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## AN EXPERIMENTAL STUDY OF ABRUPT CHANGES IN CAKE STRUCTURE DURING DEAD-END PRESSURE FILTRATION

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#### **ABSTRACT**

In a practical study, a computer automated apparatus has been used to obtain experimental data for the dead-end, constant pressure filtration of aqueous zinc sulphide suspensions. The apparatus and particulate/suspension properties are briefly described and filtration data typical of that acquired during the investigation are presented. The conditions under which unexpected changes in cake structure occur are identified. It is shown how filtration parameters such as pressure, filter cell diameter and particle dispersion all influence the onset of both irreversible and reversible changes in cake structure and how these changes induce disturbances in the expected filtrate flow. Analyses of the experimental data and their relation to previous studies suggest that more localised changes in cake structure are responsible for the effects observed. The most probable mechanism is the migration of particle fines within a forming cake leading to the establishment of preferential flow channels; alternative mechanisms are also presented and discussed. It is concluded that an abrupt change in cake form is more likely during the filtration of suspensions containing loosely networked particles and when filter cell dimensions are larger.

#### **KEYWORDS**

Filtration; Tomography; Structured solids; Networked particles; Cake collapse; Compressibility

## INTRODUCTION

In 1953, Rietema¹ published a research paper presenting data for the constant pressure dead-end filtration of near spherical polyvinyl chloride particles from aqueous suspension. A primary finding was the observation of so-called 'retarded packing compressibility' (RPC), where parts of a forming filter cake appeared to abruptly change structure and collapse when a critical height was reached. Although several theories were postulated by Rietema, this intuitively strange and previously unreported phenomena was ultimately attributed to the initial flow stabilisation of the cake layers formed closest to the filter medium, and thought to be related to the electrokinetic properties of the challenge particles. The relatively high velocity of liquids passing through the cake interstices toward the start of filtration was considered sufficient to generate artificial support to these layers and as the filtrate flow lessened due to cake formation the flow induced support was reduced until a collapse occurred. Subsequent workers (e.g. Shirato *et al.* <sup>2,3</sup> and Murase *et al.* <sup>4</sup>) dismissed Rietema's results as they did not (and still do not) fit with existing and developing theories of cake filtration and attributed RPC to the presence of the intrusive pin electrodes that Rietema had used to monitor cake formation.

The research literature pertaining to RPC and the seemingly related phenomena involving abrupt changes in filter cake structure part way through a filtration, is limited and confined to the report of a few data sets<sup>5-11</sup>. Whilst some researchers have not related their findings to those of Rietema or other workers, their data suggest that abrupt changes in structure can occur at both the laboratory and process scale and in either conventional filters or those fitted with measurement probes. The current work presents a practical investigation of filtration where abrupt changes in cake structure were observed and attempts to relate the occurrence to operational parameters including filtration pressure, scale of operation, nature of challenge particulates and filter cake structure.

#### **EXPERIMENTAL APPARATUS AND CHARACTERISATION**

The computer automated apparatus employed in the investigation has previously been described in detail 12 and is related to some of the previous work by one of the authors that details aspects of experimental procedure and repeatability 13-17. The temperature regulated pressure filter unit comprised a stainless steel storage vessel used to contain the stirred feed suspension and connected, but interchangeable, Nutsche type filter cells having separation areas of either 5, 23, 170 or 490 cm². Each cell was of identical height and incorporated a similar suspension distributor plate in addition to thirty pairs of diametrically opposed, 1 mm diameter, wire electrodes positioned at 5 mm vertical spacings above the filter medium; the electrodes measured values in a single vertical plane within each filter cell. The electrodes protruded ~2 mm into a cell and were used to obtain transient solids concentration profiles within a downward filtering suspension or cake by an electrical resistance method. Constant filtration pressures were applied via an interfaced electronic regulator over the range 0-600 kPa and the filtrate flow was semi-continuously monitored with an interfaced electronic balance.

Zinc sulphide, grade L manufactured by Sachtleben Chemie, was the chosen solid for the filtration experiments and suspensions were prepared by dispersing the powder to the required concentration in distilled water. As a necessary aid to dispersion, Dispex N40 (Allied Colloids Ltd.) was added at a dosing of 0.1% based on the dry mass of powder. Suspension pH was adjusted by the addition of analar grade glacial acetic acid or sodium hydroxide and the resultant mixture was stirred gently to ensure homogeneity. In this manner a range of particle and filtration properties could be achieved (see Table 1). The influence of particle surface charge was interpreted through the measured relationship between  $\zeta$ -potential and suspension pH. Scanning electron micrographs (SEMs) of powder samples showed a near spherical, oblate ellipsoid shape for discrete zinc sulphide particles. A hydrophilic, microporous, Gelman Sciences Versapor membrane with a 0.2  $\mu$ m rating, a nominal thickness of 185  $\mu$ m and a measured hydraulic permeability of 7.0x10<sup>-15</sup> m² was used in all filtration experiments.

#### **EXPERIMENTAL RESULTS**

The experimental data reported in this paper were obtained for constant pressures of 100-600 kPa and are representative of the dataset obtained during the investigation. Due to experimental constraints no tests were performed in filter cells without sensing electrodes, however, previous work by one of the authors has shown their inclusion to have a negligible effect on cake formation provided their diameter is less than 2 mm and their protrusion from a cell wall is less than 30 mm<sup>12</sup> (see also the discussion section of this paper). All experiments were performed at a constant temperature of 25±1°C and in all cases a visually clear filtrate was obtained throughout.

Figures 1-3 show data that illustrate the expected behaviour for zinc sulphide filtrations over a number of experimental conditions. When the particles were more dispersed in the feed suspension, for example at higher values of pH, a near incompressible cake tended to form. Figure 1 illustrates this whereby transient electrode readings of solids concentration in the cake were similar throughout its changing height and essentially constant over the duration of a filtration. There was no evidence of RPC or related phenomena during experiments performed with suspensions containing these more discrete particles. For zinc sulphide suspensions at lower pHs, where the measured particle sizes were larger and the suspended particles exhibited a degree of (relatively fragile) structure, Figure 2 shows that the cakes that were formed had a greater degree of compressibility. Here, the measured solids concentrations varied throughout the cake height and tended to increase both toward the filter medium and with time as filtration progressed. It is noted in passing that other constant pressure data obtained with the described apparatus and, for instance, aqueous calcite, talc and titania suspensions covering a wide range of cake

compressibilities and process conditions showed no abrupt changes in cake structure during filtration 12,13,15,16.

Whilst not evident in all experiments (e.g. Figure 3), a significant number of tests with zinc sulphide suspensions at lower pHs showed evidence of phenomena related to abrupt changes in filter cake structure. Figures 4 and 5 illustrate that after periods of otherwise normal cake formation, a marked change in the expected progression of cake solids concentration was observed and accompanied by a noticeable, and sometimes substantial, increase in the measured filtrate flow rate. Although (for clarity) data are only shown for cake concentrations at single heights in each filter cell, when a sudden change in cake structure occurred many readings from the electrode pairs in the cake would register a deviation from the expected values: readings from electrodes in the suspension above the cake remained almost constant during this time. These results indicate that when changes happen, they can be manifested over a significant portion of cake rather than in a remote region. In some cases, such as the experiment represented by Figure 5, following a measured change in cake structure a period of time elapsed at the raised filtration rate before cake formation appeared to continue as previously expected; this is indicated by the similar gradients of t/V vs. V before and after the change in structure. Figure 6 illustrates the solids concentration profiles measured just before and just after the onset of the change in cake structure apparent in Figure 4. Although the macroscopic changes measured by the electrode pairs were relatively minor, they were often sufficient to register substantially increased filtration rates. A reduced measure of solids concentration equates to the presence of more liquid and perhaps preferential flow channel(s) and has implications for the overall system mass balance. It is noted that transient in-situ measurements of solids concentration and visual observations at the end of experiments suggested the continual presence of suspension close to the initial feed concentration above a forming cake (see also Figures 1 and 2). None of the experiments performed showed evidence of a sudden, RPC type, cake collapse leading directly to a reduced filtrate flow.

The degree to which the abrupt changes in cake structure/filtration performance were observed was dependent on several factors and in some cases the chosen experimental conditions.

## **Effects of Filtration Pressure**

At lower pH where the measured size of feed particles tended to be larger, and in particular with filter cells of area in excess of 5 cm<sup>2</sup>, the pressure applied during a filtration was generally seen to have an influence. Figure 7 gives an example where the cake thickness at the transition in structure, ( $h_{tr}$ ) was estimated from electrode measurements by assuming cake to be present at solids concentrations above 30% v/v and calculated theoretically by mass balance from:

$$h_{tr} = \frac{\left(V_f\right)_{tr} \left(1 + \left(e_{av}\right)_{tr}\right)}{A\left\{\frac{\rho_s}{\rho} \left(\frac{1}{s} - 1\right) - \left(e_{av}\right)_{tr}\right\}}$$
(1)

where  $(V_f)_{tr}$  is the cumulative volume of filtrate at the transition, s is the solids mass fraction in the feed, A is the filter area,  $\rho$  and  $\rho_s$  are the filtrate and solids densities respectively and  $(e_{av})_{tr}$  is the average voids ratio of the cake just prior to the transition (i.e. a measure of the macroscopic cake structure). For the given experimental conditions it is evident that theoretical predictions of cake thickness significantly exceed the experimentally measured values and that above a threshold pressure (~300 kPa for the chosen data) there was a reduced influence of the filtration pressure on the critical cake thickness. For the lower feed concentration of 5% v/v, but otherwise identical experimental conditions, the cake structure transition occurred at reduced cake thickness.

## Effects of Suspension pH

In general terms, when the pH of a suspension was reduced there was a tendency for the filtration rate to be higher. This could be expected from comparisons of the characteristic particle sizes in the feed (as shown in Table 1) and similar data have been previously reported by the authors<sup>12</sup>. When particles were more discrete, such as at pH = 10.5, none of the experiments showed evidence of abrupt transitions in cake structure; Figure 8 illustrates this via the linearity of the respective t/V vs. V plot. Figure 8 also shows, however, that when the suspension pH was lower and the larger feed particles exhibited a degree of structure, there was an increased tendency for relatively sudden changes in a forming cake to occur. In the example (at pH = 2.5) there was an alteration in cake structure accompanied by an abrupt change in the gradient of the t/V vs. V plot. For such cases, there was also a more pronounced upward curvature of the dataset prior to the abrupt change that may indicate continuing cake compression and/or a degree of particle settling.

## **Effects of Filter Area**

Whilst changes in the degree of RPC type effects with filter area were noted, it was difficult to establish definitive trends in all cases. The clearest indicator was that no abrupt changes in cake structure were observed for experiments with the smallest filter cell. Although alterations to suspension pH, filtration pressure and feed concentration each had an influence on the measured filtration rates, only progressive changes in cake thickness and solids concentration were recorded throughout the duration of the experiments with the 5 cm² cell. Cake formations proceeded in the intuitively expected manner such as that displayed in Figures 1 to 3 and these data suggest that when there is relatively close proximity of the cell walls to the centre of the filter cell the inherent properties of the forming cake can be sufficient to prevent any abrupt changes. Other data obtained with the larger filter cells, however, showed some progressive deviations in filtration performance. Figure 9 gives an example where, for the particular set of experimental conditions, the measured cake thickness at an abrupt transition in cake structure was reduced for a larger filtration area. Such a result is perhaps intuitive as the cell walls in filters of larger diameter will offer less support to the forming cake. In other data sequences obtained over different process conditions there was suggestion of similar behaviour, but the observed trends were less defined.

### **Effects of Suspension Concentration**

There was some, but limited, evidence to suggest that the concentration of solids in the feed suspension influenced the timing and onset of abrupt changes in filtration behaviour. Figures 10 and 11 indicate this whereby apparent changes in cake structure seemed to occur at lower filtrate volumes and hence after reduced time intervals, with a reduced feed suspension concentration.

## **DISCUSSION**

The experimental data in Figures 1-11 demonstrate the extent to which relatively abrupt changes in cake structure can influence constant pressure filtration and the operational parameters that affect the overall process. In all cases, where a change in cake structure was observed an initial reduction in cake solids concentration was measured and an accompanying, sometimes temporary, increase in filtrate flow was recorded. Similar behaviour has been previously noted by Fathi-Najafi and Theliander<sup>9,10</sup> and discussed in more detail by Sørensen *et al.*<sup>11</sup>, seemingly in the context of anomalous results. These authors contradict the earlier work of Rietema<sup>1</sup> and Baird and Perry<sup>6</sup> who exclusively noted cakes that collapsed during filtration with the consequence of reduced filtration rates and increased cake resistances. Whilst Table 2 indicates some of the previously proposed reasons for abrupt changes in cake structure, it also serves to highlight the complexity of the processes and the almost exclusive reliance on qualitative rather than quantitative assessment. In the opinion of the authors, it is reasonable to infer that both the data reported in this paper and the data from other researchers are either different, but perhaps related, phenomena or different facets of the same phenomenon.

The example data presented in Figures 4 and 5 indicate that substantial increases in filtrate flow can be registered when relatively abrupt changes in cake structure occur. Although an accurate quantification of the changes is difficult, preliminary calculations can provide some insight. The average cake permeability ( $k_{av}$ ), which is directly proportional to the liquid flow rate according to Darcy's Law provided the cake thickness remains constant, can be calculated using respectively either the Kozeny or Happel models:

$$k_{\rm av} = \frac{X^2 \varepsilon_{\rm av}^3}{180 \left(1 - \varepsilon_{\rm av}\right)^2} \tag{2}$$

$$k_{av} = \frac{x^2}{36} \frac{6 - 9(1 - \varepsilon_{av})^{0.33} + 9(1 - \varepsilon_{av})^{1.67} - 6(1 - \varepsilon_{av})^2}{(1 - \varepsilon_{av})(3 + 2(1 - \varepsilon_{av})^{1.67})}$$
(3)

where x is a representative particle size and  $\varepsilon_{av}$  is the average porosity of a cake. Applying equations (2) and (3) to the data in Figure 4 indicates that  $\varepsilon_{av}$  needs to change over the ranges 0.57 to 0.69 or 0.57 to 0.71 to account for the measured increase in liquid flow after the change in cake structure. Corresponding calculations for the data shown in Figure 5 indicate predicted changes in average porosity of 0.74 to 0.79 and 0.74 to 0.81 respectively. Although the Happel model predicts marginally greater changes in both example datasets, all calculations showed that relatively large and probably unrealistic, macroscopic changes in average porosity are required to promote the observed flow increases. This suggests that more unobstructed and localised changes in cake structure may be responsible for the recorded deviations in flow. The inference is supported by experimental data such as Figure 6, where only a relatively small increase in the porosity measured over the cell diameter and through the cake thickness was recorded for a more than three fold increase in filtration rate. When interpreting such information it is important to note that the electrode probes do register some change in cake structure. However, as measurements were taken in a single vertical plane within a filter cell and inherently represent average values over the cell diameter, any significant but more localised changes in structure are effectively beyond the resolution of the measurement technique and may thus be only partially represented.

The abrupt changes in electrode readings also have implications for the system mass balance as it may be inferred that the amount of solids present in the system is suddenly reduced. In reality this is not the case as filtrates remained clear in all tests. However, the presence of preferential flow channel(s), perhaps caused by the local migration of particles, could readily account for changes in electrode readings. If portions of the flow channels are within the vicinity of the electrodes then a change in measured value may be observed without inferring an incorrect mass balance. It is noted that physical samples removed from the cakes at the end of all experiments agreed closely with electrode readings in terms of macroscopic solids concentration and confirmed overall mass balances errors, usually at the lower end of the range 0-3%.

For more localised changes in a cake to occur it seems likely that preferential, and essentially unobstructed, flow channels need to form within a growing filter cake and remain 'open' for a significant period of time. This infers that the remaining largely unaffected cake must be sufficiently strong to retain the original structure. Whilst 'channelling' is well reported within the sedimentation literature<sup>18</sup> such theories cannot be directly transposed to filtration due to the inherent fundamental differences of sediment and cake formation. Moreover, abrupt structural changes in a filter cake would need to occur in several different, but related, ways in order to account for the observed effects. Several hypotheses can be put forward:

 A relatively abrupt, seemingly irreversible change in cake structure leading to an increased filtration rate (compare with Figure 4). Here, part(s) of a cake become sufficiently eroded by the flowing filtrate to generate preferential flow channel(s). In order for this to happen the significantly more porous portion(s) of the cake must be surrounded by regions capable of withstanding further significant changes in structure and be of a sufficiently convoluted nature to eliminate significant ingress of new solids from the cake/filtering suspension. Such channels may tend to form nearer to the filter medium and may close again after a sufficiently long period of filtration due to the increased solids pressure.

- 2. A relatively abrupt, localised change in cake structure that facilitates a temporary period of increased filtration rate. After a time the channel(s) formed tend to close due to the ingress of freshly filtered solids or, perhaps, collapsed solids from the already formed cake (compare with Figure 5). For this mechanism to occur a degree of structure needs to exist in the cake surrounding a channel, however, there may be a tendency for such a channel to form further away from the filter medium where there is a greater probability that fresh solid material may enter after a period of time.
- 3. A relatively abrupt, but more macroscopic change in cake structure that leads to an irreversibly reduced filtration rate. Here, more significant portions of a forming cake collapse when a critical thickness is reached. These particles then form into a cake of reduced overall thickness, permeability and (inevitably) porosity. Such an alteration in cake structure would almost certainly be indicated by readily measurable macroscopic changes in cake properties and is a descriptor of the early data reported by Rietema<sup>1</sup> and Baird and Perry<sup>6</sup>.

Whilst the authors recognise the difficulty of quantifying exact mechanisms and the conjecture of the hypotheses, they do represent explanations of both the current and previously obtained experimental results. The hypotheses also represent different facets of what may be the same phenomenon, namely a relatively abrupt change in cake structure leading to a disruption in the expected rate of filtration and their occurrence is supported by other work.

It is well established that cake structure undergoes progressive variation with filtration time due to the compressibility induced by liquid flow through cake interstices 12,13,19-22. The degree of compressibility is influenced by factors including particle shape, surface properties and the state of dispersion. By examining micrographs, Banda and Forssberg<sup>23</sup> and Windhab and Friedmann<sup>28</sup> provided experimental evidence that particulates within a filter cake are not always in point contact with each other or, by inference, with the filter walls. In such instances there is scope for more significant cake and liquid movements, a fact that was demonstrated experimentally by Bakker et al. who applied mechanical vibrations directly to filter cakes and filter apparati. Whilst the sometimes severe and abrupt changes in cake structure observed by Bakker are indicative of the potential for cake collapse, it seems unlikely that sufficient mechanical vibrations will frequently occur in practice. Perhaps the strongest evidence to support the authors' hypotheses is the limited data available from Sørensen *et al.*<sup>11</sup> who examined the constant pressure filtration of waste water solids suspensions. Sørensen reported data of a very similar form to that shown in Figure 5, where a temporary increase in filtrate flow part way through a filtration test was, unlike the current authors' data, accompanied by a sudden increase in the turbidity of the filtrate. After a period of time the filtration rate and turbidity level in the filtrate returned once more to the original level. Sørensen concluded that when conditions allow, smaller sized particles in a filter cake can migrate locally due to the tortuous flow of the filtrate. The inference is that their movement creates regions of either increased flow resistance and/or promotes the formation of preferential flow channels from which solids are transported away. In the current work it is noted that although the presence of fine particle sizes was recorded during characterisation tests, the use of a relatively small pore size membrane prevented the passage of fine particulates into the filtrate. Some researchers have previously attempted to remove sections of formed filter cakes and determine, for instance, particle size distributions. These measurements are worthwhile for many cakes but difficult to perform reliably, and in the context of the current work unrealistic as form changes occurred more locally within cakes exhibiting a degree of (fragile) structure that would be lost when physical sampling is used.

It is interesting to note that all reports of abrupt changes in cake structure have occurred during the filtration of suspensions containing networked particles, examples being suspensions where either flocculation or coagulation has previously taken place or when strong electrokinetic interactions are present. The data in this paper show that the potential for cake collapse is significantly reduced when the particles in suspension are more discrete or less structured. The potential for abrupt structure changes also appears to be reduced when a filter cell of a smaller diameter is used and this suggests that the filter walls may tend to artificially support a cake when there is a relatively small distance between them. Wall effects have previously been reported to influence the filtration process in compression-permeability (C-P) cells<sup>24-26</sup>. Tiller<sup>24</sup> reported C-P data for solka-floc suspensions where the aspect ratio (i.e. height/diameter) of the formed cake was varied over the range 0.1-1.2. As the aspect ratio was increased more of the mechanical pressure applied to the cake was effectively dissipated by the cell walls, thereby reducing the transmitted pressure and filtration rate.

Whilst filter walls are likely to have a somewhat different effect in gas driven pressure filtration, some researchers<sup>2-4</sup> have previously dismissed abrupt changes in cake structure as an artefact of intrusive electrode measurement probes. It is suggested that the probes provide artificial support to a growing cake and help to prevent normal cake compression. Although the experimental data presented in this paper were obtained using nominally intrusive electrodes, it is the authors' opinion that both the state of particle dispersion and the relative dimensions of the feed particles to the filter cell diameter influence the onset of changes in structure to a much greater extent. Such a conclusion is strongly supported by previous work by one of the authors as well as those researchers who have observed changes in filter cake structure in the absence of intrusive probes<sup>5,8-11,17,27</sup>.

#### CONCLUSIONS

The experimental data presented in this paper highlight the degree to which abrupt changes in cake structure can take place during dead-end pressure filtration. In all the investigated cases, a measured reduction in the cake solids concentration part way through a filtration was accompanied by an increase in filtrate flow. In some cases the increased flow was temporary and seemingly reversible such that after a period a cake structure would return to the expected form and filtration rate would be reduced to the expected level. The onset of these abrupt changes was influenced by several filtration parameters.

For a given set of experimental conditions, a threshold filtration pressure appeared to exist above which the cake thickness at an abrupt change remained nearly constant. When the pH of a feed suspension was adjusted to give more discrete particles and/or the filter cell diameter was smaller, there was a reduced tendency for sudden changes in cake structure to occur. It is considered that abrupt changes are more likely to occur during the filtration of suspensions containing structured or more loosely bound particles. In the opinion of the authors, the changes result from more local variations in cake structure brought about by simultaneous particle fines migration and the subsequent formation of preferential flow channels.

### **NOMENCLATURE**

- A filter area (m<sup>2</sup>)
- e voids ratio (-)
- h cake height (m)
- k cake permeability (m<sup>2</sup>)
- s solids mass fraction in the feed (w/w)
- $V_f$  cumulative volume of filtrate (m<sup>3</sup>)
- x particle size (m)

- ε cake porosity (-)
- $\rho$  filtrate density (kg m<sup>-3</sup>)
- $\rho_{\rm s}$  solid density (kg m<sup>-3</sup>)
- av average value
- tr transition value

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## **FIGURES AND TABLES**

рН	50% particle	ζ-potential (mV)	Compressibility
	size (µm)		index <sup>*</sup>
2.5	12.44	-12	0.30
6.0**	15.64	-31	0.37
10.5	3.53	-40	0.02

<sup>\*\*</sup> natural pH of suspension \* typical values

Table 1: Characterisation information for zinc sulphide suspensions with 0.1% added Dispex N40. Particle size and  $\zeta$ -potential measurements were determined using a Malvern MasterSizer and ZetaSizer respectively.

Researcher	Description of the aqueous suspension(s)	Intrusive probes	Postulated cause(s) of changes in cake	Attributed cause
Rietema <sup>1</sup>	PVC spheres at various states of dispersion	Yes	<ol> <li>Migration of cake particle fines</li> <li>Slow compression</li> <li>Presence of gaseous bubbles</li> <li>Destabilisation of cake by flow</li> <li>Filter vibration</li> </ol>	Destabilisation of cake structure by reducing filtrate flow
Bakker <i>et al.</i> <sup>5</sup> ; Heertjes <sup>27</sup>	Polystyrene spheres with a dispersant	No	-	Vibration *
Baird and Perry <sup>6</sup>	Mixtures of calcium carbonate and diatomite (in city water)	Yes	None given	None given
Fathi-Najafi and Theliander <sup>9,10</sup>	Calcium carbonate; calcium silicate; lime mud ***	No	-	Formation of unobstructed flow channels within a cake
Sørensen <i>et al.</i> <sup>11</sup>	Anaerobically stored wastewater solids	No	<ol> <li>Sedimentation of the filtering suspension</li> <li>Formation of channels in a cake</li> <li>Influence of filter cell walls</li> <li>Migration of particle fines within a cake</li> </ol>	Migration of particle fines within a cake

<sup>\*</sup> physical vibrations applied to filter and/or cake; \*\* all described as being in a state of agglomeration in suspension

Table 2: A summary of previous research where abrupt changes in cake structure during dead-end filtration have been observed.

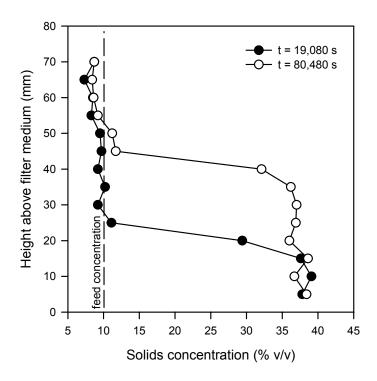


Figure 1: General nature of the solids concentration profiles measured in a filter cell during near incompressible cake formation (suspension concentration = 10% v/v; suspension pH = 10.5; filtration pressure = 100 kPa; filter area = 23 cm<sup>2</sup>).

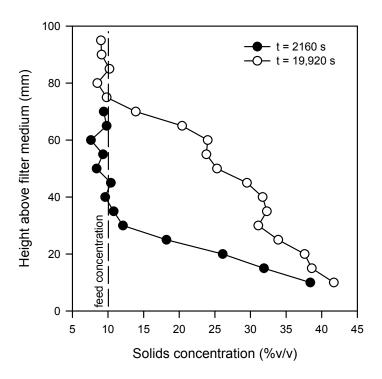


Figure 2: Examples of the solids concentration profiles measured during a filtration where a more compressible cake forms (suspension concentration = 10% v/v; suspension pH = 6.0; filtration pressure = 300 kPa; filter area = 23 cm<sup>2</sup>).

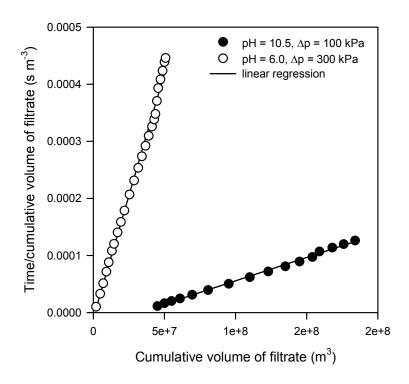


Figure 3: Examples of standard filtration plots where an abrupt change in cake structure is not observed (suspension concentration = 10% v/v; filter area =  $23 \text{ cm}^2$ ).

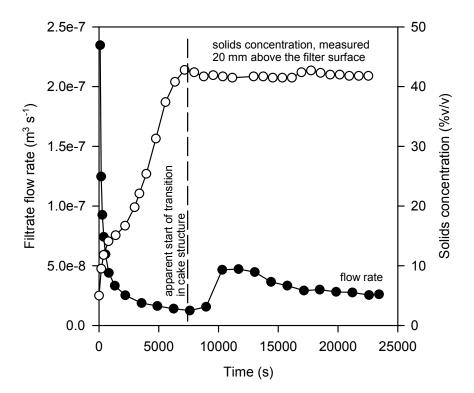


Figure 4: Typical measurements of cake formation and filtration rate where an apparently irreversible change in cake structure has occurred after ~7500 s (suspension concentration = 5% v/v; suspension pH = 2.5; filtration pressure = 450 kPa; filter area = 23 cm<sup>2</sup>).

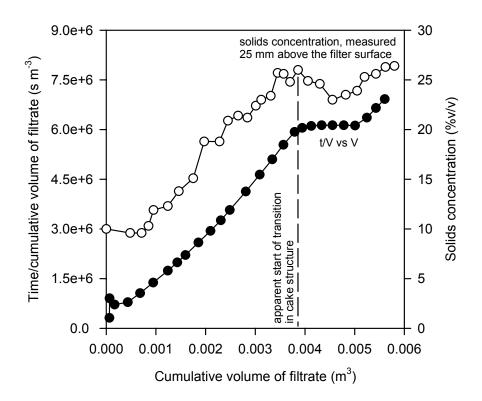


Figure 5: Typical experimental data where a forming filter cake undergoes a seemingly reversible change in structure (suspension concentration = 10% v/v; suspension pH = 6.0; filtration pressure = 200 kPa; filter area = 490 cm<sup>2</sup>).

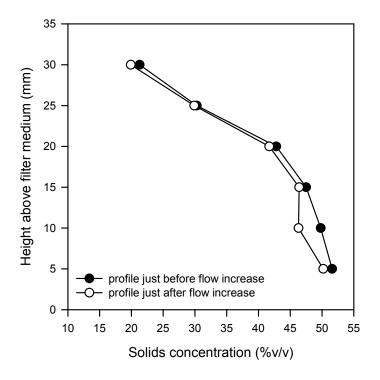


Figure 6: An example of solids concentration profiles just prior to and just after a measured change in cake structure (experimental conditions as given in Figure 4).

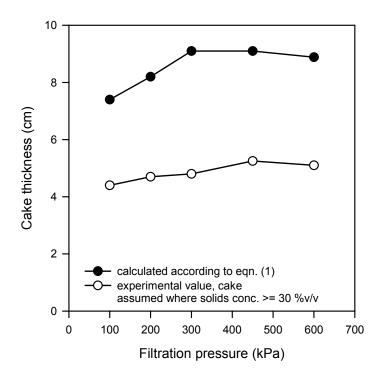


Figure 7: The typical influence of filtration pressure on the thickness of cake at structural transition (suspension concentration = 10% v/v; suspension pH = 2.5; filter area =  $23 \text{ cm}^2$ ).

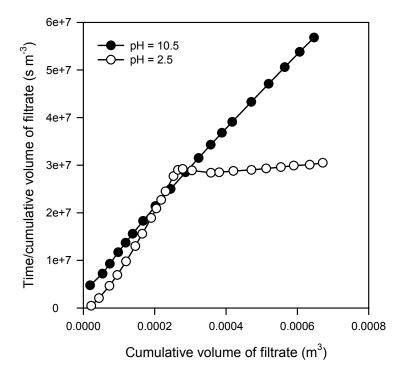


Figure 8: Filtrations for feed suspensions at different pHs where an apparent transition in cake structure has occurred at the lower pH (suspension concentration = 5% v/v; filtration pressure = 600 kPa; filter area = 23 cm²).

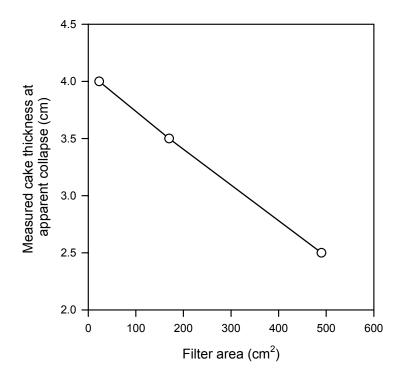


Figure 9: The influence of filter area on the cake thickness at an abrupt change in structure (suspension concentration = 10% v/v; suspension pH = 6.0; filtration pressure = 300 kPa).

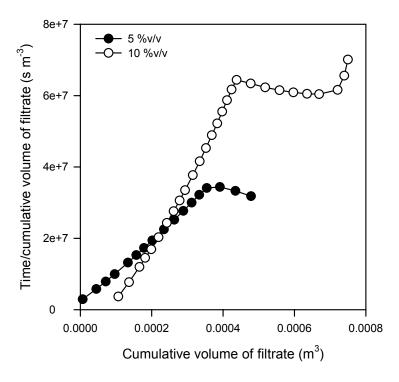


Figure 10: Effects of feed concentration on the timing of abrupt changes in cake structure (filtration pressure = 450 kPa; filter area = 23 cm²; pH = 2.5).

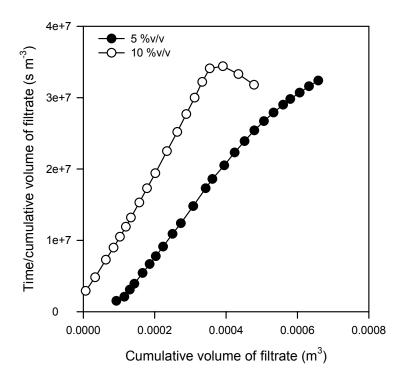


Figure 11: Effects of feed concentration on the timing of abrupt changes in cake structure (filtration pressure = 450 kPa; filter area = 23 cm²; pH = 6.0).