filter cake formation region predicted by Eq. 8, followed by filter cake compression until the whole suspension is at the equilibrium solids volume fraction for the applied pressure. The pressure is then stepped with the suspension compressed to equilibrium through the range of pressures up to 200 kPa.

The compressive yield stress $P_y(\phi)$ measurements using both single and stepped pressure filtration have been collected and are shown in Figure 3. The measured solids volume fractions at each pressure vary by an average of 0.7%. These variations show no systematic difference between single and stepped pressure filtration, but are due to random and precision errors, in addition to variation of sample properties over time.

Hindered settling function determination

Experimental t/V vs. V behavior for single pressure filtration is shown in Figure 4, showing reasonably linear behavior

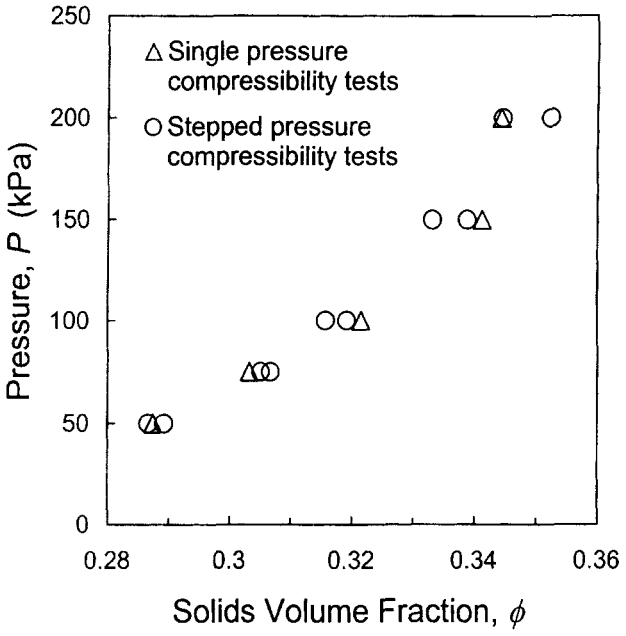

Figure 3. Compressive yield stress $P_y(\phi)$ determined from single and stepped pressure filtration.

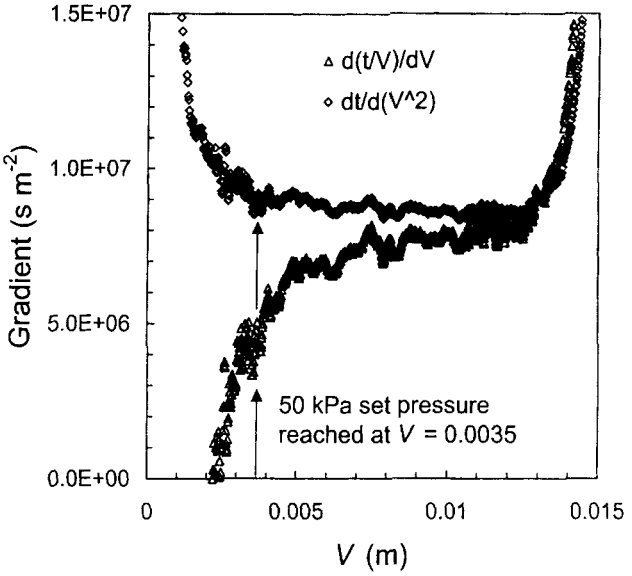

Figure 5. Gradients dt/dV^2 and $d(t/V)/dV$ for single pressure filtration at 50 kPa.

once the 50 kPa set pressure has been reached ($V = 0.035$ m), followed by compression at the end of the test. The experimental gradients $d(t/V)/dV$ and dt/dV^2 for the 50 kPa filtration test are shown in Figure 5. It is noted that all experimental and predicted gradients, $d(t/V)/dV$ and dt/dV^2, shown in Figure 5 and Figure 6, were determined from least-squares fits of ten consecutive data points to smooth out experimental fluctuations. Equation 9 predicts that $d(t/V)/dV$ should be constant until the piston face reaches the filter cake, but Figure 5 shows that this gradient is not stable until approximately half way through the test. The problem is that, on startup, the set pressure is not attained instantaneously. Initially, air pressure in the pneumatic cylinder must build up to overcome friction at the piston-cylinder contact. The pressure then slowly increases to the set point so as to minimize pressure overshoot. This leads to a miscalculation of the time when the experiment effectively starts. Equation 10 accounts for this time issue and predicts that $d(t/V)/dV$ underestimates the desired quantity, even after the pressure set point has been reached. Fortunately, this time error is not intro-

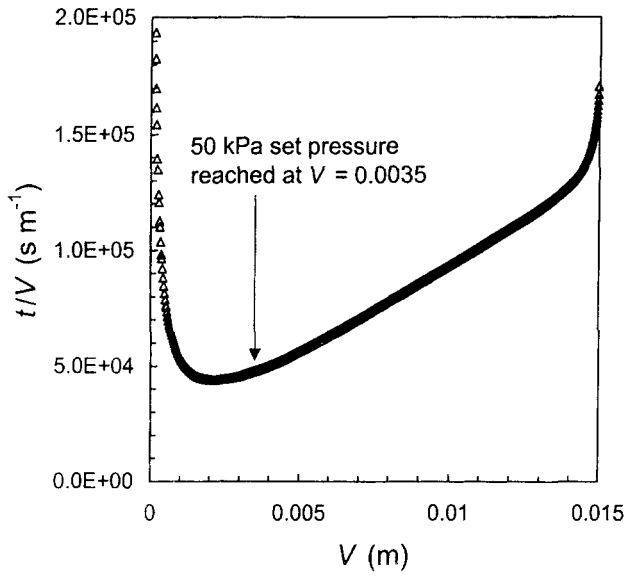

Figure 4. t/V vs. V plot for single pressure filtration at 50 kPa.

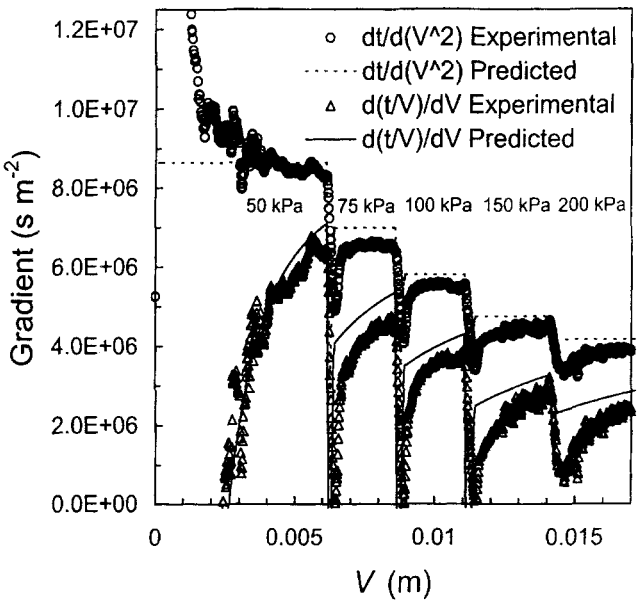

Figure 6. Experimental and predicted gradients dt/dV^2 and $d(t/V)/dV$ for a stepped pressure filtration permeability test.

duced when dt/dV^2 is used. The initially high experimental gradient dt/dV^2 is partially due to the lower applied pressure prior to reaching the pressure set point and the effect of membrane resistance. The influence of membrane resistance is predicted to dissipate with the extent of filtration, according to Eq. 29, and Figure 5 shows that it is experimentally insignificant once the set pressure has been reached. Therefore, modeling predicts that dt/dV^2 can produce results similar to those using $d(t/V)/dV$ for a single applied piston pressure, but experimental results suggest an improvement in accuracy because dt/dV^2 is unaffected by time errors associated with experimental startup.

Experimental and predicted gradients for a stepped pressure permeability test are shown in Figure 6. The values of dt/dV^2 obtained from single pressure filtration have been used to predict the results in stepped pressure filtration using Eqs. 9, 16 and 25. During initial filter cake formation at 50 kPa, the dt/dV^2 prediction does not take membrane resistance into account and therefore underestimates the experimental gradient. This influence is diminished with time, as predicted by Eq. 29. The gradient dt/dV^2 is well predicted during filter cake growth at higher pressures in accordance with Eq. 25. From Figure 6, it is evident that the gradient $d(t/V)/dV$ in a stepped pressure test is not constant during filter cake formation or growth. Equations 10 and 24 predict that the gradient will vary with the inverse of the specific volume of filtrate squared ($1/V^2$). The predicted gradient $d(t/V)/dV$ has been plotted against the experimental results and correlates well with the experimental data. During filter cake compaction, the dramatic changes in gradients predicted by Eqs. 15 and 16 are observed, but not as strongly as predicted due to the step in pressure not being instantaneous.

The observation that the gradients $d(t/V)/dV$ and dt/dV^2 are both well predicted using the modified Darcy's law equations suggests that the model can predict stepped pressure

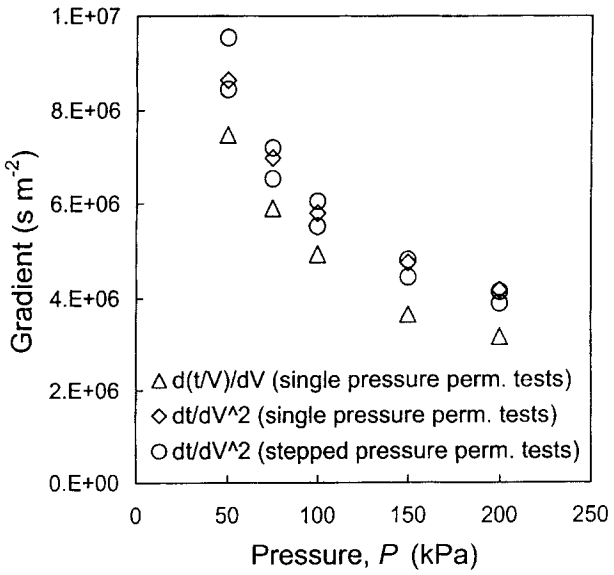

Figure 7. Gradients dt/dV^2 and $d(t/V)/dV$ determined from single and stepped pressure filtration.

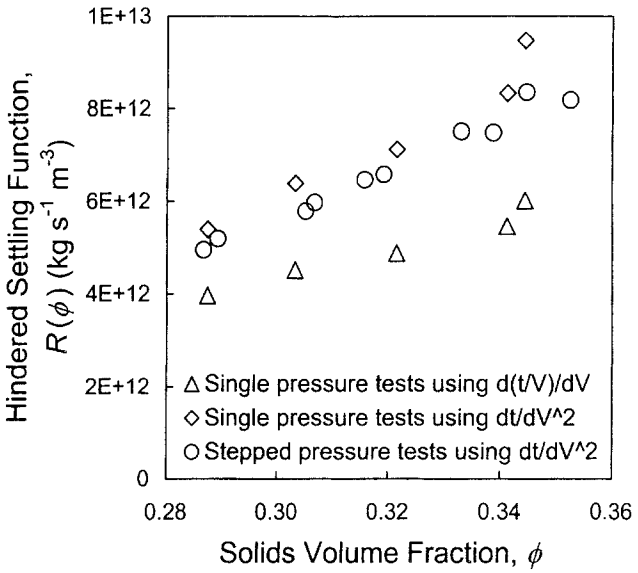

Figure 8. Hindered settling function $R(\phi)$ determined from single and stepped pressure filtration using gradients dt/dV^2 and $d(t/V)/dV$.

filtration behavior with reasonable accuracy. The shortcoming of the modified Darcy's Law modeling is that it assumes a uniform solids volume fraction in the filter cake. The true situation involves a filter cake with a solids volume fraction distribution varying from the final volume fraction at the base to the initial volume fraction at the top. Fortunately, for the modeling conclusions to be valid, it is only required that the same solids distribution profile be obtained from different pressure regimes. For example, the solids distribution profile of a suspension compressed 10 mm at constant pressure being equal to that obtained if it was compressed to the same extent using stepped pressure filtration. How this issue affects the applicability of the stepped pressure calculations using dt/dV^2 requires verification with more comprehensive modeling techniques such as those employed by Landman and Russel (1993). For now, experimental results are compared.

Figure 7 compares experimental gradients from single and stepped pressure filtration. As expected, all $d(t/V)/dV$ gradients calculated from single pressure filtration are significantly below the dt/dV^2 values (18% on average) due to the time error in Eq. 10. The single and stepped pressure dt/dV^2 results are reliable with an average deviation of 3.6% for each applied pressure. Figure 8 compares experimental hindered settling functions $R(\phi)$ for single and stepped pressure filtration. Single pressure $d(t/V)/dV$ values under-predict $R(\phi)$ values (27% on average) and thus significantly overpredict the permeability of the suspension. dt/dV^2 values in both single and stepped pressure filtration predict $R(\phi)$ with an average deviation of only 4.3%. The data clearly indicates that dt/dV^2 should be used for $R(\phi)$ determination.

Sample characterization time

For the five single pressure filtration tests, the total characterization time was around 7 h, including a sample changeover time of 40 min per test. Figures 2 and 6 show filtration data for stepped pressure filtration compressibility

and permeability tests. These two tests alone enable characterization of compressibility and permeability of the zirconia suspension in a time that is halved to 3.4 h. When results from more than 5 pressures are required, the time saving becomes even more significant. This quicker characterization reduces the likelihood that suspension properties will change due to influences such as flocculation degradation, pH drift or chemical reaction. Thus, characterization via stepped pressure filtration is clearly advantageous.

Conclusions

Stepped pressure filtration allows characterization of suspension compressibility and permeability using only one stepped pressure compressibility filtration test and one truncated stepped pressure permeability filtration test. The compressibility test determines the compressive yield stress $P_y(\phi)$ for a range of solids volume fractions ϕ. The permeability test determines the hindered settling function $R(\phi)$ for the same solids volume fractions. A new calculation method uses dt/dV^2 to accurately determine $R(\phi)$. The new dt/dV^2 method is more accurate than the previously used $d(t/V)/dV$ method because $d(t/V)/dV$ becomes a function of specific filtrate volume V with pressure variations.

Further Work

At low pressures, the influence of membrane resistance can become significant. In the proposed stepped pressure filtration technique, waiting for the membrane resistance to become insignificant in the calculated gradient increases filtration time. When sample availability is limited, this limits the number of pressure steps that can be performed. If this error is predicted and corrected for, then more pressures can be tested with the same quantity of sample.

Values of P and V are readily available during a filtration test so only the filtrate viscosity η, and membrane resistance α, need to be determined in advance to apply a correction to the gradient. The filtrate viscosity η can be determined from a range of standard rheological techniques. The membrane resistance α can be determined from the filtration of a liquid with no suspended solids. Solids free filtration behavior using Eq. 3 with filter cake height $B = 0$ predicts constant velocity filtration

$$\frac{dV}{dt} = \frac{P}{\eta\alpha}. \tag{34}$$

Therefore, the membrane resistance is calculated using

$$\alpha = \frac{P}{\eta\dfrac{dV}{dt}}. \tag{35}$$

As a consequence, membrane resistance in stepped pressure filtration can be predicted and eliminated. Alternatively, if this influence cannot be quantified accurately, then the final results should just take longer, but not be affected in terms of accuracy.

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Notation

A = specific volume filter cake resistance
A_1 = specific volume filter cake resistance corresponding to the application of pressure P_1
A_2 = specific volume filter cake resistance corresponding to the application of pressure P_2
$B = \phi_1$ filter cake height
$B' = \phi_2$ filter cake height during ϕ_1 filter cake compaction
$B'' = \phi_2$ filter cake height during filter cake growth
$B_1 = \phi_1$ filter cake height at time t_1
$B_2 = \phi_2$ filter cake height at time $t_1 + t_2$
h_0 = initial suspension height
h_f = equilibrium suspension height
P = piston pressure
P_1 = applied piston pressure, $P_y(\phi_1)$
P_2 = applied piston pressure, $P_y(\phi_2)$
$P_y(\phi)$ = compressive yield stress
r = membrane resistance
$r(\phi)$ = hindered settling factor
$R(\phi)$ = hindered settling function, $(\lambda/V_p)r(\phi)$
t = time of filtration
t_0 = initial filtration time
t_1 = time when pressure is stepped
t_2 = time for filter cake compaction
V = specific filtrate volume, volume of filtrate divided by membrane area
V_0 = initial specific filtrate volume
V_1 = specific filtrate volume when pressure is stepped
V_2 = specific filtrate volume required to compact filter cake from ϕ_1 to ϕ_2
w = weight of cake solids per unit volume of filtrate
α = membrane resistance
α_m = specific mass filter cake resistance
β^2 = filtration parameter
ϕ = suspension solids volume fraction
ϕ_0 = initial suspension solids volume fraction
ϕ_1 = filter cake solids volume fraction corresponding to applied pressure $P_1 = P_y(\phi_1)$
ϕ_2 = filter cake solids volume fraction corresponding to applied pressure $P_2 = P_y(\phi_2)$
ϕ_g = gel point
ϕ_f = equilibrium solids volume fraction
η = filtrate viscosity
λ/V_p = Stokes drag coefficient divided by average particle or floc volume

Literature Cited

Buscall, R., and L. R. White, "The Consolidation of Concentrated Suspensions," *J. Chem. Soc. Faraday Trans. I*, **83**, 873 (1987).

Dahlstrohm, D. A. et al., "Liquid Solid Operations and Equipment," *Perry's Chemical Engineers' Handbook*, 7th ed., R. H. Perry and D. W. Green, eds., McGraw Hill, Sydney, **18**, 74 (1997).

de Kretser, R. G., S. P. Usher, P. J. Scales, K. A. Landman, and D. V. Boger, "Rapid Filtration Measurement of De-Watering Design and Optimization Parameters," *AIChE J.*, in press (2001).

Green, M. D., "Characterisation of Suspensions in Settling and Compression," PhD Thesis, Univ. of Melbourne Dept. of Chemical Engineering, Australia, (1997).

Green, M. D., K. A. Landman, R. G. de Kretser, and D. V. Boger, "A Pressure Filtration Technique for Complete Characterisation of Consolidating Suspensions," *Ind. Eng. and Chemistry Res.*, **37**, 4152 (1998).

Howells, I., K. A. Landman, A. Panjkov, C. Sirakoff, and L. R. White, "Time Dependent Batch Settling of Flocculated Suspensions," *Appl. Math. Model.*, **14**, 77 (1990).

Landman, K. A., and W. B. Russel, "Filtration at Large Pressure for Strongly Flocculated Suspensions," *Phy. of Fluids A*, **5**, 550 (1993).

Landman, K. A., C. Sirakoff, and L. R. White, "Dewatering of Flocculated Suspensions by Pressure Filtration," *Phy. of Fluids A*, **3**, 1495 (1991).

Landman, K. A., J. M. Stankovich, and L. R. White, "The Measurement of Filtration Diffusivity D(ϕ) of a Flocculated Suspension," *AIChE J.*, **45**, 1875 (1999).

Landman, K. A., and L. R. White, "Solid/Liquid Separation of Flocculated Suspensions," *Adv. in Colloid and Interface Sci.*, **51**, 175 (1994).

Landman, K. A. and L. R. White, "Predicting Filtration Time and Maximizing Throughput in a Pressure Filter," *AIChE J.*, **43**, 3147 (1997).

Landman, K. A., L. R. White, and R. Buscall, "The Continuous Flow Gravity Thickener: Steady State Behaviour," *AIChE J.*, **34**, 239 (1988).

Landman, K. A., L. R. White, and M. Eberl, "Pressure Filtration of Flocculated Suspensions, *AIChE J.*, **41**, 1687 (1995).

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