

Rapid Filtration Measurement of Dewatering Design and Optimization Parameters

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To characterize and optimize the compressive dewatering of suspensions, rapidly determinable material parameters are required. A mathematical framework exists for characterization of dewatering using three parameters: the compressive yield stress $P_y(\phi)$, the hindered settling function $r(\phi)$, and a solids diffusivity $D(\phi)$. A new variation on traditional constant pressure, batch filtration tests is described along with a computer-controlled filtration apparatus that enables complete characterization of a suspension with respect to dewatering in a matter of hours. The testing methods are validated experimentally on flocculated zirconia suspensions along with calculation of the material dewatering parameters. Application of the calculated parameters to modeling of a real filtration test shows excellent agreement. Practical implications of the modeling are also discussed.

Introduction

Solid/liquid separation processes such as pressure filtration, centrifugation, and thickening are important unit operations in the minerals and chemical industries. Such dewatering operations are useful in that they allow efficient recovery of water and at the same time, produce tailings or the desired product at a high concentration of solids. Effective dewatering is vitally important to the cost and efficiency of many processes. For example, the transportation and mixing of high solids slurries is generally more cost-effective than for low solids slurries and, since the cost of water is substantial, any reduction in use is important. The area of land required to dispose of high solids tailings is also reduced relative to traditional disposal methods. Dealing with waste tailings is, in itself, a worldwide issue (Glenister and Abbott, 1989).

Dewatering processes have become more common in recent years (Lange, 1989; Haerle and Haber, 1996) in the wet processing of ceramic metal oxide suspensions. Application of compressive pressures to an initially low solids concentration suspension in a porous mold results in dewatering to form a very high solids concentration, "green body." The main

advantage of high solids wet processing is the minimization of shrinkage on firing, which is a major cause of ceramic cracking and strength reduction. The key to almost all dewatering operations lies in the use of either chemical additives such as high molecular weight polymer flocculants, or surface chemical conditions to control the interparticle interactions in the suspension, thereby controlling the rate and extent of dewatering.

Optimizing dewatering (the extent and time of dewatering) of particulate suspensions is a difficult process. Recently, Landman and White (1997) produced a model that describes the process of pressure filtration from fundamental considerations by utilizing two well-defined fundamental quantities: the compressive yield stress $P_y(\phi)$, which characterizes the extent of dewatering and the hindered settling factor $r(\phi)$ (effectively an inverse permeability) which quantifies the rate of dewatering (Landman and White, 1997, 1992; Buscall and White, 1987). Both these parameters have been subject to substantial investigation, however, their usefulness as a practical tool for optimization of "real" systems has been limited by the rate at which current experimental methods allow determination.

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Centrifugal methods can be employed for measurement of compressive yield stresses but the time required to obtain one set of data is of the order of weeks (Green and Boger, 1997; Channell and Zukoski, 1997; Bergstrom et al., 1992). The pressure filtration technique can be employed for the measurement of both compressibility and permeability, however, once again, times for complete characterization of a material are of the order of a couple of days. For use as a practical tool, the data required for the theoretical framework needs to be available in under a day.

This article describes modifications to the operation of a traditional laboratory-scale filter press which enables rapid determination of $r(\phi)$ and $P_y(\phi)$. The material investigated is a well characterized zirconia suspension, pH adjusted to the isoelectric point, or point of strongest flocculation (Leong et al., 1993). Validation of the new testing method is presented. The data obtained are then used to practically apply the Landman-White theory to model filtration behavior from experimental data acquisition to the calculation of $r(\phi)$, $P_y(\phi)$ and the solids diffusivity $D(\phi)$. The model can be used further to predict filtration behavior for different design or operational conditions. Results presented elsewhere (Aziz et al., 2000) show how the theory can be utilized in the flocculation optimization of not just pressure filtration, but all dewatering operations.

Theory

The compressive behavior of a suspension can be characterized by a compressive yield stress $P_y(\phi)$ (Buscall and White, 1987). In a flocculated suspension, above a sufficiently high solids concentration, an interconnected particle network exists which has a physically measurable network strength which depends on the local volume fraction of solids ϕ . This

strength is defined as the compressive yield stress. The network will remain in its original form until an applied stress ΔP on the network exceeds the compressive yield stress. At this stage, the structure of the network will collapse, irreversible particle consolidation will occur, and the local volume fraction will increase (Buscall and White, 1987). Clearly, $P_y(\phi)$ will increase with ϕ as the number of interparticle linkages increases so dewatering occurs until ϕ is such that $P_y(\phi) = \Delta P$. $P_y(\phi)$ can only be traced in a regime where ϕ is greater than a volume fraction called the gel point ϕ_g . Below this limit ($\phi < \phi_g$), the particle concentration is too small to allow a particle network to develop and thus the compressive yield stress is zero (neglecting the suspension osmotic pressure).

Permeability is quantified by a parameter called the hindered settling factor $r(\phi)$. This factor takes into account the hydrodynamic interactions between particles which, in concentrated dispersions, increase the drag on any given particle (Landman et al., 1995). These interactions between particles hinder the relative movement of solids and liquid and thus $r(\phi)$ increases in a nonlinear fashion with volume fraction. $r(\phi)$ has the following properties: $r(\phi) \rightarrow 1$ as $\phi \rightarrow 0$, and $r(\phi) \rightarrow \infty$ as $\phi \rightarrow 1$. Importantly, $r(\phi)$ is a parameter which describes dewatering in settling, where it quantifies the drag force on a particle moving through a liquid, and, also in a networked bed, where it quantifies the drag experienced by liquid moving through a network of solids. Thus $r(\phi)$ may be used in the modeling of filtration behavior.

A number of methods are available for the determination of $P_y(\phi)$ and $r(\phi)$ (Green, 1997). As stated earlier, the main drawbacks of most of the available techniques are that either the characterization times are long, rendering techniques inappropriate for "real-time" process optimization applications or time-dependent systems, or only one of $P_y(\phi)$ and $r(\phi)$

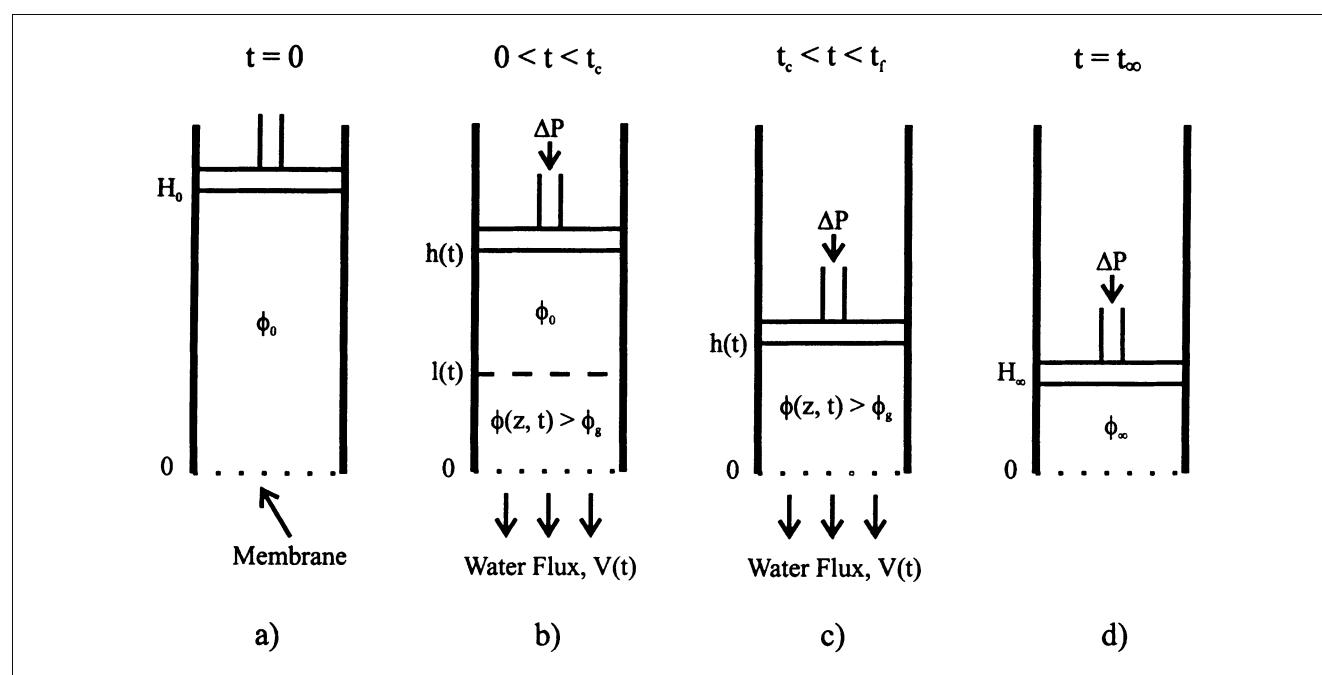


Figure 1. Constant pressure batch filtration.

are determined. In this work the method employed is a modification of a steady state batch filtration technique which has the advantage of measuring data relevant to the calculation of both $P_y(\phi)$ and $r(\phi)$.

The conventional filtration method uses a filter press in which a constant applied pressure is supplied via a piston acting on a suspension in a compression cylinder and liquid is expressed through a porous membrane at the base of the cylinder (Green et al., 1998). The filtration process is presented in Figure 1. During constant pressure filtration of a material initially at a solids volume fraction ϕ_o , buildup of a filter cake occurs from the membrane upwards and the piston moves downwards until a point that the descending piston and rising filter cake meet—the initial phase is called cake-formation and is represented in Figure 1b. The solids concentration at the membrane jumps to the final solids corresponding to the applied pressure, ϕ_∞ , as soon as filtration commences, and a concentration gradient exists in the cake, decaying with distance from the membrane to ϕ_g at the top of the cake $[l(t)]$. The time at which the cake and piston meet is designated t_c . During cake formation, the relationship between time t and the specific volume of filtrate (volume expressed per unit area of membrane) follows a quadratic relationship such that a plot of t vs. V^2 is linear. The inverse of the slope of this linear portion is designated β^2 and can be used in the determination of $r(\phi)$.

At t_c , the filter cake has a nonconstant solids concentration distribution and further consolidation of the cake occurs until a time t_∞ , at which point the filter cake has a uniform solids concentration distribution at ϕ_∞ . At equilibrium, after the sample has stopped compressing for a given applied pressure, the compressive yield stress $P_y(\phi_\infty)$, at the final volume fraction solids can be equated to the applied pressure. The latter phase of filtration is called cake compression and is characterized by an exponential type decay of the filtration rate with expressed filtrate volume. Figure 2 shows an example of t vs. V^2 data for a single constant pressure filtration experiment showing the linear, cake formation region and the exponential-type behavior in the cake compression region (Figure 1c).

By performing a constant pressure batch filtration at a range of pressures, as in the work of Green et al. (1998) and Green and Boger (1997), P_y as a function of ϕ can be directly determined and $r(\phi)$ can be calculated. The method of calculation of $r(\phi)$ utilized by Green et al. (1998) involved an approximation for the average solids concentration in the forming compact bed. The newer, linearized version of the Landman-White filtration theory allows a more mathematically exact determination of $r(\phi)$ described as follows.

According to the following equation, the slope of a plot of β^2 vs. ΔP can be used to calculate $r(\phi)$:

$$R(\phi_\infty) \equiv \left(\frac{\lambda}{V_p} \right) r(\phi_\infty) = \frac{2}{d\beta^2} \left(\frac{1}{\phi_o} - \frac{1}{\phi_\infty} \right) (1 - \phi_\infty)^2 \quad (1)$$

where ϕ_∞ is the equilibrium volume fraction at ΔP (Landman et al., 1999), V_p is volume of a particle and λ is the Stokes drag coefficient for a single particle in an infinite medium ($= 6\pi a_p \eta$ for spherical particles, a_p is the radius of

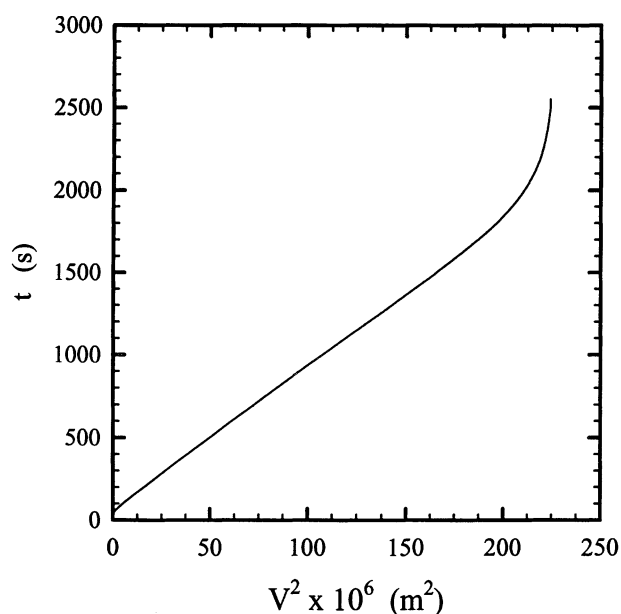


Figure 2. Filtration plot for 50 kPa constant, single pressure filtration of 10% v/v zirconia suspension.

$h_o = 0.02287$ m.

a particle and η is the fluid viscosity). Note that, in general, it is impractical to calculate the exact value of λ/V_p in Eq. 1 due to the size required in the calculation being the size of an aggregate in the suspension. Thus, the definition of the hindered settling factor usually refers to the entire term $(\lambda/V_p) r(\phi)$, and this will be denoted $R(\phi)$ here.

Since the overall dewatering process depends on both compressibility and permeability Landman et al. (1995) derived a parameter $D(\phi)$ that combines $P_y(\phi)$ and $r(\phi)$ for the dewatering process. $D(\phi)$ acts as a volume fraction dependent solids diffusion coefficient such that

$$D(\phi_\infty) = \left[\frac{\frac{dP_y(\phi_\infty)}{d\phi_\infty}}{\frac{\lambda}{V_p} r(\phi_\infty)} \right] (1 - \phi_\infty)^2 \quad (2)$$

As a diffusion coefficient, a higher $D(\phi)$ indicates a material that dewateres faster.

Equation 2 above can also be expressed directly from the relationship between β^2 and ϕ_∞ as follows (Landman et al., 1999)

$$D(\phi_\infty) = \frac{1}{2} \frac{d\beta^2}{d\phi_\infty} \left(\frac{1}{\phi_o} - \frac{1}{\phi_\infty} \right)^{-1} \quad (3)$$

Equation 3 has the advantage of calculating $D(\phi_\infty)$ directly from β^2 vs. ϕ_∞ data which means only one differentiation of the experimental data is required. The $D(\phi)$ parameter can be incorporated into models of all types of dewatering operations.

Landman and White (1997) linearized their theory of filtration to make it more useful for engineering applications and showed that the filtration time t_f to reach any average solids concentration up to ϕ_∞ has a roughly inverse proportionality to $D(\phi)$ for a given filtration pressure. $D(\phi)$ is, therefore, a material property that characterizes dewatering. The functional form of t_f is given by

$$t_f = h_o^2 \left(\frac{\phi_0}{\phi_\infty} \right)^2 \frac{T_f}{D_\infty} \quad (4)$$

where t_f is the true time of dewatering, ϕ_o and ϕ_∞ are the initial and final volume fractions respectively, T_f is the scaled (dimensionless) time-to-filtration, and D_∞ is $D(\phi_\infty)$. T_f has a complex dependency on ϕ_o and $D(\phi)$ over the range of ϕ_o to ϕ_∞ . The calculation of T_f depends on whether the filtration time corresponds to a time within the cake formation or the cake compression regions of filtration. For cake formation, T_f is calculated from

$$T_f = \frac{A}{B^2} \left[\frac{1}{\phi_o} - \frac{1}{f\phi_\infty} \right], \quad (5)$$

where A is a numerical constant for a filtration at a given pressure that incorporates a dimensionless filter cake formation time (the dimensionless time at which cake formation changes to cake compression), ϕ_∞ is the equilibrium volume fraction solids for the given pressure and f is a stopping fraction defined as the desired final average cake solids concen-

tration as a fraction of ϕ_∞ . B is a numerical constant that is a function of the initial feed solids volume fraction and a parameter related to the integration of a $D(\phi)$ -based quantity over the range of volume fractions from ϕ_0 to ϕ_∞ (Landman and White, 1997).

For cake compression (when f is close to 1)

$$T_f = C + \frac{4}{\pi^2} \ln \left[\frac{f\phi_\infty E}{1-f} \right], \quad (6)$$

where C is a numerical constant for a filtration at a given pressure that incorporates the dimensionless filter cake formation time and E is a constant that is related to ϕ_∞ and the integration of the $D(\phi)$ -based quantity over the range of volume fractions from ϕ_0 to ϕ_∞ . Note that the constants listed in Eqs. 5 and 6 are calculated explicitly from the $D(\phi)$ values of the material for which filtration is to be simulated and the simulation "experimental conditions." Full definitions and derivations for the cake formation and compression region equations are presented in Landman and White (1997).

Thus, calculation of the time-to-filtration requires a knowledge of the behavior of the $D(\phi)$ parameter over a range of solids concentrations. Once known, t_f can be calculated for any pressure over the range of pressures from which $D(\phi)$ was determined and the effect of changing initial solids concentration, desired final solids or filter cake thickness can be determined. The above equations refer specifically to pressure filtration, however, they can be adapted for the vacuum filtration case where the filtration effectively takes place from an infinite reservoir of feed.

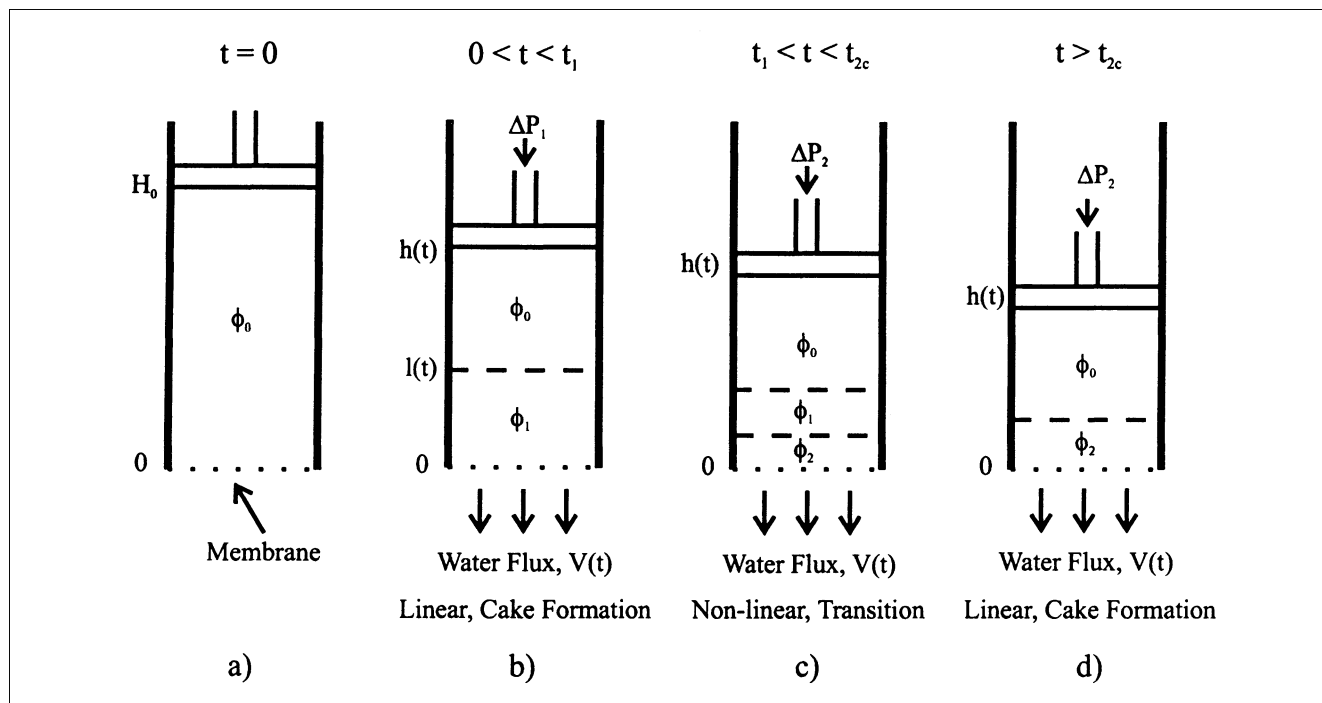


Figure 3. Filter cake changes on stepping pressure during cake formation.

Calculation of the $D(\phi)$ and T_f parameters was completed using a computer program written in Mathematica using the theory described in Landman and White (1997).

Re-designing the experiment

In order to utilize the theoretical framework described above for optimization purposes and in particular for the characterization of the performance of a range of additives or dose rates, it is necessary to obtain the dewatering parameters rapidly. Traditional laboratory batch filtration affords characterization of suspensions in a matter of days and ideally requires filtration at least of five pressures for accurate utilization of results. Thus, at least five individual filtration experiments are required for characterization of a suspension at one additive condition [each experiment yields data for one $P_y(\phi)$ and one $R(\phi)$ point].

To increase the speed of characterization, an alternative testing procedure was developed which requires only two separate filtration experiments to completely characterize a suspension over a range of pressures. Suspension characterization is separated into a compressibility (a full filtration) and a permeability (a truncated filtration) test.

The compressibility test is conducted as a normal batch filtration experiment at the lowest pressure desired and the filtration is allowed to run to equilibrium. At the end of the lowest pressure filtration, the pressure is then increased to the next level and compression to equilibrium is performed. The pressure is then increased again and so on. Thus, from one single experiment, the final solids for the full range of desired pressures can be determined, giving $P_y(\phi)$.

The permeability test determines values of β^2 for the same pressures selected in the compressibility test. Characterization is completed by ascertaining when the slope of a T vs. V^2 plot is linear for the lowest pressure selected. As soon as the slope is stable, the pressure is then stepped up to the next pressure, *while still within the linear, cake formation region of filtration*. A short transition region of filtration behavior follows each pressure step, which gives way to a new linear region with a slope characteristic to the second pressure. Again, once the second slope has been determined, the pressure can be stepped up to the next desired level and so on. The slopes determined will be applicable for the calculations as long as all of the data are obtained before the filtration moves into the cake compression region (Figure 1c).

The processes occurring when the pressure is stepped while filtering within the cake formation region is presented in Figure 3. In Figure 3b, filtration commences at the first pressure, P_1 , and the filter cake begins to form at the membrane with a volume fraction, of ϕ_1 . A simplifying assumption that the formed filter cake is at a constant volume fraction, ϕ_i such that $P_y(\phi_i) = \Delta P_i$ (the applied pressure) has been validated by Usher et al. (2001). On stepping the pressure from ΔP_1 to ΔP_2 at time, t_1 , the formed cake begins to compress from ϕ_1 to ϕ_2 , a volume fraction consistent with the new applied pressure. A region of transition from ϕ_1 to ϕ_2 moves upwards through the formed filter cake until the entire cake is at ϕ_2 (Figure 3c). Filtration then proceeds as normal for the new pressure ΔP_2 (Figure 3d).

In terms of the behavior of t vs. V^2 , the filtration should be linear during the stage indicated in Figure 3b, nonlinear

within the stage indicated in Figure 3c and then linear again once the uniform cake at the new set pressure has been attained in Figure 3d.

The theoretical validation and further justification for the use of the multiple-pressure step technique is presented separately (Usher et al., 2001). In that article it is shown that using simple Darcian considerations, the filtration behavior can be modeled using the assumption of constant volume fraction distribution within the filter cake and the approximate models are compared with experimental results. The agreement is good.

Thus, from the compression test, P_y as a function of ϕ is determined. The corresponding β^2 values are determined from the results of the permeability test which is conducted over the same set pressures as the compression test. The combination of the results of each test yields the data required for the calculation of $R(\phi)$ and $D(\phi)$.

Within the current article, results will be presented that demonstrate that the stepped pressure filtration technique and apparatus yields valid results in a fraction of the time taken previously for dewatering characterization. Additionally, the application of the dewatering parameters for optimization and design will be demonstrated.

Experimental Apparatus Design

Hardware

The filtration apparatus was based on that of Green et al. (1998) but with substantial modifications as detailed in Figure 4. The filtration apparatus uses a pneumatic cylinder to apply pressure to a sample contained in a compression cell, forcing liquid out through a filter membrane at the cylinder base which is supported by a permeable sintered metal disk.

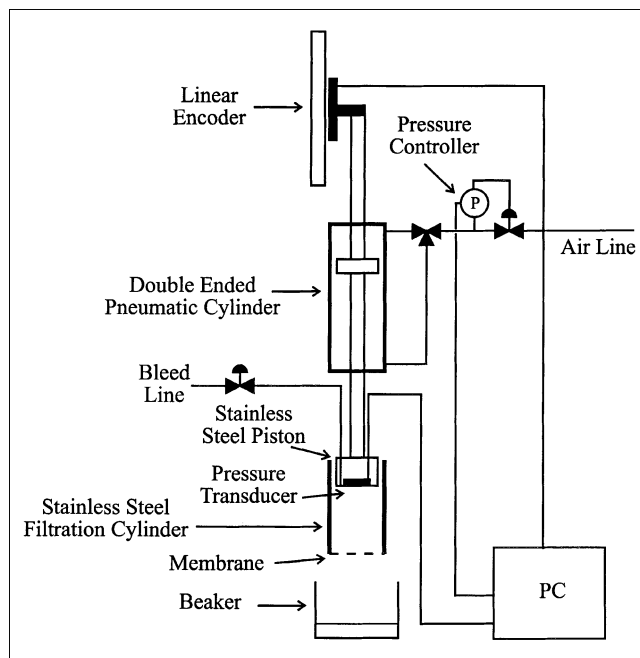


Figure 4. Filtration rig showing all essential components.

The pressure of filtration is monitored using a pressure transducer mounted flush in the piston face, and the rate of filtration is monitored through the rate of movement of the piston. Originally, Green et al. (1998) also measured the rate of filtrate efflux via a balance under the filtration cell, however, monitoring of the piston displacement was found to be the more effective method, especially for the reduced scale of experiments conducted in this work.

To reduce filtration testing times, the overall scale of filtration experiments and, thus, filter cake thicknesses were reduced. Consequently, in order to maintain acceptable accuracy of position determination and, hence, volumetric flow of filtrate, a linear encoder was utilized to measure the displacement of the piston. The linear encoder, in conjunction with X4 edge triggering signal conditioning, improved spacial resolution to 10 μm .

To improve pressure control and facilitate the pressure stepping required for stepped pressure experiments, a Bronkhorst "EL Press" pressure controller was employed to control the pressure in the pneumatic cylinder. The set point of the pressure controller was utilized in a cascade control loop to control the pressure at the piston face. The pressure control system allowed pressures ranging from 5 to 300 kPa to be tested.

Software

The running of the filtration apparatus is controlled using a program written in LabVIEW which allows testing in three modes: single pressure, multiple pressure compressibility, and multiple pressure permeability tests. Single pressure experiments are traditional filtration experiments such as those performed by Green et al. (1998) and Channell and Zukoski (1997). The multiple pressure compressibility test involves determination of ϕ_{∞} at a range of pressures in the one experiment. The program conducts a filtration experiment to its completion at the lowest pressure set and determines when piston movement has dropped below a minimum rate and then steps to the next set pressure and waits for the piston movement to slow, and so on. The multiple pressure permeability test involves determination of β^2 at the same pressures as the compression test. The program conducts a normal filtration experiment at the lowest pressure until the data for t vs. V^2 are linear within user defined tolerance ($\pm \sim 1.75\%$) over a user defined number of preceding data points (usually around 100 points). Once linear, the program then steps to the next pressure and waits for linearity, and so on.

Overall, the LabVIEW program logs and manipulates the basic data, and controls the experimental pressure in addition to monitoring the criteria used to evaluate when to step between pressures. A graphical interface allows the user to view the progress of the experiment, monitoring transient behavior of the t vs. V^2 data and the slopes calculated.

The final output data provided by the program for a compressibility test is ϕ_{∞} for the pressures investigated and a slope m of the linear portion of the t vs. V^2 plot for the first set pressure— β^2 is also calculated. The final output data for the permeability test is m and β^2 for the same pressures. The results can then be combined for the calculation of $R(\phi)$ and $D(\phi)$.

Materials and Methods

The material used in all tests was zirconia supplied by Hanwah Ceramics, Rockingham, Western Australia. The preparation of the zirconia suspensions followed the procedure of Leong and Boger (1990) and Green et al. (1998). Samples were prepared at the desired solids concentrations by dispersing the solids in 0.01 M KNO_3 at a pH below 5, where there is a repulsive interaction between the particles. Samples were sheared in an Ultra Turrax high shear mixer to break up any aggregates and then the pH adjusted to that required for testing. All experiments were conducted at a pH of 6.8, just below the iso-electric point of zirconia, where the samples were strongly flocculated. The gel point of the suspensions was determined to be 0.053 volume fraction solids at a pH of 6.8 via a new sedimentation method which will be discussed in an upcoming publication.

Filtration experiments were conducted on samples with initial solids concentrations between 0.131 and 0.042 volume fraction solids. As such, results above and below the gel point could be compared. It should be noted that while the basis of the Landman and White model is for $\phi_o < \phi_g$, it has been observed that linear behavior in t vs. V^2 also exists for $\phi_o > \phi_g$ (Green et al., 1998; Channell and Zukoski, 1997) and that results obtained for tests commenced above and below the gel point are in agreement. Some comments will be made later regarding other considerations in choosing an initial solids concentration for filtration experiments.

At the conclusion of a compressibility test, the cake was always weighed and dried, to measure the final cake solids concentration. This was critical, as during loading of the suspension into the filtration, cell air can be entrained into the sample and the presence of air bubbles leads to errors in the computer determination of the solids. The computer-determined solids concentrations were always back corrected based on the final measured solids in the cake.

Results and Discussion

Validation of the experimental rig and procedure

The validity of the use of a filtration apparatus for the determination of $P_y(\phi)$ and $R(\phi)$ has been well established and validated by a number of workers (Green et al., 1998; Green and Boger, 1997; Channell and Zukoski, 1997), however, the development of the stepped pressure filtration procedure required validation.

Figure 5 shows a typical t vs. V^2 trace for a compressibility test and demonstrates clearly the behavior of the cake as it is progressively compressed under increasing pressures. Note that for this test, a value of β^2 is only obtained for the first pressure, however, this serves as a comparison for the β^2 at the same pressure in the permeability test. Comparison of $P_y(\phi)$ data obtained from single pressure, and step pressure tests is presented in Figure 6. Good agreement is indicated. Note that the data in Figure 6 are for a different set of pressures than those in Figure 5.

There has been some discussion in the literature regarding the existence of initial solids concentration dependencies in measurement of $P_y(\phi)$ (Green and Boger, 1997) which would be expected to be manifested in a test such as the step pressure compressibility test as ϕ_o for each pressure, aside from

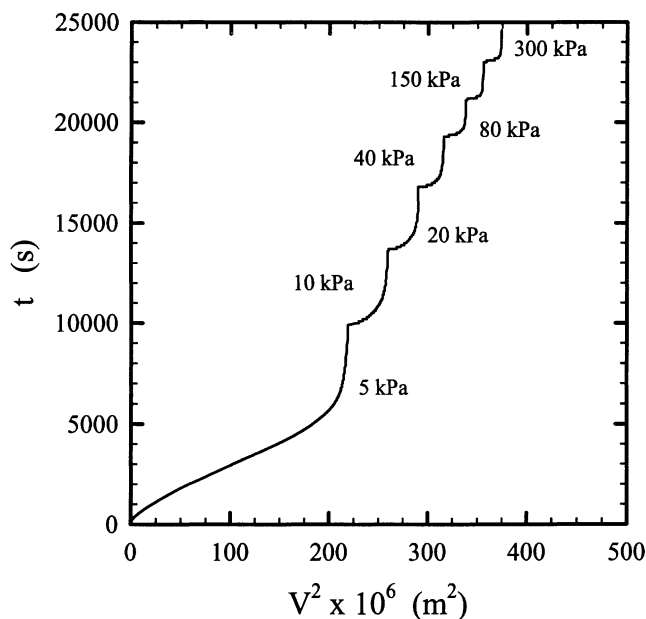


Figure 5. Example of t vs. V^2 for stepped pressure filtration compressibility test.

Zirconia, pH = 6.8, $\phi_o = 0.073$, $h_o = 0.02489$ m.

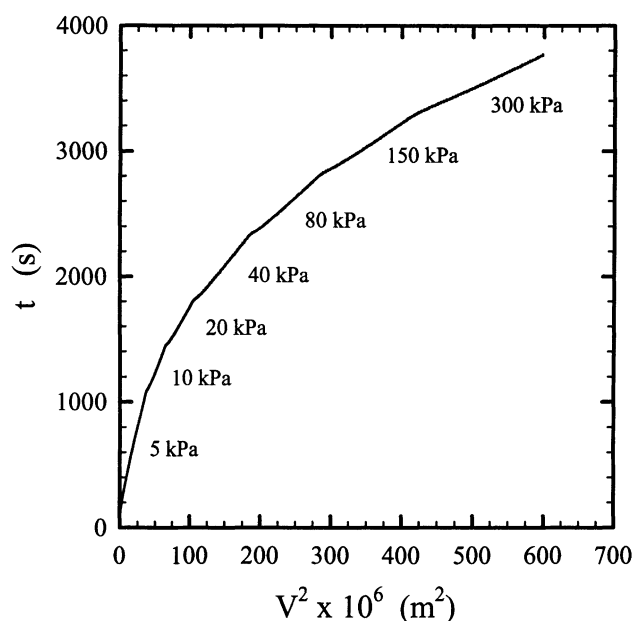


Figure 7. Example of t vs. V^2 for a stepped pressure filtration permeability test.

Zirconia, pH = 6.8, $\phi_o = 0.073$, $h_o = 0.06185$ m.

the first pressure, is ϕ_o from the preceding pressure. It is possible that the effects observed by Green and Boger came out of either the data manipulation required for processing results from the centrifuge technique employed, or from effects relating to the geometry of application of the compressive force which is very different to that in the filtration cell. A major advantage of the filtration technique is the absence

of data manipulation in determination of $P_y(\phi)$. Data are available in the literature comparing centrifuge and filtration results for $P_y(\phi)$ which indicate an absence of a ϕ_o effect and agreement between the two techniques (Miller et al., 1996). Additionally, Channell et al. (2000) observed that through introducing heterogeneities into a suspension, a ϕ_o effect could be induced in $P_y(\phi)$, however, it was also observed that the use of high shear in the preparation of a suspension removed any heterogeneities and effect of ϕ_o . As the sample preparation method in the current study involves the use of high shear homogenization, it seems unlikely that sample heterogeneities will exist. Data will be presented later that supports the absence of a ϕ_o effect in the present study.

Figure 7 presents a typical t vs. V^2 trace for a stepped pressure permeability test at the same pressures as the compressibility test in Figure 5. It can clearly be seen that the slope of the plot decreases as the pressure is increased. Close inspection reveals a short region of nonlinear filtration at the onset of each new pressure, which is in line with the theoretical validation described in the theory section and in a separate article based on modeling of the filtration behavior during a pressure step (Usher et al., 2001). The important point to note is that the permeability test must be completed entirely while the filtration is in the linear, cake formation region. Figure 8 shows a comparison of values of the slope ($1/\beta^2$) of the t vs. V^2 trace for stepped pressure and single pressure tests. At high pressures, the agreement is very good while at lower pressures there is some scatter in the data. However, there is no trend, and this suggests that the scatter is the result of random experimental error.

With the reduction in the scale of the filtration rig, error considerations have become more important in the treatment of data and efforts to reduce errors are ongoing. Pressure

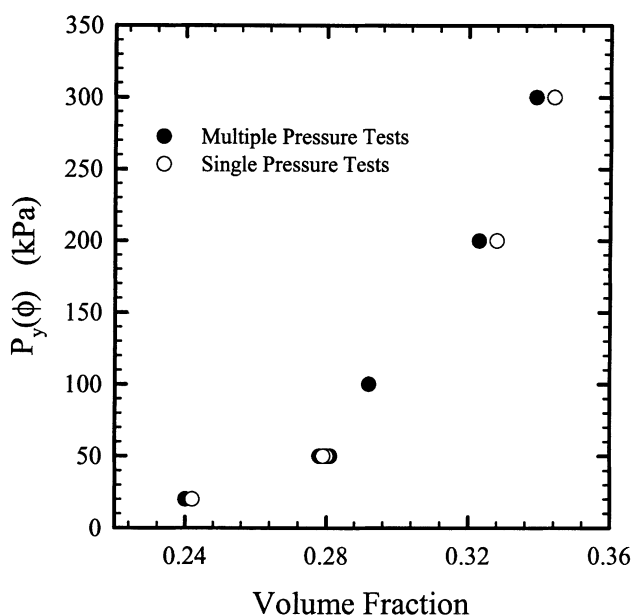


Figure 6. Comparison of $P_y(\phi)$ data obtained from both stepped and single pressure filtration tests.

Zirconia, pH = 6.8, $\phi_o = 0.098$.

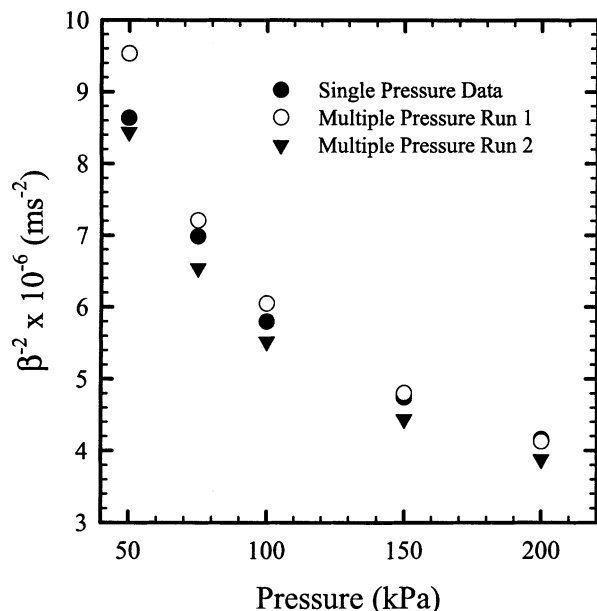


Figure 8. Comparison of $1/\beta^2$ data obtained from both stepped and single pressure filtration tests.

Zirconia, pH = 6.8, $\phi_o = 0.10$.

drop errors will be most significant for data obtained at lower pressures due to the limits of the pressure controller employed and the self-weight of the suspension being tested becoming a factor in the driving force for filtration. At present, an approximate correction to the applied piston pressure is made based on the amount of material present in the filtration cell, however, an accurate correction requires prior knowledge of the $P_y(\phi)$ behavior and ϕ_g of the sample being tested which, in most cases, are unavailable.

Additionally, for smaller cake heights, the expansion of the frame of the rig under applied pressure is significant and requires correction. At maximum pressures, the rig frame expands by around 0.3 mm which is significant when cake heights of 5 mm and under are considered. A correction is utilized to take into account the expansion of the rig frame which was found to be linear with applied pressure.

A number of errors affect the slope of the t vs. V^2 plot which, according to theory, should be constant. In fact, accounting for the contribution of errors using Darcy's Law leads to the slope being equivalent to

$$\frac{dt}{dV^2} = a + \frac{b}{V}.$$

Thus, dt/dV^2 has a V^{-1} dependence which diminishes as V becomes larger. Two main errors contribute to the second term in the equation above: membrane resistance and the presence of air bubbles, which are compressible, in the sample.

The effect of membrane resistance is small for larger pressures or materials with a high value of $R(\phi)$ in the cake, but may be significant at low pressures, specifically at early experimental times. In a stepped pressure permeability test, the

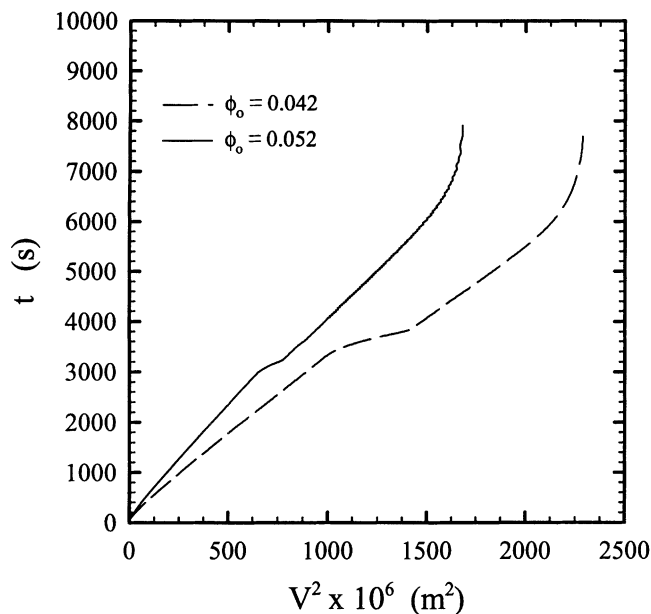


Figure 9. Unusual filtration behavior observed in 50 kPa single pressure filtration tests on zirconia at $\phi_o < \phi_g$.

$h_o = 0.05710$ m for $\phi_o = 0.042$ and $h_o = 0.05035$ for $\phi_o = 0.052$.

effect of membrane resistance will constantly decrease throughout the test; however, for the presence of air bubbles, each time the pressure is increased, the air compresses further and an additional error is added. Correction for both of these errors can be estimated and, specifically, for the presence of air bubbles, work is underway to incorporate the effect into the data analysis. The relative percentage of the errors are also minimized by using large sample heights in the filtration cell for the permeability tests.

Figure 9 shows results for single pressure filtration of zirconia with different values of ϕ_o at 50 kPa. The results show odd filtration behavior at intermediate times with the presence of a period of rapid filtration (lower slope in t vs. V^2). Such behavior was observed in results obtained by Green et al. (1998) and was attributed to settling of the suspension within the filtration cylinder or channeling effects. The current results suggest that, as a rapid filtration region was not present when $\phi_o > \phi_g$ (c.f. Figure 2) the phenomena is directly attributable to sedimentation of the suspension within the filtration cylinder during the time frame of the experiment. Unpublished results using a clear filtration cylinder confirm this conclusion.

The onset of the region of rapid filtration is associated with the upwards growth of the filter cake intersecting the descending interface between the settling suspension and clear liquid supernatant. Afterwards, the filtration rate increases as the cake is no longer growing and liquid is being pushed through the cake under pressure. Once the liquid has been expelled and the piston hits the top of the filter cake, compression of the cake commences and the filtration slows. Following this description, it is expected that the filtration rate dV/dt within the rapid filtration region should then remain

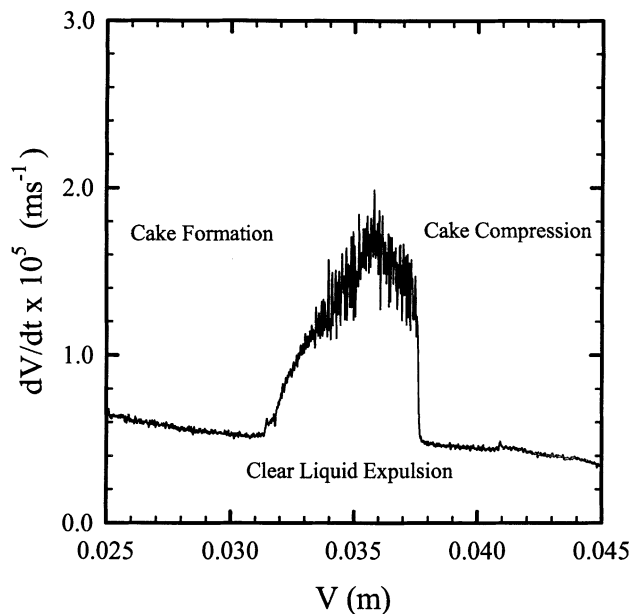


Figure 10. dV/dt for single pressure filtration data for zirconia in the region of rapid filtration.

$\Delta P = 50$ kPa, $h_o = 0.05710$ m, $\phi_o = 0.042$.

constant as described by Darcy's Law for packed bed filtration, where t varies directly with V . Analysis of sedimentation within filtration has been completed by a number of workers (Bockstal et al., 1985; Landman and Russel, 1993; Tiller et al., 1995) with experiments and predictions showing that dV/dt stabilizes to a constant value equal to dV/dt at the onset of region of clear liquid expression. Figure 10 shows close analysis of the slope of t vs. V for the filtration of zirconia at $\phi_o = 0.042$ in the region of rapid filtration. It is clear that dV/dt is not constant in the region of rapid filtration and, in fact, the filtration rate increases above the predicted Darcian rate (this is representative of observations with all zirconia tests for $\phi_o < \phi_g$). The most feasible explanation for the increase in filtration rate is the presence of channels in the cake that allow more rapid exit of liquid than is theoretically predicted. Why such an observation appears to be unique to this work is a subject of further investigation.

It should be noted that the filtration results obtained by Green et al. (1998) which showed very large regions of rapid filtration were for single pressure filtrations of zirconia for $\phi_o = 0.05$ with experimental times of up to 40 h. For such time spans, the suspensions would have settled almost to an equilibrium volume fraction within the cylinder, before the rising cake met the falling sediment interface. Thus, the rates of filtration within the cake formation region, prior to the onset of rapid filtration, would be consistent with filtration of an initial solids equivalent to the settled layer solids, not the actual value of $\phi_o = 0.05$. In the calculation of $R(\phi)$ in Green et al. (1998) an initial solids concentration dependency was observed (Figure 7a in Green et al. (1998)). It seems likely that this effect is actually an artifact of the discrepancy between the assumed ϕ_o and the actual ϕ in the sediment which would have dictated the filtration rate in the linear, cake formation region.

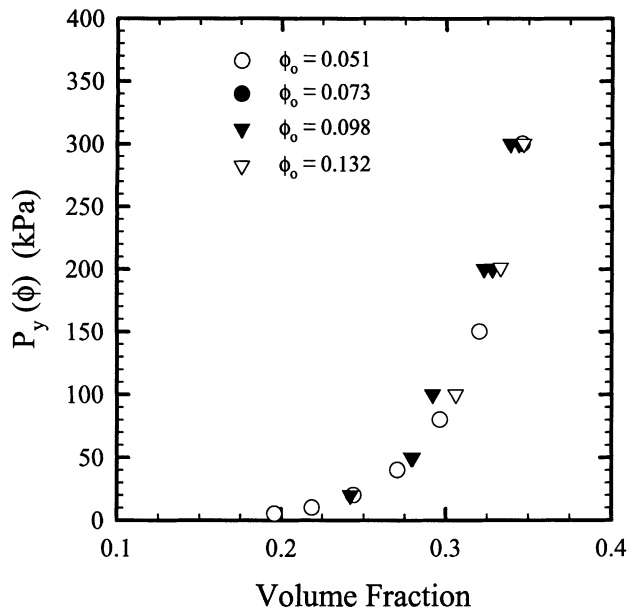


Figure 11. $P_y(\phi)$ data from stepped pressure filtration tests for a range of initial solids concentrations.

Zirconia, pH = 6.8.

In spite of the differences between previous results and the current work, the importance of awareness of the consequences of competing time frames for sedimentation and filtration for $\phi_o < \phi_g$ has been illustrated. The main conclusion for the present work is that, generally, dewatering parameter data derived from filtration results will be more reliable for tests where $\phi_o > \phi_g$.

Determination of the parameters

Figure 11 shows $P_y(\phi)$ data obtained from stepped pressure filtration of zirconia samples at initial volume fraction solids of 0.051, 0.073, 0.098 and 0.132. Clearly, the agreement between all of the samples is very good and there is no evidence of the presence of an initial solids concentration dependency in $P_y(\phi)$.

Calculation of $R(\phi)$ is completed by plotting β^2 data against pressure, fitting with an appropriate functional form and then determining $d\beta^2/dP$ for use in Eq. 1. In practice, the data are fitted with a power law, which fits the data well at intermediate pressures, but tends to overestimate $d\beta^2/dP$ at high pressures. Data used in this work were fitted with a power law, but currently work is underway to more accurately fit the β^2 data, as long-term development of the technique will require more accurate determination of $d\beta^2/dP$ and therefore $R(\phi)$. Calculated results for $R(\phi)$ for the zirconia suspensions at initial solids of 0.051, 0.073, and 0.098 are presented in Figure 12. Very good agreement is observed between samples where $\phi_o > \phi_g$ and reasonable agreement is exhibited for the sample with 0.051 initial solids concentration. It is quite possible that the discrepancy with the lower initial solids data is due to small amounts of settling occurring during the course of the permeability tests. As the set-

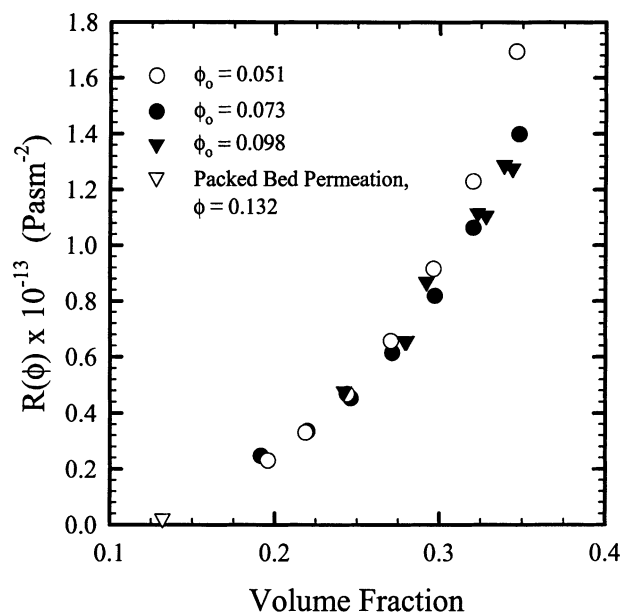


Figure 12. $R(\phi)$ data for a range of initial solids concentrations from stepped pressure filtration tests including data from packed bed permeation.

Zirconia, pH = 6.8.

ting is a progressive process, its effect would be expected to become more significant at larger times in a permeability test, that is, at higher pressures or final solids concentrations. It is evident from Figure 12 that the disagreement between results widens at higher solids concentrations, supporting this postulate.

Note that in Figure 12 there is also an $R(\phi)$ value determined by simple packed-bed permeation of liquid under gravity through a 0.132 volume fraction solids bed similar to the manner described in Green (1997) and Landman and White (1992). A procedure has been developed whereby $R(\phi)$ values may be determined for very low solids concentrations since, in modeling of dewatering processes, the form of $R(\phi)$ data at low solids is very important. The data at low solids concentration agrees well with the stepped pressure filtration results at higher solids concentrations. Full treatment of the characterization of both $P_y(\phi)$ and $R(\phi)$ at low solids concentrations will be presented elsewhere.

Figure 13 shows the calculated solids diffusivity during filtration $D(\phi)$ for different initial solids concentrations. All of the results were calculated using Eq. 3 which requires differentiation of only the β^2 data as a function of ϕ . While agreement is good at lower solids, the data diverge at higher solids with a spread of around 33% at high solids. The calculation of $D(\phi)$ effectively depends on the shape of the $P_y(\phi)$ curve and very small variations in the calculated values of ϕ_o at higher pressures can dramatically affect the slope of this curve. Thus, it is apparent that small errors in the data obtained from the filtration rig can contribute to a large variation in calculated results at high solids and, by definition, high pressures.

Physically, the implications of the data in Figure 13 are that the solids diffusivity consistently increases with solids

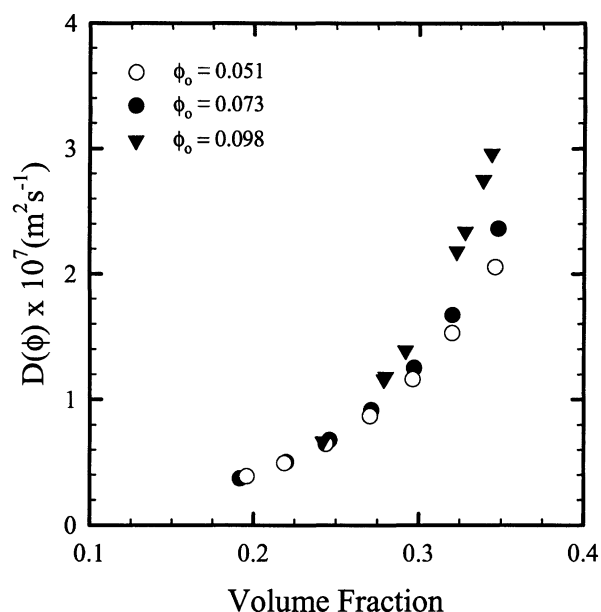


Figure 13. $D(\phi)$ data for a range of initial solids concentrations from stepped pressure filtration tests.

Zirconia, pH = 6.8.

concentration. While seeming counter-intuitive, it must also be considered that each value of solids corresponds to an applied pressure dictated by the form of the $P_y(\phi)$ curve. Plotting of $D(\phi)$ vs. pressure as in Figure 14 indicates that $D(\phi)$ plateaus at higher pressures indicating limiting benefits in increased dewatering rates with increased pressures. $D(\phi)$

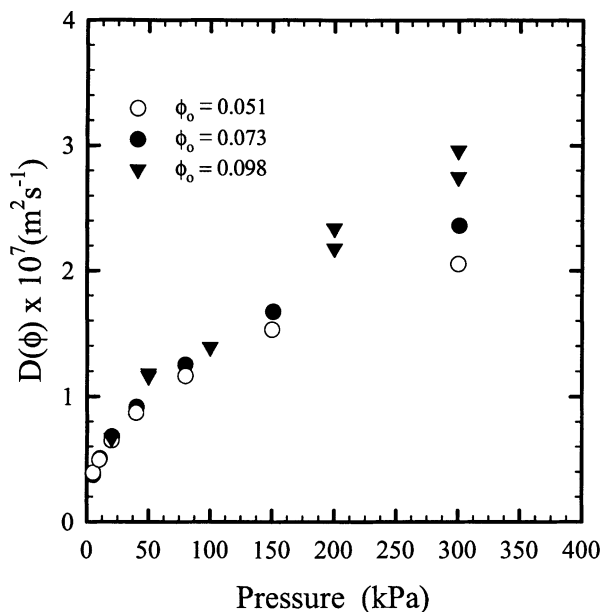


Figure 14. $D(\phi)$ vs. P data for a range of initial solids concentrations from stepped pressure filtration tests.

Zirconia, pH = 6.8.

results for a wide range of materials have been obtained and, in general, the form of $D(\phi)$ seen here is nearly always observed. However, results from sewage sludge indicate that for highly compressible materials which form very impermeable cakes, there may actually be a maximum in the $D(\phi)$ plot with pressure, indicating an optimum pressure for operation of dewatering processes. These results are to be presented in a separate article. In terms of comparison of dewatering rates of different materials, the material that has a higher solids diffusivity at the desired final solids will always be the fastest to dewater. It is in this way that the values of $D(\phi)$ may be used for optimization purposes.

Design applications

The results in Figures 11 through 13 may be used in modeling and design of dewatering processes. The mathematical framework now exists for the modeling of not just filtration processes (Landman and White, 1997) but also batch sedimentation (Howells et al., 1990) and continuous thickening (Landman et al., 1988) using $P_y(\phi)$, $R(\phi)$ and $D(\phi)$. In the theory section, equations were presented that described the relationship between $D(\phi)$ and T_f and t_f , the scaled (dimensionless) time and real time of filtration respectively.

The results for zirconia (determined at an initial solids volume fraction of 0.073) from the filtration tests were input into the Mathematica program incorporating the theory described by Landman and White (1997). The program was used to simulate a real single pressure filtration test which was conducted on the filtration rig using zirconia for the test conditions: $\Delta P = 100$ kPa, $\phi_o = 0.1316$ and $h_o = 0.02122$.

Figure 15 shows the comparison of the experimental vs. predicted t vs. V^2 behavior for the 100 kPa filtration for the 0.0132 initial volume fraction zirconia. The agreement is very

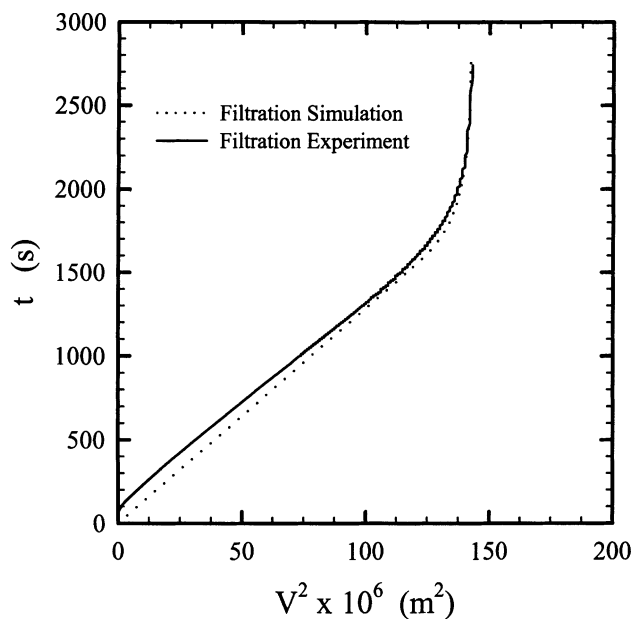


Figure 15. Comparison of simulated and experimental data for 100 kPa, single pressure filtration of zirconia.

Zirconia, pH = 6.8, $\phi_o = 0.1316$, $h_o = 0.02122$.

good with predicted vs. experimental times being close, particularly within the compression zone with the end point of the test being predicted by the simulation. The good agreement is especially pleasing given the utilization of data obtained from filtrations at $\phi_o = 0.073$ in prediction of filtration behavior at a different ϕ_o . The small discrepancy between the data in the cake formation region at early times is due, in part, to the “nonideal” conditions present in a real filtration, that is, application of full pressure in this experiment took 150 s. Additionally, the simulation takes no account of the influence of membrane resistance or other error factors.

Note that the calculation of filtration times from $P_y(\phi)$, $R(\phi)$ and $D(\phi)$ data within the filtration simulation program requires fitting of a functional form to the discrete data obtained from stepped pressure filtrations and can be sensitive to the form of fit, especially at low volume fractions. Thus, in calculations, the gel point and $R(\phi)$ determined via packed-bed permeation can be utilized to improve the accuracy of the low volume fraction $P_y(\phi)$ and $R(\phi)$ data, respectively. Detailed treatment of the calculation of filtration times from the experimental data is the subject of ongoing work.

The real power of the theoretical framework described lies in the ability to optimize the operation and design of filter presses, given the fundamental parameter data. For example, given a fixed, desired final solids concentration, the effect of changing operating parameters such as filtration pressure and feed solids on filtration cycle times may be evaluated. In a batch operation, Landman and White (1997) described optimization of a filter for maximum throughput by altering the initial charge of material fed to the filter. Analysis of optimum operating conditions for maximum throughput can also be evaluated for continuous filtration processes.

Figure 16 shows the effect of changing operating pressure and feed solids on scaled filtration times (T_f) for zirconia filtration to a target final volume fraction solids of 0.28. The results are all calculated for the same amount of feed suspension, so in terms of solids throughput, rather than filtration cycle time, the results would scale with the feed solids. However, on the basis of a constant total amount of feed suspension, the results indicate that, at low pressures, the feed solids of 0.25 will actually take longer to reach a final solids of 0.28 than the lower feed solids materials. At intermediate pressures, the feed solids that gives the lowest filtration cycle times changes depending on the pressure. Such analysis of the effects of changing process parameters is useful for both design in terms of selecting the operating pressure of the filter and, also, in the optimization of the operation of the filter in terms of predicting the effect of fluctuations in feed solids on cycle times or final solids.

While optimization and design in filtration is one major outcome of the present work, wider applications lie in the input of the dewaterability parameters described into models for the design and operation of batch sedimentation processes, clarifiers and thickeners and centrifuges. Work is progressing in these areas.

Conclusions

A new technique for the rapid characterization of the fundamental dewatering parameters $P_y(\phi)$ and $R(\phi)$ and the derived parameter $D(\phi)$ has been described. Characteriza-

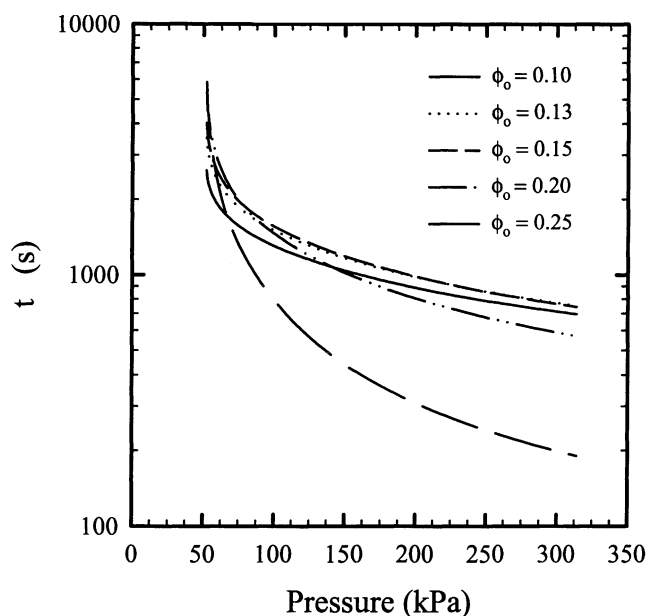


Figure 16. Simulated filtration data for zirconia single pressure filtration to $\phi_f = 0.28$ for various ϕ_o and applied pressures.

Zirconia, pH = 6.8, $h_o = 0.02122$.

tion times for materials using stepped pressure filtration have been reduced relative to conventional filtration methods such that data may be obtained in hours rather than days, rendering the technique useful for many dewatering optimization applications.

In conjunction with the Landman-White filtration theory, characterization and optimization of the dewatering properties of suspensions may be carried out nonempirically. Additionally, modeling of real filtration behavior using data obtained from stepped pressure filtration was carried out and the agreement was excellent. A framework now exists that allows both optimization of operation and design of new equipment for not just filtration, but all dewatering operations.

Acknowledgments

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