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## USING MECHATRONICS FOR THE INTERPRETATION AND MODELLING OF THE PRESSURE FILTER CYCLE

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### SUMMARY

An experimental apparatus combining the principles of mechatronics with a dead-end pressure leaf filter is described. The computer controlled and sequenced apparatus was used to monitor performance during the cake formation, gas dewatering and displacement washing phases of filter cycles. Data relating to cake properties and liquor transport rates were acquired through electrical resistance measurements and suitable transducers. The data illustrate the versatility and accuracy of the apparatus and they are used to compare with predictions given by existing theories, particularly for cake formation. Batch experimental results obtained with aqueous suspensions of calcite are shown to compare favourably with theoretical predictions for important design parameters such as cake height and cumulative volume of filtrate whereas data for more compressible talc systems tended to show more deviation. The work presented highlights the benefit of a mechatronics approach to experimentation and emphasises the importance of data acquisition and system control to future developments in filter design.

### KEYWORDS

Filter cycle; Mechatronics; Filtration; Washing; Dewatering

### INTRODUCTION

The engineer of today is required to have a knowledge of many types of unit operation. Whilst he or she can rely on extensive databases and simulation packages for the detailed design of equipment such as distillation columns, the specification of apparatus for the separation and subsequent processing of solid/liquid mixtures is more problematical. If one restricts choice to those separations involving cake filtration, it is possible to identify a ranked list of equipment ranging from belt and rotary drum filters to leaf and diaphragm press filters to perform a given duty<sup>1,2</sup> (see Figure 1).

The inherent complexity and transient nature of the separation sequences, potentially involving cake formation, cake consolidation, gas dewatering and/or displacement washing, would normally require that many (costly) experiments be performed before a final choice of equipment is made. The problems of performing experiments in a reliable and repeatable manner are well known, if not always well documented, for all scales of operation with the result that models are frequently only applicable to the apparatus on which the experiments have been performed. Difficulties such as these have hampered solid/liquid separation research for many years and, despite a number of significant contributions by researchers in cake filtration<sup>3-10</sup>, post-treatment<sup>11-13</sup> and simulation<sup>14-20</sup>, there is still much reliance on heuristics or 'rules-of-thumb' in the design and specification of solid/liquid separation devices.

Of primary interest in this paper are the cake filtration, gas dewatering and displacement washing operations in a filter cycle. Cake filtration, which relies on the preferential entrapment of particulates upon a semi-permeable medium, is a common form of separation used in most of the chemical process industries. The filter medium is relatively thin and provides the separating surface only at the initial stage(s) of filtration, after which it acts primarily as support to the growing

cake. The relationship between the filtrate flow rate ( $dV/dt$ ), the applied pressure difference ( $\Delta p$ ) and the filter cake properties can be expressed through a modified form of Darcy's law

$$V_f = \frac{1}{c_{c,av} \alpha_{av}} \left( \frac{A^2 \Delta p}{\mu} \frac{dt}{dV_f} - AR_m \right) \quad (1)$$

where  $V_f$  is the cumulative volume of filtrate,  $t$  is time,  $c_{c,av}$  the average effective feed concentration,  $\alpha_{av}$  the average specific cake resistance,  $A$  the filter area,  $\mu$  the filtrate dynamic viscosity and  $R_m$  the filter medium resistance. Whilst eqn. (1) may be used to interpret filtration data, for instance from the integrated form at constant pressure through a  $t/V$  vs.  $V$  plot, its applicability has been questioned on occasion as it does not take into account all the factors known to influence cake formation processes<sup>21-24</sup>. Although the concept of specific cake resistance provides an indication of the ease by which a separation can be achieved, its use becomes more abstract when a degree of compressibility exists in a growing cake. When compressibility is present, as it is in the majority of practical filtrations, cake resistance changes with height throughout the cake and local values of cake resistance must be known to correctly apply eqn. (1) through the relations

$$\alpha_{av} = \alpha_0 (1-n) \Delta p_c^n \quad (2)$$

$$e_{av} = e_0 - b_1 \log(\Delta p_c) \quad (3)$$

where  $\Delta p_c$  is the pressure gradient across the cake,  $e$  is the cake voids ratio and  $\alpha_0$ ,  $n$ ,  $e_0$  &  $b_1$  are empirical constants derived from sequences of constant pressure experiments. The need to know the relationships generally becomes more important as the cake compressibility increases. Moreover, it has been recognised that the interactions between the cake and the filter medium can change both the manner in which a filter cake forms and the solids concentration profile obtained therein<sup>25</sup>.

Once a filter cake is formed there is frequently a need to post treat it by, for instance, gas dewatering and/or displacement washing. In the former a gas stream is applied to the cake top surface to force liquor through the cake interstices whilst washing requires the displacing fluid to be a 'clean' liquid. If a simplistic model of straight, uniform capillaries is assumed then the displacing fluid would ideally move through the bed as a uniform front. In reality, however, the cake interstices are characterised by a tortuous nature where a fluid element passing through the structure travels a greater distance than if the structure was formed from parallel capillaries. The value of the tortuosity factor depends upon the path travelled by the fluid element, which in turn, depends upon the structure of the porous medium. As all filter cakes exhibit degrees of irregularity and tortuosity, the induced dispersion can become important to the extent that poor dewatering and washing are observed. Such problems become extreme when channelling occurs. Here, one or more regions of a filter cake exhibit significantly lower resistance to fluid flow where a high proportion of the displacing fluid preferentially passes through, leaving area(s) of the cake essentially untouched. The degree of cake inhomogeneity is influenced by the manner in which a filter cake forms and the irregularly shaped particles encountered in solid/liquid separation are known to introduce an element of randomness to the process of bed formation due to their structural and orientation anisotropy<sup>26</sup>.

The above considerations suggest that in order to remove heuristics and develop reliable design procedures for solid/liquid separation equipment it is necessary to systematically acquire and then interpret accurate experimental data for all phases of filter cycles. The ability to interrogate particle/fluid motions is a pre-requisite to this, as is the ability to provide a test rig which removes as much transient operator interference as possible. The recent advent of mechatronics, and the

trend towards non-invasive scanning techniques<sup>27-32</sup>, has presented separations technologists with the opportunity to examine the filter cycle in new and novel ways.

In the work presented here, mechatronics, which integrates electronics and computer technology with applications requiring the control of mechanical systems and processes, have been utilised to provide an apparatus capable of examining each phase of a sequential filter cycle. A detailed description of the apparatus is presented, along with samples of the data which have been generated. Some of these data, in particular those obtained for cake filtration, are compared with predictions given by existing theories.

## EXPERIMENTAL PROCEDURES AND APPARATUS

The experimental investigation of the filter cycle involved the use of aqueous mineral suspensions and suitable filter media. To aid the interpretation of the filter cycle tests it was necessary to first identify and subsequently characterise suspensions and media. The characterisation tests were performed using standard laboratory equipment including that commercially available from Malvern Instruments™ and Coulter Electronics™.

### Suspension Characterisation

Some initial (constant pressure) filtration tests using suspensions of the mineral powders calcite and talc dispersed in distilled water showed the formation of nearly incompressible and moderately compressible filter cakes respectively. SEM's of the calcite powders indicated a rhomboidal particle shape with a 50% size of 11.3  $\mu\text{m}$  as measured by laser light scattering (see Figure 2). Measurements of zeta ( $\zeta$ -) potential indicated the iso-electric point (IEP) to be in the region of pH = 9.0 and calcite to be typical of a relatively low surface charge material in the chosen distilled water environment. Talc, by contrast, exhibited a plate-like shape and a 50% size of 8.5  $\mu\text{m}$ . In this case an examination of the  $\zeta$ -potential variation with pH showed the IEP to occur at a pH = 2.0 and a maximum (-ve)  $\zeta$ -potential = -55 mV to occur at pH = 11.0 when Analar grade HCl and NaOH were used to alter pH. The addition of a  $\text{NaNO}_3$  salt tracer (to track the progress of displacement washing operations in the filter cycle tests) was shown to have a negligible effect on measured  $\zeta$ -potentials.

The calcite and talc systems were chosen for their different natures, the ability to characterise properties and their representation of 'real' suspensions with size distribution.

### Membrane Characterisation

In order to provide for cake filtration, and hence limit the influence of internal pore fouling, it was necessary to preclude as many particulates as possible from the septum during filtration. This was achieved by using 0.2  $\mu\text{m}$  rated Gelman™ Versapor membranes of 185  $\mu\text{m}$  thickness as filter media. Porosimetry tests indicated 10, 50 and 90% pore sizes of 0.17, 0.25 and 0.30  $\mu\text{m}$  respectively for a typical membrane sample (see Figure 2) and negligible changes in measured pore sizes to occur when samples of membranes were tested after being challenged with process suspension.

### Filter Cycle Apparatus and Test Procedure

The principal hardware used in the filter cycle apparatus comprised a dead-end pressure filter cell and electronic circuitry to provide electrical resistance measurements<sup>33,34</sup>. Referring to Figure 3, the filter cell of area 80  $\text{cm}^2$  was constructed from stainless steel (s/s) and designed and tested in accordance with BS5500 for operation at internal pressures up to 1000 kPa. The cell allowed for the installation of a membrane and incorporated sixteen horizontally oriented rings of electrodes situated at intervals along its length. The first six rings were equi-spaced at 10 mm intervals above

the septum, the next five were equi-spaced at 20 mm intervals, with the final five rings equi-spaced at 30 mm intervals. Each ring contained sixteen electrodes evenly spaced around the inner periphery of the cell, with every electrode protruding 3 mm from the internal cell wall.

Electrode pairs were switched via dedicated electronic circuitry and used to give an indication of cake structure, cake saturation and/or suspension status at pre-determined times for each phase of a filter cycle test. For suitable measurements to be taken each electrode was able to either generate an electrical current (termed pulsed), receive a current (termed earthed) or be neither pulsed nor earthed (termed floating). The co-ordination of the electrodes was performed using a system of CAD/CAM designed and manufactured printed circuit boards. A master board and sixteen daughter boards were developed in conjunction with a mimic display board and two interface circuit boards. Their combination allowed data transfer to and from a dedicated personal computer fitted with Advantech™ PCL-812PG and PCL-860 DVM add-on cards such that a digital signal could be used to switch electrodes to the pulsed or earthed states. The procedure allowed an alternating electric current, provided by a 2 kHz square wave generator having a mark-space ratio of unity, to pass between a chosen pair of electrodes. The voltage generated across each electrode pair in the filter cell was measured and calibrated to the solids concentration<sup>35</sup> prior to experiments using a combination of the add-on cards installed in the computer and software specifically developed for the apparatus. In this manner it was possible to measure transient liquid and solids concentrations throughout the cell, gauge changes in cake height and thus determine true average values for parameters such as cake porosity by numerical integration<sup>36</sup>.

The remainder of the experimental apparatus is shown schematically in Figure 4. It comprised two s/s storage vessels, incorporating temperature sensors and stirrers, connected by s/s piping to the filter cell. The vessels stored the feed suspension and wash water respectively. Dried and filtered compressed air at pressures up to 800 kPa was supplied to the control valves on the rig pipework and also fed to the storage vessels and filter cell. The latter was relayed via an electronic pressure regulator such that the driving pressures for all phases of a cycle were provided. The mimic display panel indicated which electrodes were pulsed and earthed within the filter cell at any time and a heater/cooling system regulated the temperature of the filter cell and storage vessels by continuously passing heating/cooling water through their surrounding jackets. This counteracted any fluctuations in ambient temperature and eliminated temperature transients induced by slurry and liquor flow during an experiment. Filtration was initiated by feeding air through the storage vessel to force suspension into the filter cell at constant pressure. To perform a dewatering operation, compressed air was introduced directly into the filter cell at the desired pressure and its flow rate monitored using an electronic rotameter. When a washing phase was included in the filter cycle air was again introduced through the storage vessel and liquor samples were taken using a twenty interval, rotary indexing table situated directly below the filter cell; the samples being analysed after a test had ended by conductivity measurements. An electronic balance, also situated below the filter cell, enabled liquor transport rates to be monitored semi-continuously and mass balance calculations to subsequently be performed. All components related to data acquisition and rig operation were sequenced by the computer through dedicated computer software.

Prior to the start of a batch experiment the feed suspension was made to a known solids concentration between 5 and 20% v/v from a dry powder and distilled water with up to a 1.5 g l<sup>-1</sup> NaNO<sub>3</sub> salt tracer being added to allow a washing phase to be monitored. The pH was adjusted when necessary by the addition of analar grade HCl or NaOH. No other dispersants or flocculants were added and the mixture was stirred in the storage vessel for a suitable period to produce a homogeneous mixture at the desired temperature (generally 25±0.5°C). During the stirring period, fluids were continuously pumped around the heater/cooler circuit to equilibrate temperatures. With a membrane installed in the filter cell the chosen filter cycle conditions were programmed into the computer and the test performed automatically. Data relating to flow conditions and cake structure/saturation were displayed in real time during a test, with a more comprehensive dataset being permanently stored by the computer on disk for future reference. A filter cycle would always

include an initial cake formation phase at constant pressure, followed by the chosen combination of washing and dewatering.

## FILTER CYCLE EXPERIMENTAL RESULTS

The experimental results presented are representative of more than 100 filter cycle tests and these conveniently divide into four sections. The first three sections examine data for the individual cake formation, dewatering and washing phases respectively, whilst the fourth shows some data for sequential combinations of all phases. All experiments were performed over the pressure range 0-600 kPa with a pH = 9.0 for calcite feeds and a pH = 6.5 for talc feeds.

### Cake Formation (Filtration) Phase

It has long been recognised that it is difficult to generate reliable and repeatable experimental data for cake formations at constant, and indeed variable, pressures<sup>37,38</sup>. Figures 5 and 6 suggest that with the apparatus developed, where operator interference has essentially been removed, it is now possible to provide more accurate experimental data for constant pressure filtrations over a range of conditions. The sample data presented for 10% v/v calcite and talc suspensions, and pressures between 50 and 600 kPa, show sequential changes on the standard  $t/V$  vs.  $V$  plots (linear regressions of these and corresponding  $dt/dV$  vs.  $V$  plots all yielded coefficients of determination ( $r^2$ ) in excess of 0.99). As the test pressure was raised, so the gradients of the data sequences decreased correspondingly. This is in agreement with the integrated form of eqn. (1) which for the calcite data gives average specific cake resistance ( $\alpha_{av}$ ) values over the range  $1.1 \times 10^{10}$  to  $1.5 \times 10^{10}$  m kg<sup>-1</sup> with the calculated filter medium resistance ( $R_m$ ) varying between  $1.1 \times 10^{10}$  and  $2.0 \times 10^{11}$  m<sup>-1</sup>. The corresponding data for talc showed  $\alpha_{av}$  to vary between  $4.3 \times 10^{10}$  and  $1.9 \times 10^{11}$  m kg<sup>-1</sup> with  $R_m$  apparently taking values between  $3.8 \times 10^{10}$  and  $3.0 \times 10^{11}$  m<sup>-1</sup>. The latter values for  $\alpha_{av}$  emphasise the more compressible nature of the talc system. The apparent variations in  $R_m$  are considered to be due to a degree of filter medium compression<sup>39</sup>. Other sequences of constant pressure tests at feed suspension concentrations of 20% v/v confirmed the trends in the data shown.

It was previously described how, by switching series' of diametrically opposite electrode pairs within the filter cell, transient solids concentration profiles could be measured throughout cake formation. Figure 7 shows a typical example for a 10% v/v calcite suspension filtered at a pressure of 400 kPa. Here scans, taken over four distinct vertical planes towards the end of filtration when  $t_f = 560$  s, illustrate the (expected) incompressible and relatively homogeneous nature of the cake. By examining similar data at various times during filtrations it was seen that as a cake formed so the solids concentration increased rapidly from 10% v/v to approximately 38% v/v and then remained essentially constant at that value irrespective of the applied filtration pressure. The accuracy of such data were confirmed by mass sampling<sup>†</sup> and their form contrasted sharply with the more compressible cakes formed during the filtration of talc suspensions. For the example data shown in Figure 8, solids concentrations are seen to rise relatively slowly from the feed concentration of 10% v/v to a range of cake concentrations up to 38% v/v depending on the height above the filtering surface. With talc, and the range of pressures between 50 and 600 kPa, the cake solids concentration at a distance of 1 cm above the septum reached values between 30% v/v and 41% v/v respectively after approximately 2000 s filtration. The continual rearrangement of the particulates in the cake during filtration is a result of the drag force exerted by the flow of liquid through the cake interstices; the degree of rearrangement being a function of the cake compressibility. With calcite, little rearrangement of particles was seen once the cake had formed, however, talc cakes were generally seen to change structure throughout a filtration.

<sup>†</sup> Mass balance comparisons between numerical integrations of local solids concentrations over the cake height and the known mass of solids used in an experiment typically showed differences of less than 5%.

As a consequence of measuring solids concentration profiles in the filter cell it was possible to evaluate changes in filter cake height with time. For calcite experiments it was relatively easy to determine the position of the filter cake surface as the solids concentration would change very rapidly during the transition from suspension to cake (assumed > 30% v/v). With talc, however, it was more difficult to identify the transition. Figure 9 shows some data for talc suspensions where the change of cake height with time as a function of pressure can be seen. The cakes, assumed to be present at concentrations greater than 30% v/v, formed more rapidly at higher filtration pressures as expected. Data showing similar trends were recorded for the filtration of calcite suspensions.

## Dewatering Phase

When a dewatering period was chosen to follow cake formation, data like that presented in Figures 10 and 11 were obtained. Figure 10 indicates that during filtration, data would normally show as a straight line on a  $t/V$  vs.  $V$  plot, however, when dewatering started a sharp increase in gradient would be observed. Here, the displacing gas was progressively removing less liquid from the cake as time proceeded, leaving the cake in an unsaturated condition. The volume of liquid extracted from a suspension by filtration and dewatering operations increased as the cycle pressure was raised.

For a cycle involving filtration and dewatering, Figure 11 shows the apparent solids concentration history over one scanning plane for a calcite filter cake/suspension as a function of time. During the filtration phase the concentration of solids within the saturated cake was essentially constant throughout its depth (c.f. Figure 7). For the experimental conditions employed the cake dewatering phase commenced when 1120 s had elapsed and the solids concentrations measured by the electrode pairs from this time increased sharply as the liquid was removed from the cake interstices. At the end of dewatering the reduced saturation of the cake resulted in apparent solids concentrations between 75% v/v and 90% v/v depending on the position within the cake. These values of solids concentration are misleading however, as the particle arrangement in the cake and the cake height were known to remain essentially unaltered during dewatering. Mass sampling of the cake at the end of the test showed the true solids concentration to remain at approximately 38% v/v. This was confirmed by re-plotting the data in Figure 11 to show cake moisture content as a function of time. The average moisture content at the end of filtration was in the region of 35% and those at the end of dewatering decreased from approximately 16% at the base of the cake to 10% towards the top. Moreover, for experiments involving calcite suspensions and dewatering, some evidence of non-uniform or preferential dewatering was observed. Tests using multiple vertical scanning planes showed this, with different saturation profiles being obtained in different planes as the dewatering process progressed<sup>34</sup>.

## Washing Phase

Filter cycle experiments involving cake formation and washing produced several results worthy of note. Figure 12 illustrates data for five experiments using calcite suspensions where cake formations at constant pressures of 300 kPa were followed by displacement washing operations at constant pressures between 100 and 600 kPa. The highly repeatable nature and accuracy of the experimental apparatus and procedures is again apparent from these data.

As the pressure was raised so the wash liquor flow rate usually increased. Analysis of the washing samples by conductivity measurements after an experiment allowed washing curves to be constructed. In summary these plots of dimensionless solute concentration ( $c/c_0$ ) vs. time ( $t$ ) showed that calcite cakes were washed more rapidly than talc cakes formed under similar conditions, and that between two and four wash ratio's were required to reduce  $c/c_0$  below 0.1. Moreover, by the appropriate switching of electrode probes in the cell it was possible to track the movement of the washing front through a cake. Although such data are very limited at present, Figure 13 shows an example where a talc cake was formed and subsequently washed at 50 kPa.

At the start of washing all the positions in the cake corresponded to the solids concentrations at the end of filtration and a measured conductivity of ~2 mS. Here the cake was in the saturated state containing mother liquor and the salt tracer. As the displacement washing proceeded those electrode pairs situated towards the top of the cake showed reducing conductivity due to the progressive movement of the distilled water wash front through the cake with time. Samples of washings taken using the rotary table confirmed that no breakthrough had occurred when the experiment was terminated after ~7000 s.

### Combined Filter Cycle

The data shown in Figures 5-13 are extracted from filter cycle experiments, generally involving all three potential phases. Whilst it is difficult to adequately present data for all three phases on one graph, Figure 14 shows cumulative volume of filtrate vs. time data for constant pressure cycles involving calcite suspensions and the sequence filtration - washing - dewatering. The progressive changes in the data are self evident and again highlight the benefit of being able to perform all operations in one apparatus.

## MATHEMATICAL MODELLING AND DISCUSSION

The use of heuristics in the selection and design of solid/liquid separation equipment has for many years provided the basis by which plant is specified. Whilst these 'rules of thumb' provide workable solutions to sometimes complex problems, their use does little to forward our fundamental understanding of separation processes. In order to develop these it is a prerequisite that the most accurate and representative experimental data are available as, ideally, are the models against which to compare and even predict them. The inherently transient nature of solid/liquid separation can make it very difficult to provide accurate and repeatable experiments at even a laboratory scale as the interference from an operator can significantly alter the results achieved. The experimental problems usually increase when separations are performed at larger scales where it becomes more difficult to handle the quantities of material involved.

One of the aims of developing the experimental apparatus described in this paper has been to generate (at a reasonable scale) accurate, and repeatable, experimental data against which theoretical models can be compared and developed if necessary. The data shown in Figures 5-14 indicate that the apparatus is capable of providing such data for a range of feeds and experimental conditions. In order to facilitate the comparison of experimental data with mathematical models a computer simulation of the pressure leaf filter cycle has been produced as a development of an author's previous work<sup>14,15</sup>. Briefly, the simulation is based on three (existing) models, with one model being applied to each of the three potential phases in a cycle<sup>†</sup>. The filtration phase has been modelled using so-called 'conventional' or 'classical' filtration theory modified in an attempt to account for cake compressibility such that

$$t_f = \frac{\alpha_{av} \mu \rho M_s}{2A^2 \Delta p (1 - M_s (1 + e_{av} (\rho/\rho_s)))} V_f^2 + \frac{\mu R_m}{A \Delta p} V_f \quad (4)$$

and

<sup>†</sup> The models used in the simulations have been chosen to illustrate what can be achieved by the approach taken. It is realised that in some cases they represent only a selection of the models currently available in the literature and more detailed comparisons of models with experimental data will be the subject of a future paper.

$$h = \frac{V_f (1 + e_{av})}{A \left( \frac{\rho_s}{\rho} \left( \frac{1}{M_s} - 1 \right) - e_{av} \right)} \quad (5)$$

$$M_c = \frac{Ah\rho_s}{1 + e_{av}} \quad (6)$$

where  $\rho$  is the filtrate density,  $\rho_s$  the solids density,  $M_s$  the solids mass fraction in the feed,  $h$  the cake height and  $M_c$  the mass of solids in the cake. As the amount of solids in a filtering suspension is known prior to an experiment, eqn. (6) can be used to indicate the theoretical end of filtration. To use eqns. (4)-(6) in the simulation it was necessary to determine values for  $\alpha_{av}$  &  $e_{av}$  via the empirical constants  $\alpha_0$ ,  $n$ ,  $e_0$  &  $b_1$  given by eqns. (2) & (3). It is conventional practice<sup>40,41</sup> to perform series' of constant pressure experiments to determine values for the empirical constants and this was done for calcite and talc suspensions over the pressure range 50-600 kPa using the apparatus described (denoted here as Apparatus 1). Further constant pressure experimental data from a completely separate apparatus (denoted Apparatus 2) were also available for calcite and zinc sulphide suspensions up to feed concentrations of 20% v/v. This apparatus was also computer controlled and employed a filter cell of area 22.8 cm<sup>2</sup> incorporating 32 pairs of electrodes arranged in a single vertical plane. Table 1 shows a summary of the empirical constant values obtained from the two apparatus'.

The dewatering phase of a filter cycle was modelled using a theory attributed to Wakeman<sup>11</sup>, the principal equations being

$$t_d = \frac{\mu e_{av} h^2 \rho_s \alpha_{av} (1 - S_\infty)}{\Delta \rho_c (1 + e_{av})^2} \left( \frac{1}{b_2} \left( \frac{1 - S}{S - S_\infty} \right) \right)^{-b_3} \quad (7)$$

$$V_d = \frac{A h e_{av} (1 - S)}{1 + e_{av}} \quad (8)$$

where  $S$  is the cake saturation,  $S_\infty$  is irreducible saturation and  $b_2$  &  $b_3$  are previously determined empirical constants whose values were known *a priori* for the dewatering of calcite and talc filter cakes<sup>42</sup>.

To simulate performance during a washing phase a dispersion model<sup>12</sup> was used. A combination of mass balance calculations gave expressions for the wash time and cumulative wash volume such that

$$t_w = \frac{W h e_{av} \mu (\alpha_{av} \rho_s h + R_m (1 + e_{av}))}{\Delta \rho_c (1 + e_{av})^2} \quad (9)$$

$$V_w = \frac{W h A e_{av}}{1 + e_{av}} \quad (10)$$

where  $W$  is the wash ratio.

The combination of eqns. (2)-(10) allowed the production of a flexible simulation which facilitated predictions of performance from virtually first principles for filter cycles involving combinations of

filtration, washing and dewatering. The experimental data shown in Figures 5-14 have been compared with their corresponding computer simulations and some of the results are shown in Figures 15-20.

Figure 15 shows the comparisons between simulation and experiment for cake growth at two different filtration pressures. The excellence of the predicted cake height with time is indicative of the accuracy of the simulation when essentially incompressible systems, such as calcite, are considered. Moreover, the theoretical calculation of height used values for the empirical constants  $\alpha_0$ ,  $n$ ,  $e_0$  &  $b_1$  determined from Apparatus 2 to predict the performance of Apparatus 1 at an, albeit marginally, different scale. To the authors knowledge, such predictions are rare in the literature<sup>43</sup>. It is noted that filters are often specified on the basis of cake thickness, and hence throughput, and to be able to predict these parameters accurately is obviously of great benefit to filter design and specification.

The data shown in Figure 16 for talc suspensions indicate that despite the careful control of the filtration, the theoretical predictions of cake height, and cumulative volume of filtrate, as functions of time are relatively poor. Earlier it was stated that with the compressible nature of talc it is difficult to identify the position of the interface between the cake and the filtering suspension and a somewhat arbitrary value of 30% v/v was chosen to be a minimum concentration for cake to exist. Assuming that the experimental data are accurate it must be concluded that either an incorrect value for the minimum cake concentration was chosen or the model used in the simulation is flawed. The data at the lower pressure suggest that a concentration higher than 30% v/v should be chosen to identify the presence of cake as this would improve the fit between the model and the experimental data. On the contrary, the data at the higher pressure suggest the opposite that a lower concentration should be chosen. This apparent anomaly is difficult to reconcile, though the concentrations measured in the talc experiments were typically below 40% v/v and it is perhaps difficult to conceive how a solids concentration of 30% v/v should not be considered to be cake.

'Conventional' filtration theory, and to some extent 'modern' filtration theory<sup>4,5,9,41,43</sup>, use the concept of average cake resistance and voids ratio (or porosity) as means of cake characterisation<sup>†</sup>. The need to do this demonstrates our difficulties in understanding cake filtration and the continued problems of identifying single parameters to represent filtration processes. The data shown in Figure 17, from experiments at high pressures with relatively compressible zinc sulphide cakes, further emphasises the difficulties as 'conventional' theory is again shown to under-predict cake height with time, whilst 'modern' filtration theory is worse (from a design viewpoint) in that it over-predicts cake heights. Since the advent of mechatronics it is now possible to generate reliable data at a reasonable scale against which theoretical models can be compared, and hence developed. The need to develop the models is self evident from Figures 16 and 17 as is the need to establish a better understanding of some of the fundamental processes occurring.

Figures 18-20 give examples of comparisons between the simulation and experiment for dewatering and washing performance, as well as data for complete cycles. Although such comparisons are currently limited, the data clearly demonstrate the underlying potential of the approach taken. In washing simulations the dispersion theory was generally observed to over-predict washing efficiency, though in several cases theoretical predictions of washing performance were good (Figure 18). It is thought that some correction of dispersion number to account for scale is required to make the predictions more accurate, such as that already available for rotary vacuum filters<sup>20,44</sup>. On Figure 19 both simulated and experimental data indicate that as dewatering pressure is incremented over a number of tests, so equilibrium cake moisture falls. In general predictions of cake moistures were good and this is in general agreement with previous reports of the accuracy of Wakeman's model<sup>45</sup>. Figure 20 gives an example of experimental data and theoretical predictions for volume of liquid removed from the filter vs. time for calcite suspensions

<sup>†</sup> 'Modern' filtration theory employs averages in the calculation of solids concentration profiles via the characterising empirical constants calculated from sequences of constant pressure tests.

at constant cycle pressures. The comparison is reasonable, though other similar simulations for more compressible cakes gave poorer fits, indicating that more work is required to produce accurate simulations here.

## CONCLUSIONS

The work presented in this paper highlights the potential benefits of the mechatronic approach to solid/liquid separation. The data show that it is now technically feasible to provide for accurate experimentation through the integration of mechanical design, electronics and computer control with chemical engineering processes such as filtration, dewatering & washing and accurate modelling when reliable theories and scale-up data are available. However, the apparatus described is capable of providing much more than that presented here. The apparatus allows the display of solids/liquid concentration information in real-time with up to eight distinct vertical planes for any combination of filtration, dewatering and washing, in addition to a 'live' display of liquor flow rates and other measurable parameters. Moreover, the potential exists to provide for full tomographic scanning using any of the recognised techniques and thus a method for the adaptive control of an experiment to account for changes in the processing conditions. By automatically controlling the apparatus it is also technically feasible to perform experiments at conditions of constant flow rate and variable pressure/variable flow rate via flow monitoring and the software manipulation of the applied pressure. This has the benefits of mimicking the operation of any positive displacement or centrifugal pump used to initiate a separation at an industrial scale without changing the properties of the feed in an inappropriate, and unquantifiable, manner. The ability to generate such data can only help the mathematical modelling of the operations involved in the filter cycle.

The term 'chematronics' might be applied to best describe the approach taken in the current work. The now widespread availability of microprocessor based equipment such as computers, controllers, transducers and sequencers opens many new avenues of research in solid/liquid separation. The ability to monitor and control a process and display information in real time provides a great incentive to researchers to devise new and novel methods of experimentation and thus progress our understanding.

## ACKNOWLEDGEMENT

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## NOMENCLATURE

$A$	filtration area ( $\text{m}^2$ )
$b_1$ - $b_3$	empirical constants (-)
$c$	solute concentration ( $\text{kg m}^{-3}$ )
$c_c$	effective feed concentration ( $\text{kg m}^{-3}$ )
$c_0$	initial solute concentration ( $\text{kg m}^{-3}$ )
$e$	voids ratio (-)
$e_0$	voids ratio at unit applied pressure (-)
$h$	filter cake height (m)
$M_c$	mass of solids in cake (kg)
$M_s$	solids mass fraction in feed (kg/kg)
$n$	compressibility index (-)
$\Delta p$	pressure difference (Pa)
$R_m$	filter medium resistance ( $\text{m}^{-1}$ )

$r^2$	coefficient of determination (-)
$S$	filter cake saturation (-)
$S_\infty$	irreducible saturation (-)
$t$	time (s)
$V$	cumulative volume (m <sup>3</sup> )
$W$	wash ratio (-)

#### Greek symbols

$\alpha$	specific cake resistance (m kg <sup>-1</sup> )
$\alpha_0$	specific cake resistance at unit applied pressure (m kg <sup>-1</sup> )
$\theta$	dimensionless time (-)
$\mu$	filtrate dynamic viscosity (Pa s)
$\rho$	filtrate density (kg m <sup>-3</sup> )
$\rho_s$	solids density (kg m <sup>-3</sup> )

#### Subscripts

$c$	cake
$d$	dewatering phase
$f$	filtration phase
$w$	washing phase

#### Superscripts

$av$	average value
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## FIGURES AND TABLES

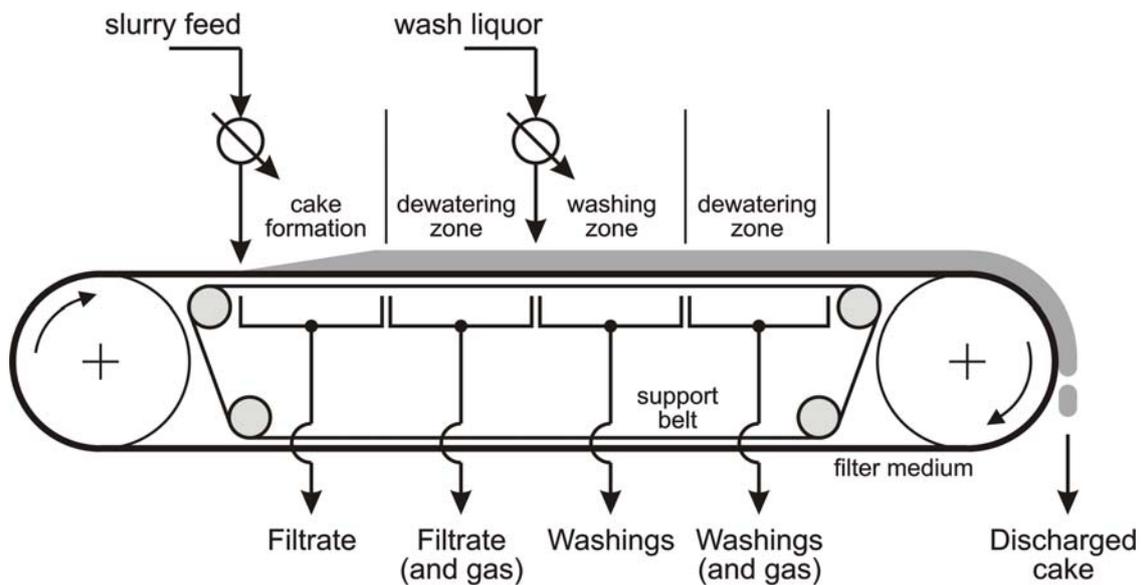


Figure 1: Typical cycle of operations on a horizontal belt filter.

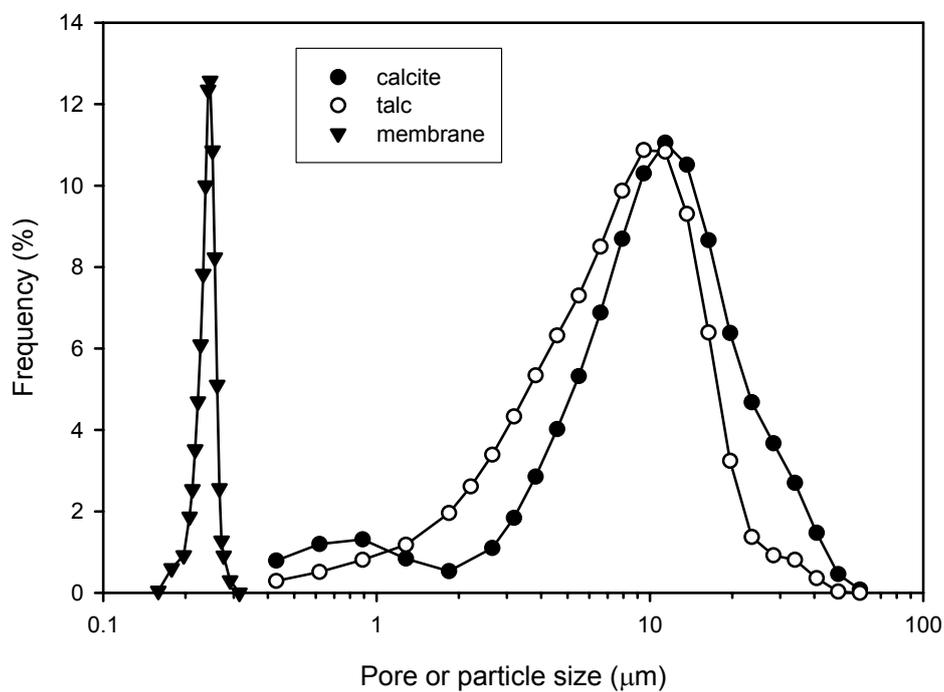


Figure 2: Particle and pore size distributions for calcite & talc suspensions and Gelman Versapor membranes respectively.

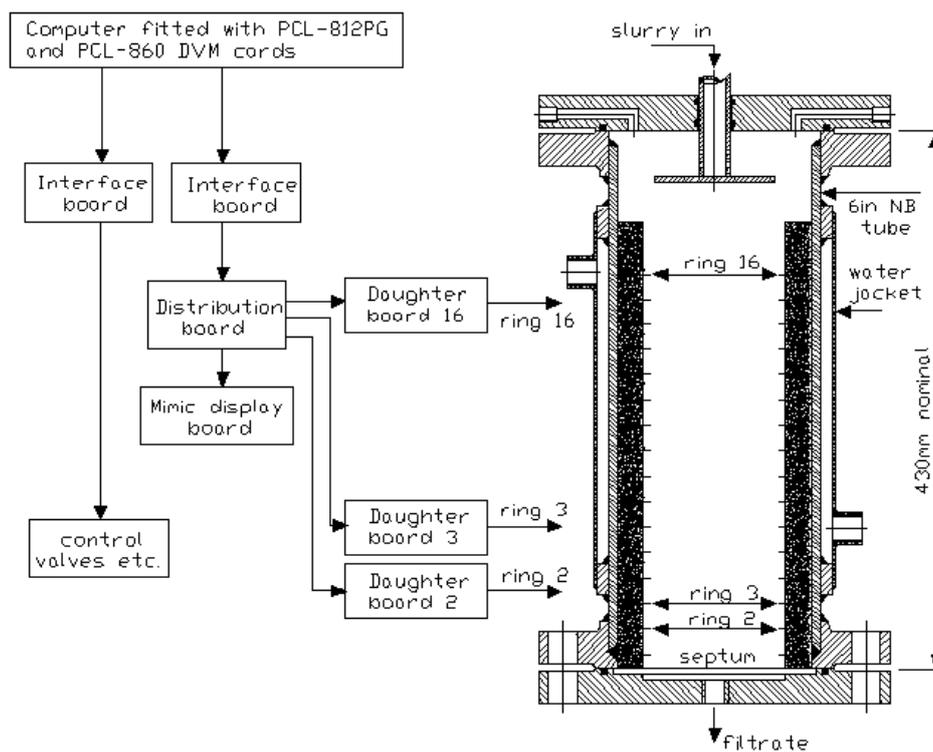


Figure 3: A schematic representation of the pressure filter cell and electrical resistance measurement circuitry.

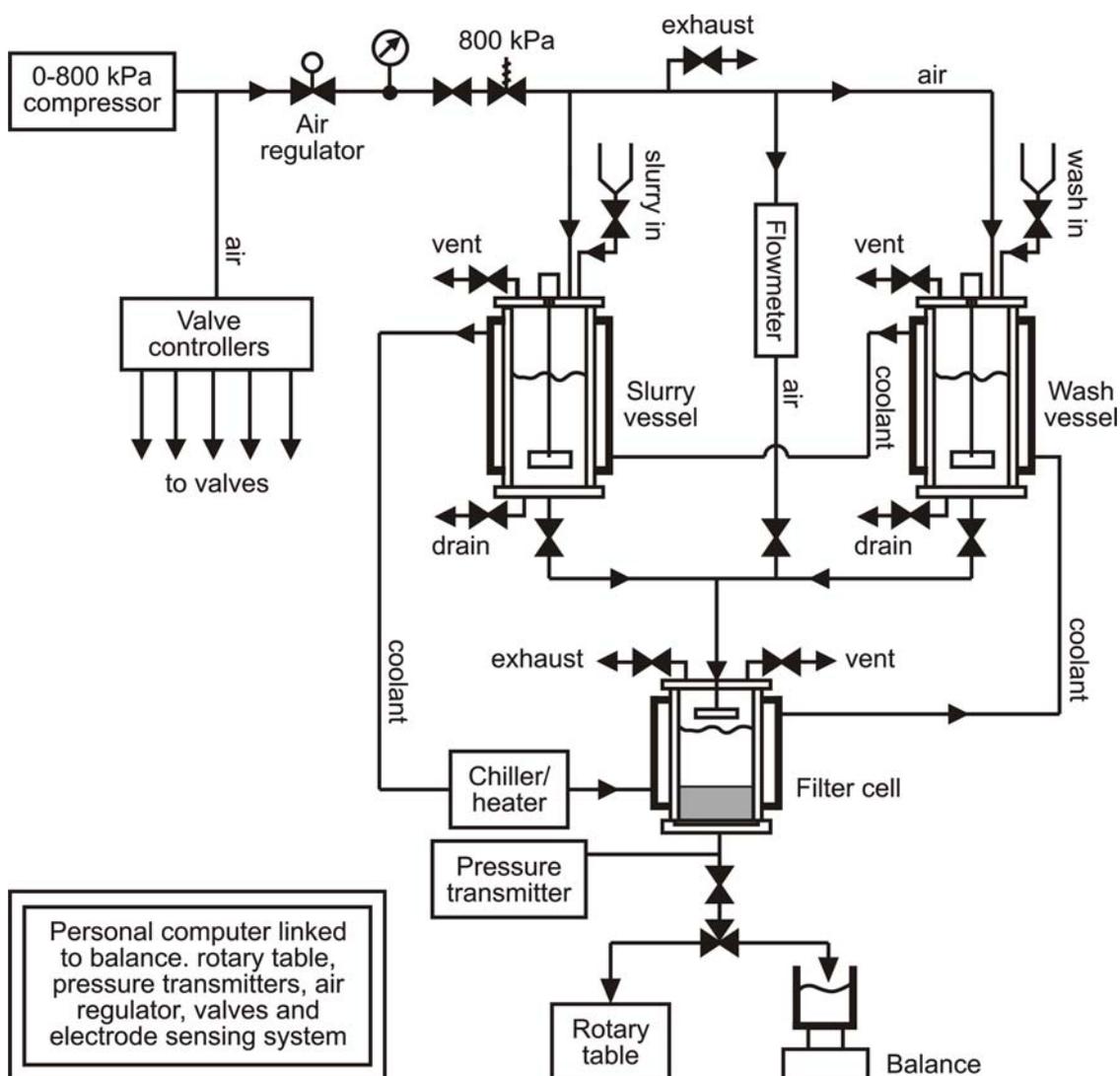


Figure 4: Schematic representation of the apparatus used in filter cycle experiments.

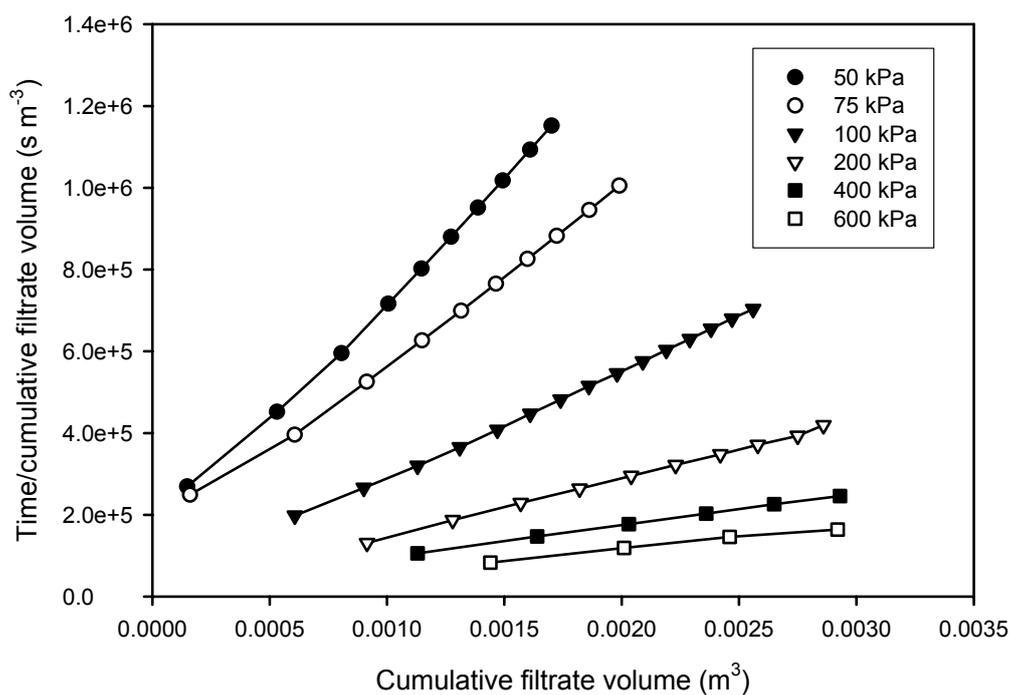


Figure 5: Reciprocal flow rate vs. cumulative volume for the constant pressure filtration of 10% v/v calcite suspensions.

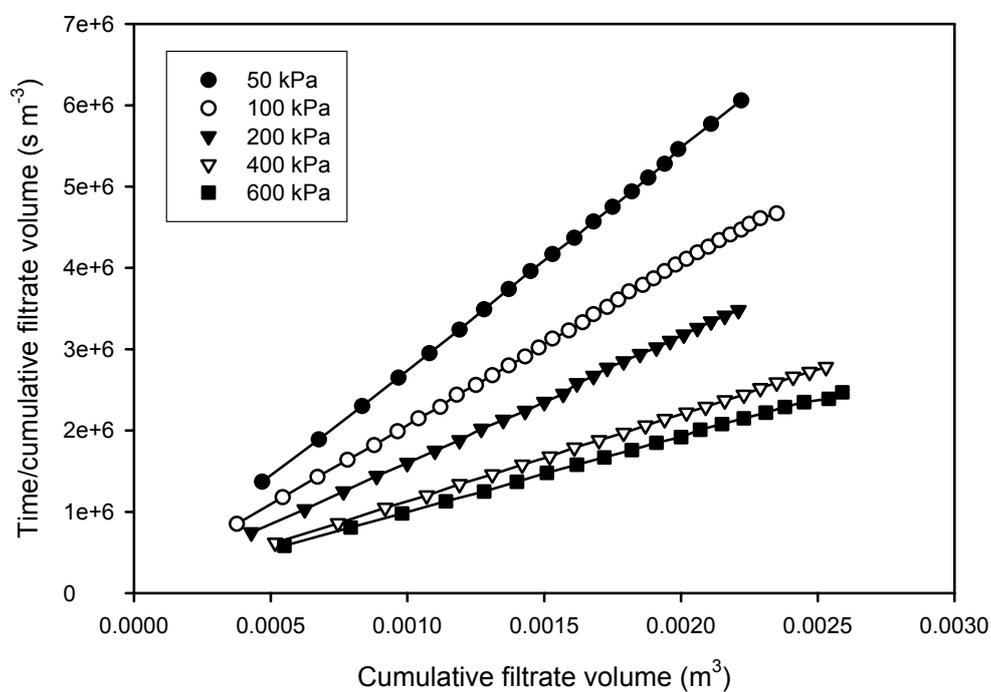


Figure 6: Reciprocal flow rate vs. cumulative volume for the constant pressure filtration of 10% v/v talc suspensions.

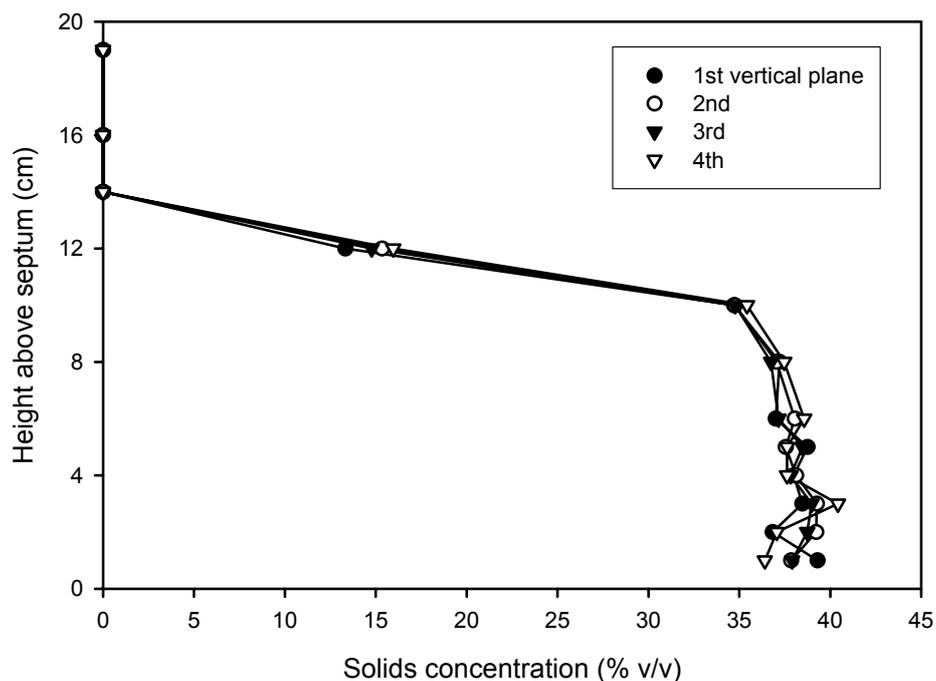


Figure 7: Solids concentration profiles in the filter cell towards the end of filtration for a 10% v/v calcite suspension.

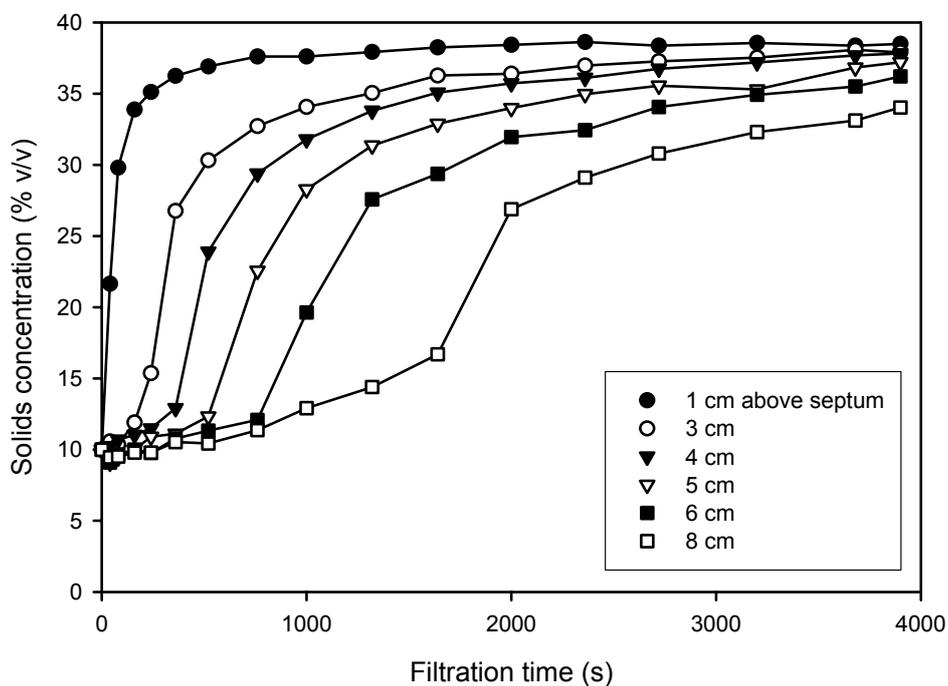


Figure 8: Solids concentration history up to 7 cm from filter medium for the filtration of a 10% v/v talc suspension.

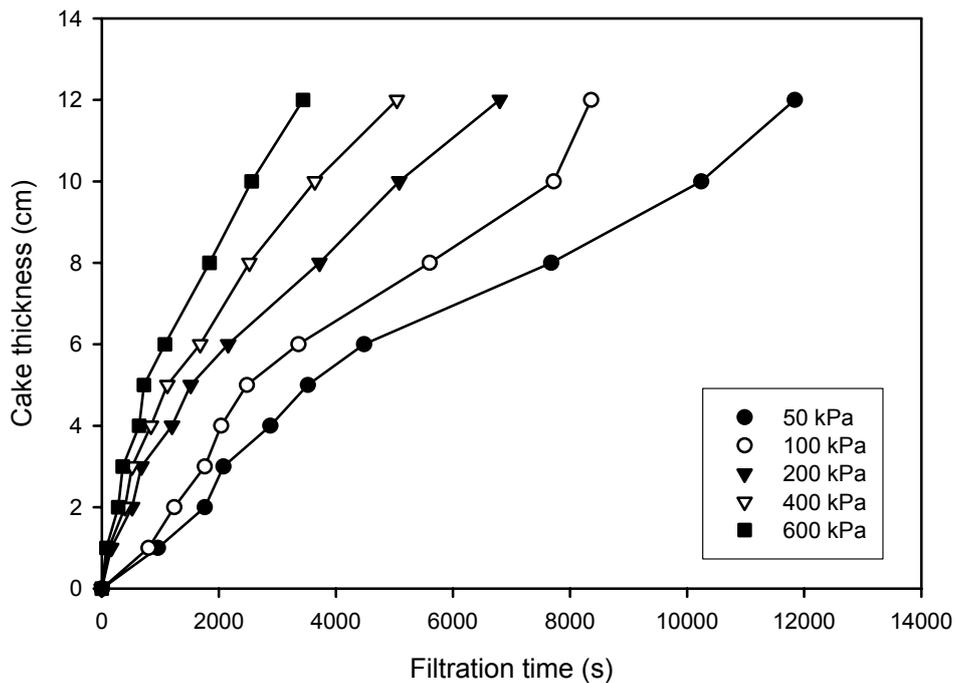


Figure 9: Cake formation history for the filtration of 10% v/v talc suspensions.

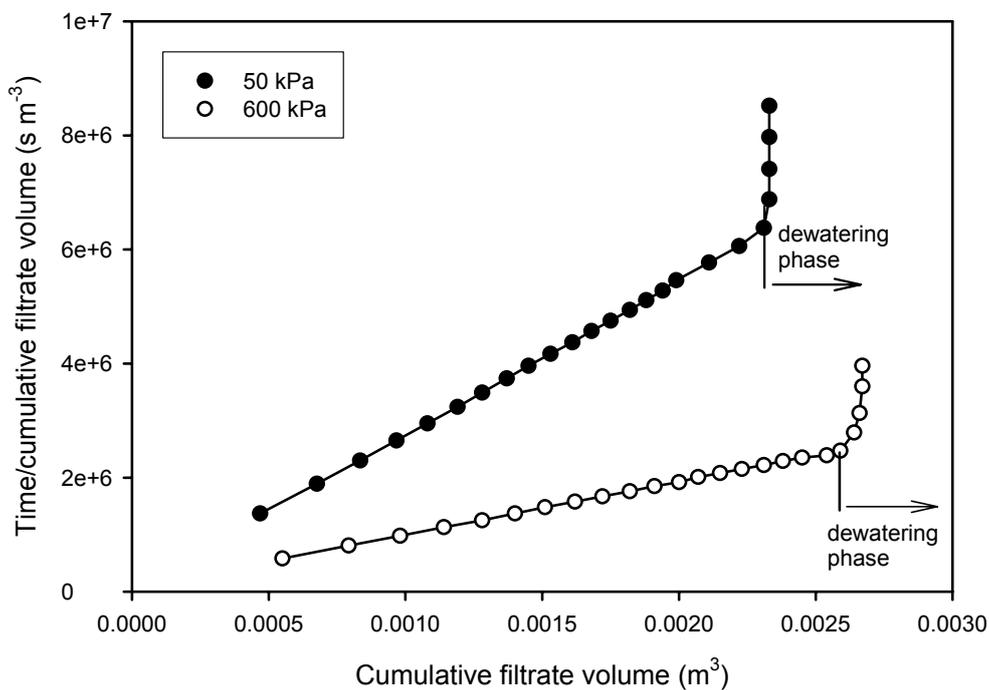


Figure 10: Reciprocal flow rate vs. cumulative volume for the filtration and gas dewatering of talc suspensions and cakes.

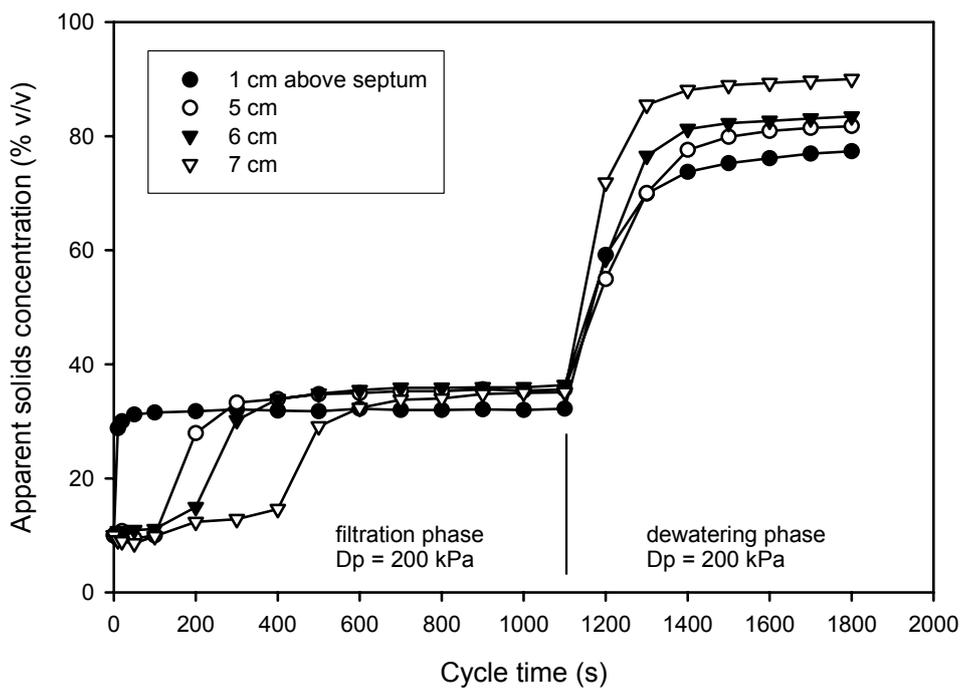


Figure 11: Apparent solids concentration profiles for the constant pressure filtration and gas dewatering of a 10% v/v calcite suspension.

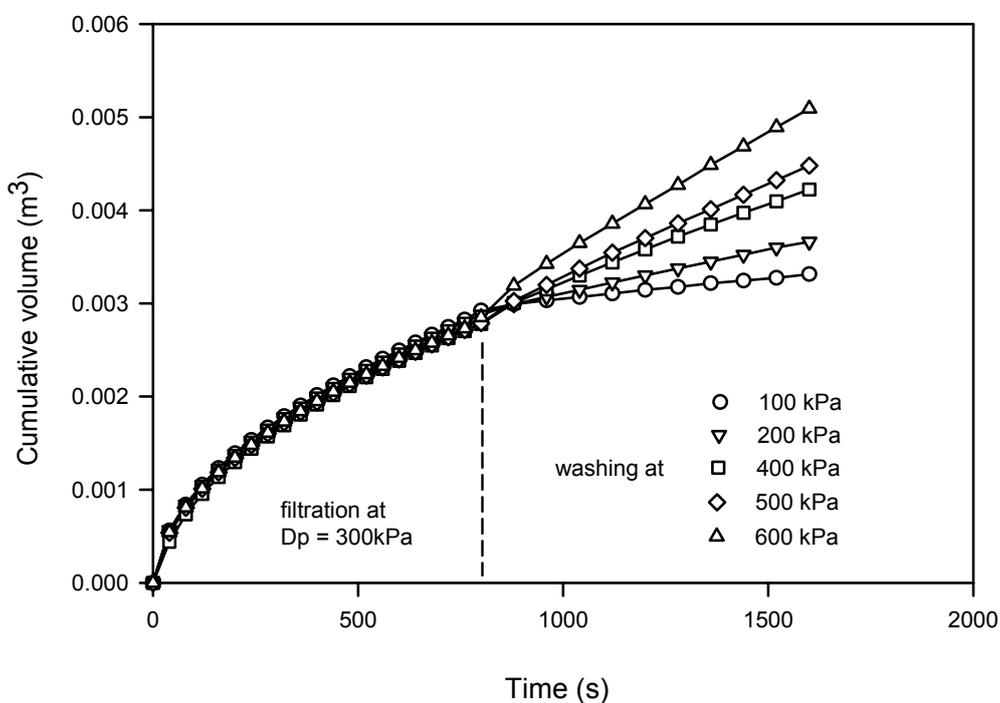


Figure 12: Cumulative volume vs. time for the filtration of 10% v/v calcite suspensions with subsequent displacement washing.

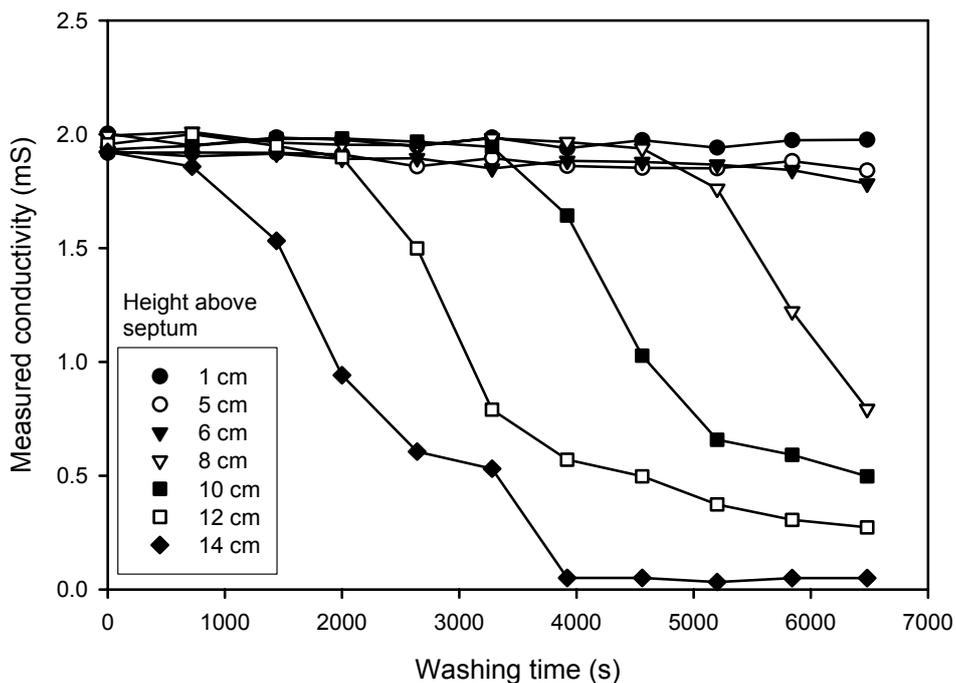


Figure 13: Representation of the movement of a washing front through a talc filter cake.

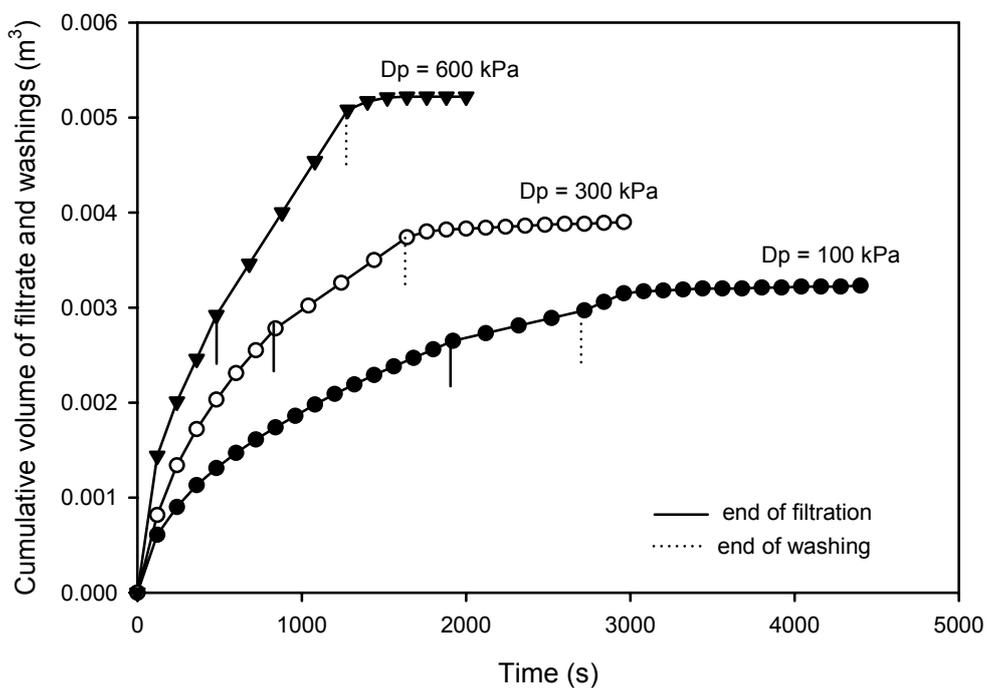


Figure 14: Filter cycle data for 10% v/v calcite suspensions at various pressures.

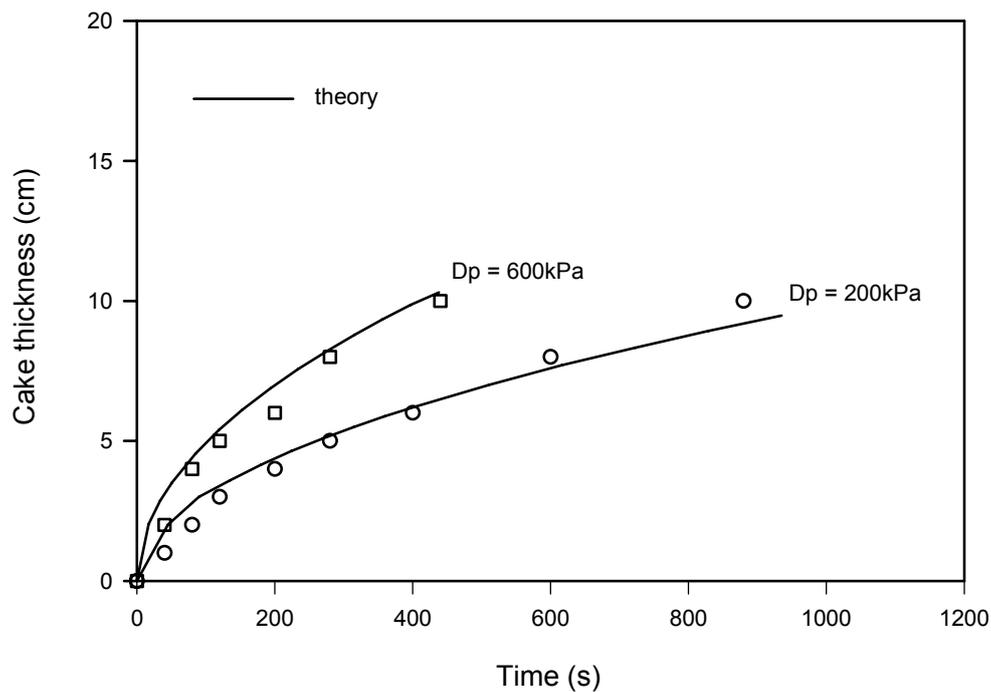


Figure 15: Cake height predictions for the constant pressure filtration of calcite suspensions.

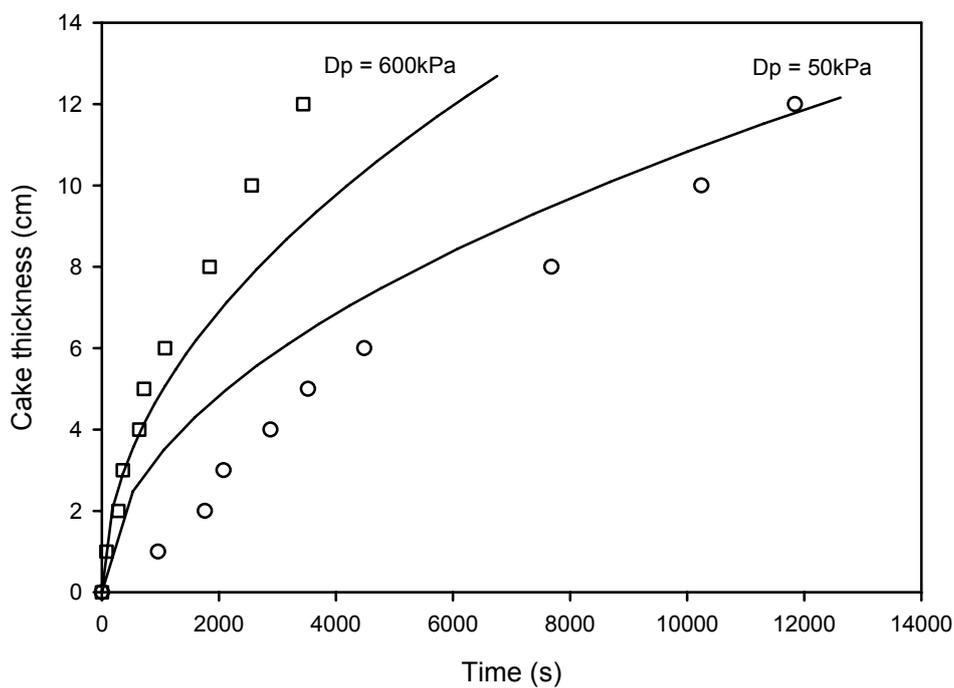


Figure 16: Cake height predictions for the constant pressure filtration of talc suspensions.

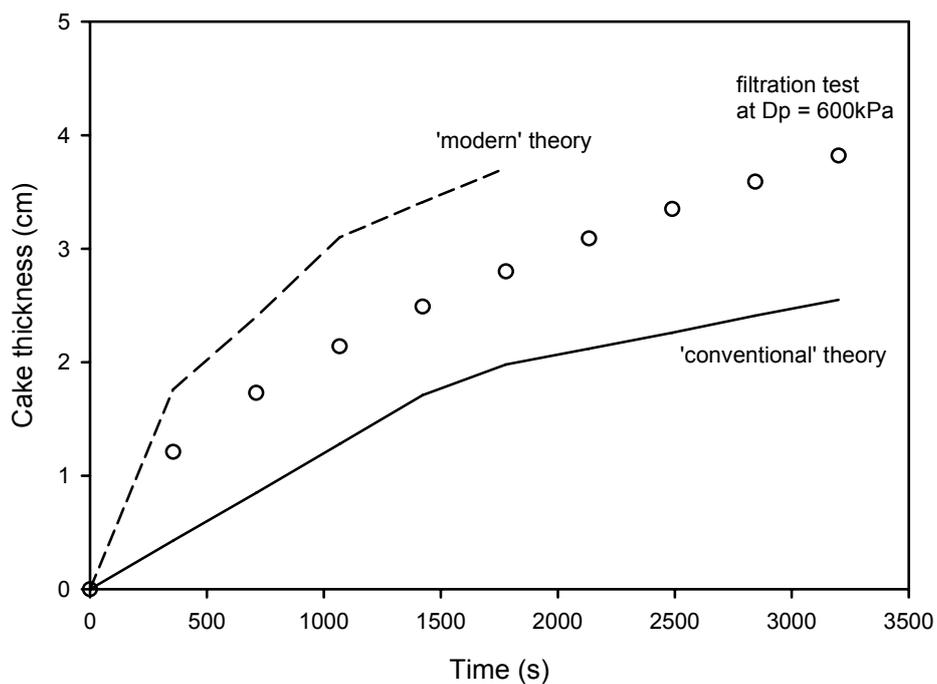


Figure 17: Comparison of 'conventional' and 'modern' filtration theories for the prediction of zinc sulphide cake heights.

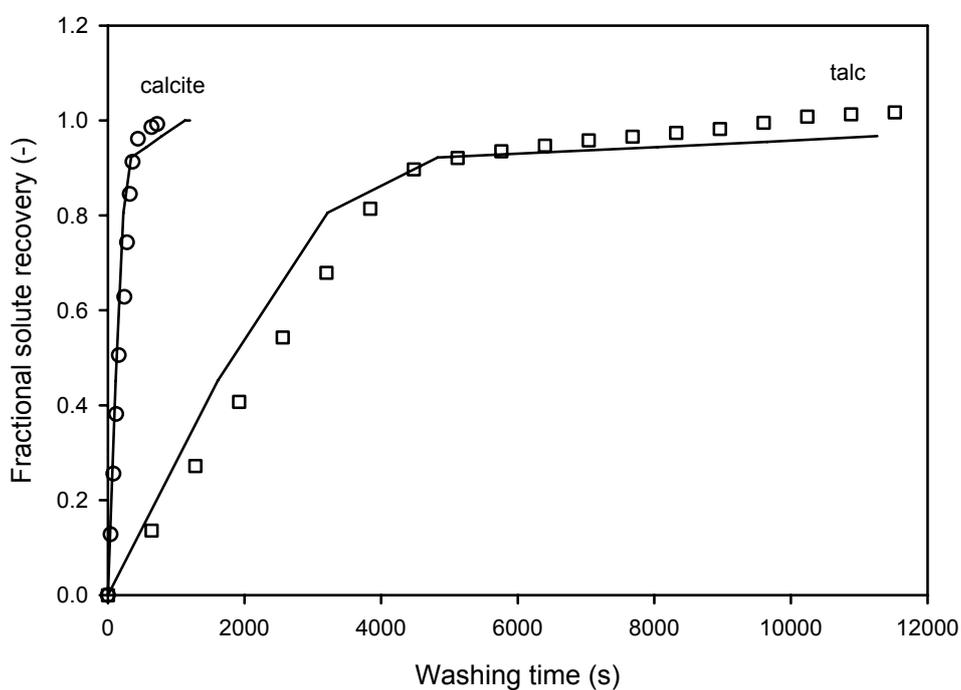


Figure 18: Prediction of washing performance at 400 kPa for talc and calcite cakes.

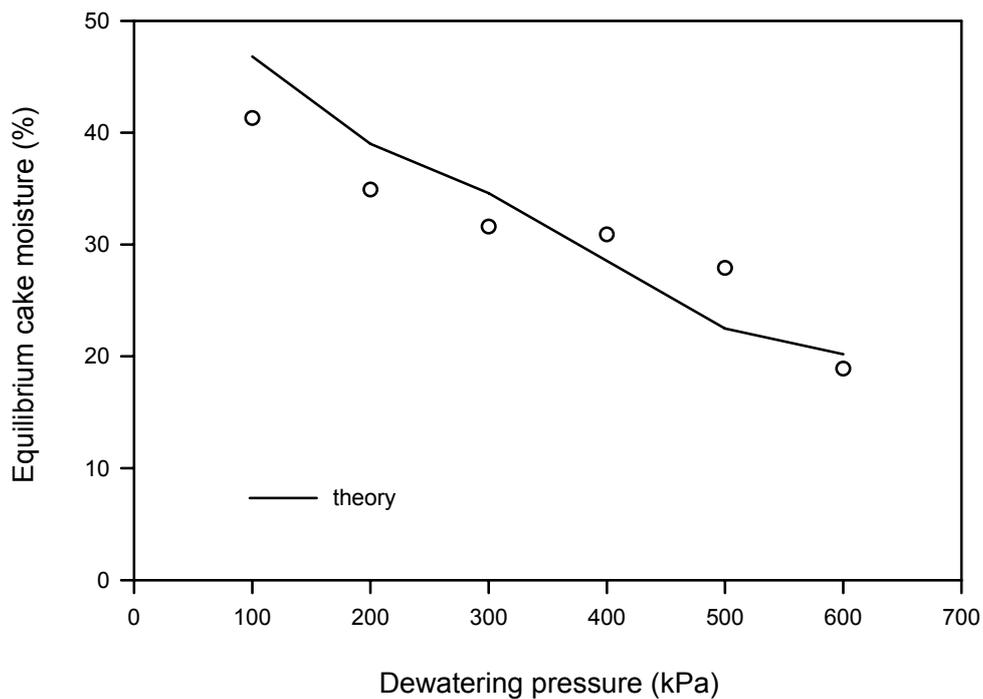


Figure 19: Average moisture contents of calcite cakes for increasing dewatering pressure.

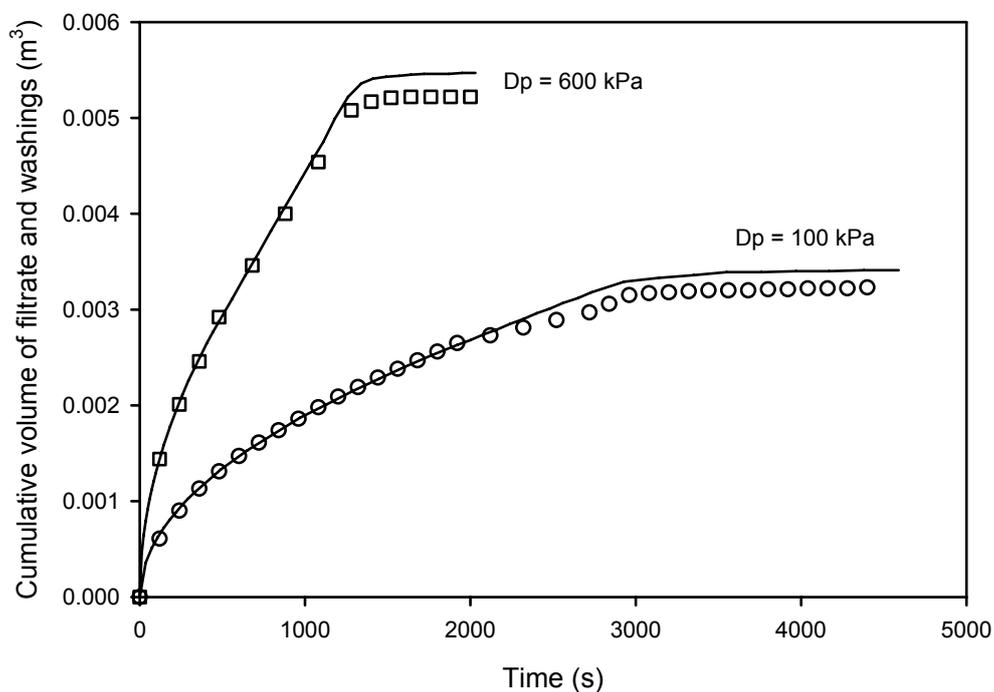


Figure 20: Comparisons of theory and experiment for filter cycles involving filtration, washing and dewatering of 10% v/v calcite suspensions.

Suspension	$\alpha_0$ (m kg <sup>-1</sup> )	$n$ (-)	$e_0$ (-)	$b_1$ (-)
5% v/v calcite <sup>†</sup>	4.20x10 <sup>9</sup>	0.084	1.775	0.077
10% v/v calcite <sup>†</sup>	2.55x10 <sup>9</sup>	0.132	2.101	0.127
10% v/v calcite <sup>††</sup>	1.17x10 <sup>9</sup>	0.198	3.415	0.322
20% v/v calcite <sup>†</sup>	1.79x10 <sup>9</sup>	0.153	2.304	0.209
20% v/v calcite <sup>††</sup>	2.66x10 <sup>9</sup>	0.104	2.873	0.231
10% v/v talc <sup>††</sup>	1.82x10 <sup>8</sup>	0.587	3.701	0.354
10% v/v zinc sulphide <sup>†</sup>	6.14x10 <sup>9</sup>	0.417	1.790	0.07

<sup>†</sup> determined on filter cell with 32 electrode pairs (Apparatus 2)

<sup>††</sup> determined on filter cell with 256 electrodes (Apparatus 1)

Table 1: Empirical scale-up constants for calcite, talc and zinc sulphide suspensions (pressures in (Pa)).