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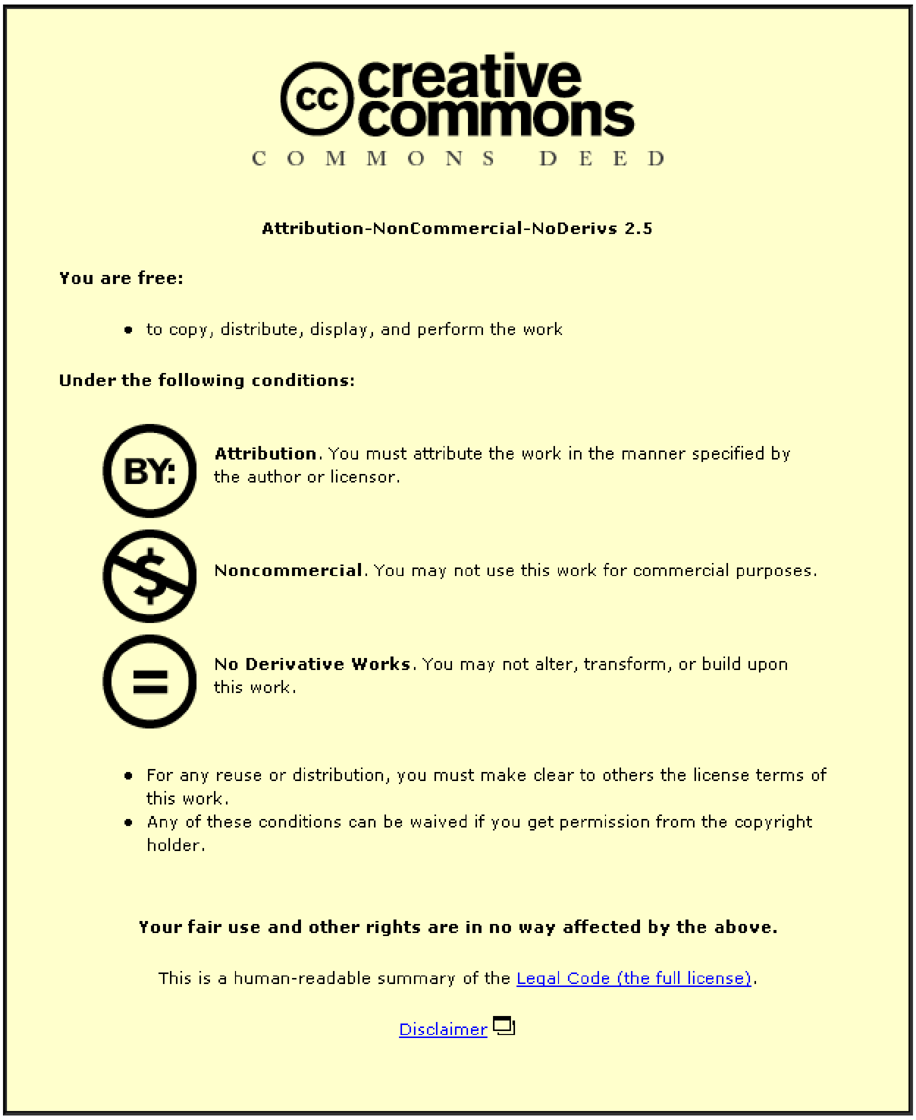

THE IMPORTANCE OF CAKE COMPRESSIBILITY IN DEADEND PRESSURE FILTRATION

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SUMMARY

Particulate suspensions were pressure filtered over a range of experimental conditions using a well controlled deadend leaf filter system. An electrical resistance measuring technique was incorporated in the system to obtain real time transient solids concentration profiles through the filtering suspensions/cakes. Data are shown which illustrate the use of the apparatus and the effects of some process variables and surface charge on cake compressibility. Three different diameter filter cells were used to obtain scale-up data and the need to correctly incorporate these scale-up parameters in filtration models is highlighted.

INTRODUCTION

The industrial filter manufacturer must design a filter that has the ability to deal with the dynamic nature of a feed suspension. The nature of the cake formed upon the septum during a filtration will govern the rate of separation of the remaining suspension. In practice an inhomogenous compressible cake is usually formed, typically with a higher solids concentration next to the septum. The cake compressibility is dependent on variables such as feed solids concentration, filtration pressure, particle size and size distribution, particle shape and surface charge.

This paper presents some results from an ongoing EPSRC funded research program investigating the effects of process variables on filter cake compressibility, pressure filtration characteristics and scale-up.

EXPERIMENTAL APPARATUS AND PROCEDURES

The experimental pressure filtration rig used in the investigation has previously been described in detail[1,2]. In summary it comprised two jacketed stainless steel (s/s) feed vessels with capacities of 5 and 20 litres and three interchangeable filter cells of diameter 2.6, 5.4 and 14.7 cm. The selected filter cell was situated downstream of the feed vessels and connected via s/s piping through pneumatically operated ball valves. The constant driving pressure of up to 800 kPa required for the filtration was supplied by a dedicated air compressor via a controlling regulator valve; this supply also being used to drive the pneumatic ball valves. The sequencing and control of the apparatus was achieved via electronic circuitry, a computer with dedicated software and transducers situated up- and down-stream of the filter cell.

Each of the three filter cells had 30 pairs of s/s electrodes positioned at 5 mm vertical intervals. The electrode pairs were arranged to be diametrically opposite and each electrode protruded 3 mm from the internal cell wall. The electrodes were connected via cabling to the electronic switching and monitoring circuitry housed within a sealed enclosure. To obtain a measure of the solids concentration between a pair of electrodes a square wave signal was applied and the resulting voltage was measured using a Fairchild^{TM} PCL-711S analogue/digital Labcard. Through the use of calibration data and Maxwell's equation this and further readings of voltage could be converted to values of solids concentration. An in-situ real-time transient concentration profile of the filtering suspension/cake could then be obtained[3].

A range of filtration tests were performed with particulate materials such as calcite ($CaCO_3$, supplied by Pumex) and zinc sulphide (ZnS, supplied by Sachtleben Chemie) using the apparatus

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described and 0.2 µm Versapor septa (supplied by Gelman Sciences). To produce a uniform dispersion of zinc sulphide particles an anionic dispersant, Dispex N40, was added. The materials, chosen to represent 'low' and 'moderately' compressible systems, were characterised in terms of particle size and size distribution, shape and ζ-potential vs. pH (see Table 1). Each suspension was prepared by dispersing the necessary particulates in single distilled water. The pH, and hence ζ-potential, of the suspensions was adjusted using analar glacial acetic acid or analar sodium hydroxide as necessary.

The prepared suspension was placed into one of the two feed vessels and mixed for 1.5 hours to obtain an equilibrium temperature of 25ºC. The suspension was then transferred to the filter cell through a computer sequenced operation of valves, and pressure was applied such that deadend filtration commenced. During a filtration, liquid removal rates were monitored using an interfaced electronic balance in conjunction with transient solids concentration profile through the depth of the filtering suspension and cake. These data were graphically displayed in real-time on the computer screen. All experimental data was stored on computer disk to enable further data processing.

RESULTS AND DISCUSSION

Sequences of constant pressure experiments were performed using both calcite and zinc sulphide suspensions over ranges of experimental conditions. When calcite suspensions were filtered, the resistance measurements taken with the electrode probes in the cell indicated the formation of essentially incompressible cakes over the pH range tested. Here, solids concentration would vary little with cake height throughout a filtration, thus giving virtually identical values for average and local cake concentrations[1]. Figure 1 shows that when a zinc sulphide suspension was filtered a more compressible cake was formed where the solids concentration often varied significantly both throughout the cake height and with time. The data presented in Figure 2 show typical variations of specific cake resistance with pressure for filtered calcite and zinc sulphide suspensions. It is seen that specific cake resistance increases with applied filtration pressure at a rate dependent on cake compressibility. For the data presented in Figures 1 & 2 it is believed that only particle size and shape influenced the compressibility of the filter cakes as these tests were performed at pH's corresponding to the iso-electric points of the feed suspensions.

It has previously been reported that for nearly incompressible materials such as calcite, changes in initial solids concentration and pressure have very little effect on either the porosity or specific resistance of a cake when a membrane is employed as the filtering medium[4]. However, for zinc sulphide suspensions at both pH = 2.5 and pH = 6.0, initial solids concentration was seen to induce changes in cake resistance such that higher resistances were recorded for lower feed concentrations. Higher specific cake resistances were also recorded for tests at pH = 2.5 compared to tests performed at pH = 6.0.

Figures 3 and 4 illustrate how surface charge can influence both cake porosity and the rate of liquid removal from the filter for a filtering zinc sulphide suspension. The lowest cake porosity and filtration rate were seen at pH = 10.5 with values of these two parameters increasing for suspensions at pH = 2.5 and pH = 6. Such results are perhaps surprising given that previous work has reported suspensions at their IEP forming looser cake structures[6]. In this previous work the resultant higher rates of filtration have been attributed to the influence of van der Waals forces and particle aggregation. For the work presented here, each zinc sulphide suspension was stabilised using Dispex N40. It is believed that at the IEP, relatively discrete, stabilised particles had the opportunity to form a compact, lower porosity cake, with van der Waals forces inducing only a degree of aggregation. At pH = 6 the particles are still discrete but the repulsion forces increase, thus allowing a higher filtration rate through the cake. By visual inspection a filter cake with a very grainy structure was observed at pH = 10.5. The particles in this case were visually more discrete and closely packed in the cake as would be expected from the data shown in Figure 4. Trends similar to those shown in Figure 4 have been observed for the crossflow membrane filtration of BSA protein and monodisperse silica feeds[7,8].

By obtaining values of specific cake resistance (α) and cake concentration (C) over series of constant pressure tests (where ΔP_c is the pressure gradient over the cake), it was possible to determine values for the scale-up parameters n & α_0 and m & C_0 such that

$$\alpha = \alpha_0 (\Delta P_c)^n \qquad (1)$$

$$C = C_0 (\Delta P_c)^m \qquad (2)$$

Table 2 gives a summary of the scale-up parameters for the tests detailed in this paper. It is seen that for zinc sulphide, the compressibility index (n) depends on particulate surface charge. For calcite, n is low and the cake resistance at unit pressure drop (α_0) is also low compared to the majority of the corresponding zinc sulphide values. Table 3 gives some scale-up parameters for the three different diameters of filter cell obtained under otherwise identical filtration conditions. Here n varies considerably over the three tests as does α_0; the latter changes by two orders of magnitude for the data sequences shown. Although this trend has been found for suspensions at pH = 6, work is still ongoing with other pH values. For each filter cell the cakes formed had apparently different properties, giving different values to the scale-up parameters. Such results give an indication of the difficulties in scaling up from small scale tests to larger sized filters and highlight the need for future modelling to address the challenge of scale-up.

CONCLUSIONS

It has been shown that the degree of cake compressibility influences the filtration characteristics of a suspension. This compressibility is in turn dependent on a number of factors including applied pressure, particle size, particle shape, pH and the nature and magnitude of particle-particle interactions. When modelling compressible cake filtration, future theory must incorporate the most influential particle properties and address the problems of scale-up. It is also noted that both n and α_0 and m and C_0 are inexorably linked and that they must all be known to successfully model filtration.

ACKNOWLEDGEMENT

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FIGURES AND TABLES

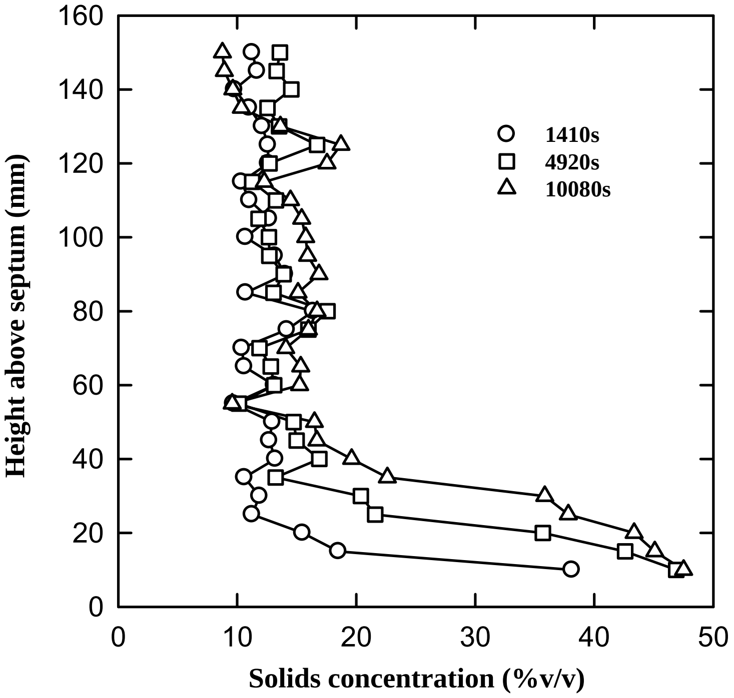

Figure 1: Concentration profiles for a filtering zinc sulphide suspension/cake.

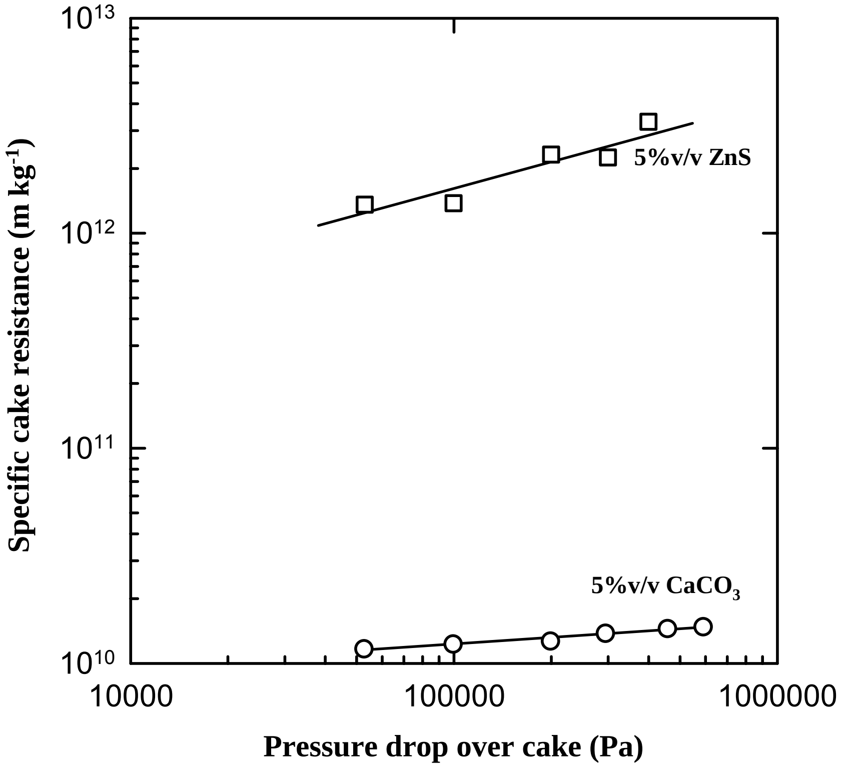

Figure 2: Effect of pressure on average specific cake resistance.

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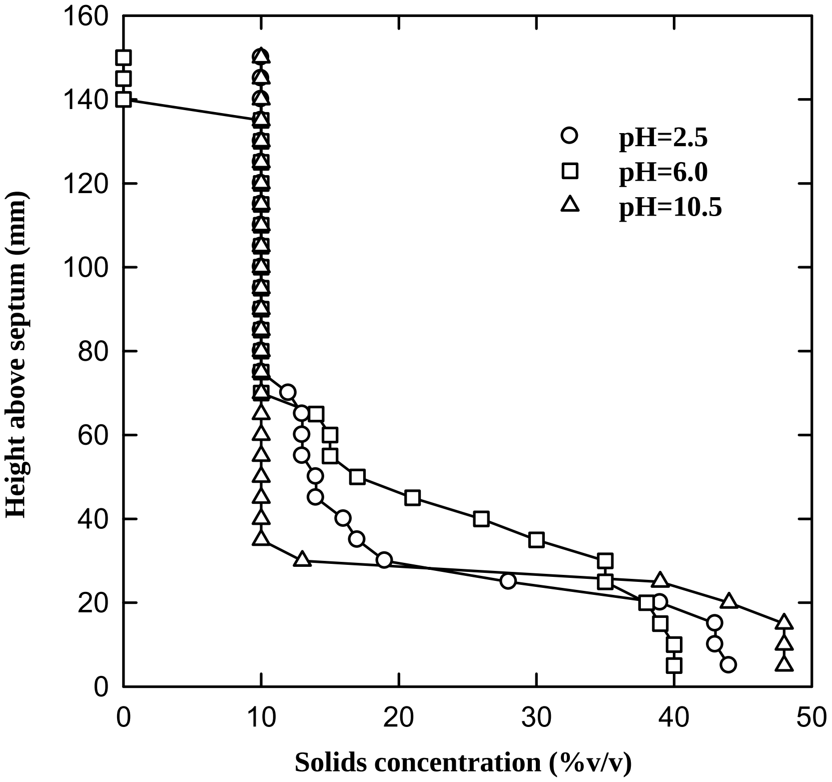

Figure 3: Solids concentration profiles for zinc sulphide suspensions @ *t* = 8000 s.

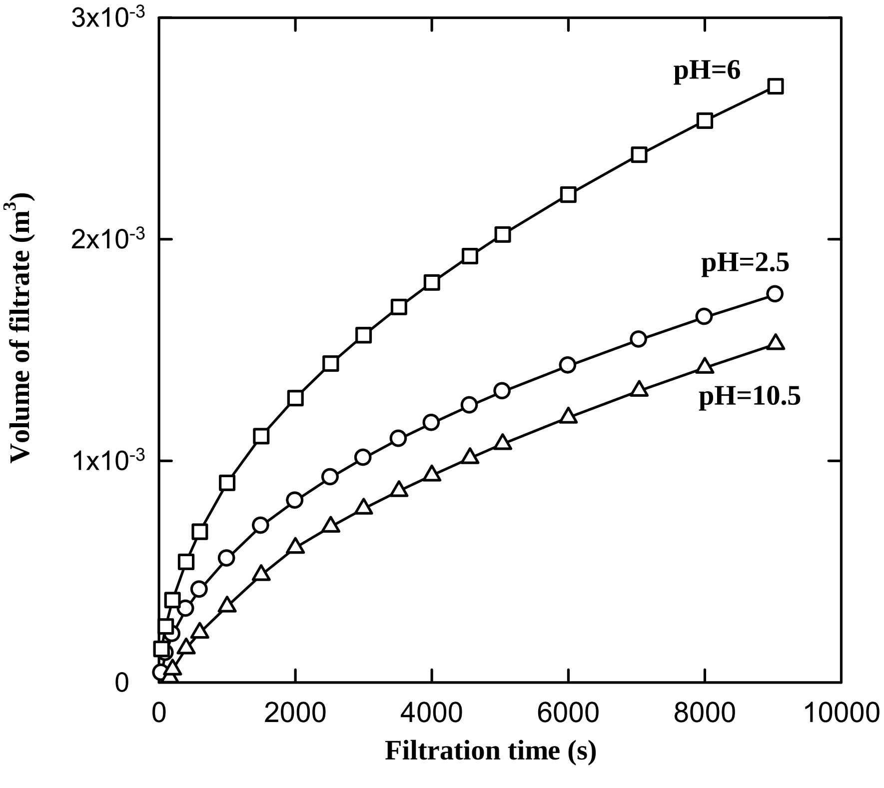

Figure 4: An effect of surface charge on the filtration of zinc sulphide suspensions.

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Solids	10/50/90 % sizes (μm)	IEP* (pH)	ζ-potential (mV)	Shape
$CaCO_3$	0.68/10.16/30.96	9	-20 @ pH = 12	rhomboidal
ZnS	0.15/0.33/0.97	2.5	-50 @ pH = 12	oblate ellipsoid

*IEP ≡ iso-electric point, pH adjusted with analar sodium hydroxide or glacial acetic acid.

Table 1: Some measured particulate characteristics.

Solids	*n* (-)	α_0 (m kg^{-1})	*m* (-)	C_0 (-)
5% v/v ZnS @ pH = 2.5	0.246	1.65×10^{11}	0.082	0.166
10% v/v ZnS @ pH = 2.5	0.245	1.06×10^{11}	0.060	0.191
5% v/v ZnS @ pH = 6.0	0.132	2.54×10^{11}	0.111	0.102
10% v/v ZnS @ pH = 6.0	0.419	5.98×10^{9}	0.130	0.077
5% v/v ZnS @ pH = 10.5	0.050	1.27×10^{12}	0.031	0.301
10% v/v ZnS @ pH = 10.5	0.015	1.72×10^{12}	0.066	0.209
5% v/v $CaCO_3$ @ pH = 9.0	0.084	4.20×10^{9}	0.014	0.355
10% v/v $CaCO_3$ @ pH = 9.0	0.132	2.55×10^{9}	0.023	0.312
20% v/v $CaCO_3$ @ pH = 9.0	0.153	1.79×10^{9}	0.004	0.274

Table 2: Summary of scale-up parameters (filter cell diameter = 5.4 cm).

Cell diameter (cm)	*n* (-)	α_0 (m kg^{-1})	*m* (-)	C_0 (-)
2.6	0.620	4.56×10^{8}	0.068	0.161
5.4	0.419	5.98×10^{9}	0.130	0.077
14.7	0.248	4.66×10^{10}	0.076	0.14

Table 3: Some filtration test data for 10% v/v zinc sulphide suspensions.

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