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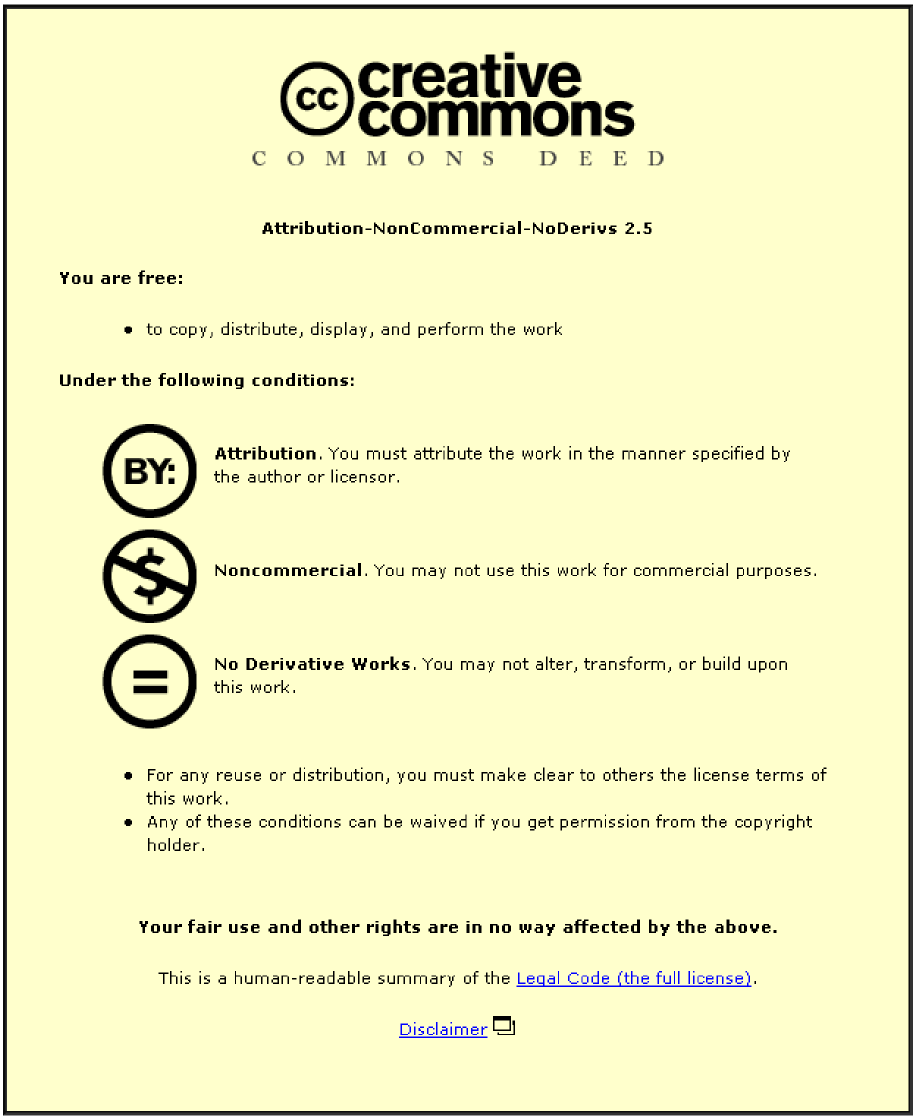

INFLUENCE OF PARTICULATE AND PROCESS VARIABLES IN COMPRESSIBLE CAKE FILTRATION

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Data are reported on particulate and process variables which influence the formation of compressible filter cakes. Variables such as surface charge, particle size and size distribution, filtration pressure and solids concentration have been investigated. To obtain a greater insight into the filter cake behaviour, an electrical impedance imaging technique for determining the in-situ solids concentration in solid/liquid mixtures has been used. Pressure leaf filtration tests of well-characterised solids dispersed in aqueous media have been performed over a range of conditions to investigate the characteristics of compressibility. The majority of the results from these tests are shown through solids concentration profiles as functions of suspension/cake height and time, and specific cake resistance and porosity as functions of pressure. The dependence of the compressibility on surface charge and other variables has been found. Comparisons have been made with the Tiller/Shirato 'modern' filtration theory and the difficulties highlighted.

INTRODUCTION

Incompressible cake filtration theory[1,2] is well documented and has long been used when designing industrial scale filters. Most filter cakes, however, exhibit a varying degree of compressibility which significantly alters the filtration characteristics of the process suspensions. Design procedures for these compressible systems have yet to incorporate all the parameters which affect the compressibility of the cake[3]. Thus, scale-up is often done from (costly) pilot scale plant data with a minimum of physical property data being used in the design of the full scale filter.

This paper presents some results from a research program studying the effects of particulate and process parameters on the compressibility of filter cakes and the filtration characteristics of the feed suspensions. The particulate materials used to form the suspensions were characterised in terms of their particle size, shape and the surface charge present in a range of aqueous environments. The filtered materials exhibited a range of cake compressibility such that when pressure filtered at a number of different conditions comparisons could be made. Design parameters have been found for use in scale-up procedures.

EXPERIMENTAL EQUIPMENT

The experimental constant pressure filtration rig comprised two jacketed stainless steel (s/s) pressure vessels of 5 and 20 litre capacities respectively and a 6.4 cm diameter, 0.71 litre capacity filter cell connected by s/s piping and pneumatically operated ball vales (see Figure 1).

The driving pressure for the filtration was supplied through a controlling regulator valve from a dedicated air compressor at pressures up to 8 bar; this compressor also supplied air to drive the pneumatic ball valves. All valves were controlled/operated and sequenced by computer through dedicated QuickBasic software which used information from pressure transducers situated at appropriate points on the rig.

The filter cell had 30 pairs of electrodes positioned at 5 mm vertical intervals such that each electrode protruded 3 mm from the cell wall. The electrode pairs were connected via cabling to the electronic switching and measuring circuitry housed in a control box. Solids concentrations in the cell could be measured by selecting a pair of electrodes, applying a square wave signal to avoid polarisation and measuring the resultant voltage using a PCL711 analogue/digital labcard. Thus,

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in-situ, real time concentration profiles were obtained through the whole of the cake[4,5] and filtering suspension.

EXPERIMENTAL PROCEDURE

The particulate materials used in the filtration experiments were calcite ($CaCO_3$, supplied by Pumex Ltd) and zinc sulphide (ZnS, supplied by Sachtleban Chemie). An anionic dispersant, Dispex N40 (Allied Colloids Ltd), was added to the zinc sulphide suspension to obtain a more uniform dispersion of particles. Analysis of particle size and size distribution, shape and zeta (ζ-) potential ranges were completed using a Coulter LaserSizer 130, scanning electron micrographs and Malvern ZetaSizer respectively (see Table 1).

The suspensions were prepared by dispersing the particulate material in single distilled water and then adjusting the pH (and hence ζ-potential) of the suspension with the addition of analar glacial acetic acid or analar sodium hydroxide where necessary. All equipment used in the test was repeatedly washed and rinsed in order not to affect the ionic concentration of the filtering suspension.

The feed suspension was placed into the chosen pressure vessel and left mixing for 1.5 hours to obtain an equilibrium temperature of 25°C. The suspension was then transferred to the filter cell by the computer sequenced operation of valves. The driving pressure was subsequently applied and the filtration commenced through a hydrophilic 0.2 μm Versapor membrane (Gelman Sciences Ltd). The quantity of filtrate produced at chosen time intervals was recorded and the slurry/cake within the filter cell was scanned to obtained the analogue/digital data and converted to a concentration profile using calibration information and Maxwell's equation[4]. A real time display of this data was provided on the computer screen by the following plots; t/V vs. V, solids concentration vs. time and solids concentration vs. height through the cake. All experimental data were stored on computer disk.

THEORY

Data produced from the pressure filtration rig has been processed using (adapted) theory developed by Shirato *et al.*[6].

The conventional filtration equation given originally by Sperry[7] describes the filtration of incompressible cakes. For compressible cake both the effective feed concentration (c, kg m^{-3}) and the specific cake resistance (α, m kg^{-1}) are assumed constant average values and so the equation can be integrated for constant pressure filtration to give

$$\frac{t}{V} = K_1 V + K_2 \qquad (1)$$

where $K_1 = \alpha\mu c/(2A^2\Delta P)$ and $K_2 = \mu R_m/(A\Delta P)$. V is the volume of filtrate (m^3) of viscosity μ (N s m^{-2}), collected in time t (s) from a filter area A (m^{-2}) with a medium resistance of R_m (m^{-1}) at a constant applied pressure of ΔP (N m^{-2}). By constantly monitoring t and V during an experiment, equation (1) can be used to evaluate the important design parameter α with c being found from a knowledge of the solids concentration through the filter cake.

From a series of tests at various applied pressures, the following equations can be applied to obtain empirically derived constants[8] over a given pressure range.

$$\alpha = \alpha_0 \Delta P_s^n \qquad (2)$$

$$C = C_0 \Delta P_s^m \qquad (3)$$

where α_0, C_0, n and m are empirical constants. These constants can subsequently be used to obtain a theoretical solids concentration through the cake[1] which can then be used for comparison with experimental data.

$$C = C_0 \Delta P_c^m \left(1 - \frac{x}{L}\right)^{\frac{m}{(1-m-n)}} \qquad (4)$$

where ΔP_c is the pressure drop over the cake (N m^{-2}), x is the distance from the medium (m) and x/L is the dimensionless distance into the cake measured from the medium.

RESULTS AND DISCUSSION

Suspensions of calcite and zinc sulphide were filtered under constant pressure over a range of experimental conditions. For the data shown in Figures 2-4 both suspensions were prepared to the same initial solids concentration with the pH's' adjusted where necessary to the iso-electric points (IEP). Figure 2 shows the solids concentration profile in the filter cell, obtained for calcite after 500 s filtration. The incompressible cake of height 35 mm has essentially the same concentration at all positions within the cake and so its average cake concentration will be identical to the local concentrations throughout the cake. Zinc sulphide, however, produces a compressible cake with the solids concentration varying significantly through both the cake height and with time as shown by Figures 3 and 4.

Applying equations (1) to (3) to sequences of experiments the specific cake resistances given by these particulates at a number of constant applied pressures are presented in Figure 5. It can be seen that the compressible zinc sulphide filter cake yields significantly higher specific cake resistances for varying pressures and that both the calcite and zinc sulphide specific cake resistances increase to varying extents for an increase in applied filtration pressure. For the data shown in Figures 2-4, only particle size and shape were thought to influence the compressibility of the filter cakes.

For an incompressible material such as calcite, changes in initial solids concentration and pressure have very little effect on porosity of the cake or the specific cake resistance[9,10]. However, Figure 6 shows that decreasing the initial solids concentration will increase the cake resistance of both a zinc sulphide suspension at its IEP (pH = 2.5) and at pH = 6.0, thus showing the importance of initial solids concentration. Figure 6 also shows an increasing specific cake resistance for a decrease in pH. The average porosity of these cakes can be seen in Figure 7 with a minimum porosity shown for 5% v/v zinc sulphide at pH = 2.5.

The effect of surface charge on the filtration of zinc sulphide suspensions is seen in Figures 8 and 9. The lowest porosity cake and filtration rate is seen for the suspension at pH = 2.5 with the highest value of porosity and rate of filtration for a suspension at pH = 6.0. The observed effects of the particle surface charge are perhaps surprising. In general it is assumed that a suspension at its IEP will aggregate due to van der Waals forces and form a looser cake structure with correspondingly higher rates of filtration[11-13]. The results seen here indicate that this does not always happen and by visual observation of the cakes formed in this set of experiments it is believed that the particles have opportunity to form a compact, lower porosity cake at the IEP due to the stabilisation of the particles with Dispex N40 and the influence of van der Waals forces. A filter cake formed at pH = 6.0 is also made up of stabilised discrete particles and with the additional presence of repulsion forces this allows a high filtration rate through the cake. These trends have been previously observed with the membrane filtration of BSA protein and monodisperse silica feeds[14,15].

Table 2 shows a summary of the scale-up parameters from the tests detailed in this paper. It can be seen that the measure of cake compressibility, *n*, is significantly higher for zinc sulphide suspensions with the cake resistance at unit pressure drop also being higher than for calcite suspensions. Figures 10 and 11 show how these scale-up constants have been used with equation (4) to compare theory and experimental data. Both figures have been produced from data obtained for the filtration of 10% v/v suspension, but the calcite particles have zero net charge whilst the zinc sulphide particles have a ζ-potential of -30 mV. For the calcite suspension at its IEP, it can be seen that Tiller and Shirato's theory models very well the behaviour of this incompressible system. Problems arise when a material with a significant surface charge is modelled as shown in Figure 11. The most significant error can be seen to be closest to the cake/medium interface which is thought to be the most influential portion of the compressible cake to the filtration process. Future models will aim to incorporate factors which are affected by parameters such as surface charge in order to accurately describe more compressible systems.

CONCLUSIONS

The compressibility of a particulate filter cake is dependent on a number of factors including applied pressure, particle size, particle shape, pH and the nature and magnitude of particle-particle interactions. Data presented in this paper have shown how the compression characteristics of a filter cake can change with these basic particle and process parameters of the filtering suspension and the subsequent effect on the filtration rate. Further theory must be developed to incorporate the most influential particle properties in models to accurately scale-up compressible cake filtration theory.

ACKNOWLEDGEMENTS

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TABLES AND FIGURES

Solids	10% size (μm)	50% size (μm)	90% size (μm)	Isoelectric point (pH)	Maximum ζ-potential (mV)	Shape
$CaCO_3$	0.68	10.16	30.96	9.0	-20 @ pH = 9	rhomboidal
ZnS	0.15	0.33	0.97	2.5	-50 @ pH = 12	oblate ellipsoid

Table 1: Characteristics of particulates used in this study.

Solids	n (-)	α_0 (m kg^{-1} Pa^{-n})	m (-)	C_0 (-)
pH = 2.5				
5% v/v ZnS	0.43	2.01×10^{10}	0.079	0.162
10% v/v ZnS	0.36	3.12×10^{10}	0.057	0.195
pH = 6.0				
5% v/v ZnS	0.23	9.62×10^{10}	0.12	0.096
10% v/v ZnS	0.395	6.22×10^{10}	0.066	0.168
pH = 9.0				
5% v/v $CaCO_3$	0.101	4.23×10^{9}	0.0045	0.404
10% v/v $CaCO_3$	0.106	3.85×10^{9}	0.013	0.359
20% v/v $CaCO_3$	0.157	1.81×10^{9}	0.008	0.392

Table 2: Summary of filtration test results (ΔP in Pa).

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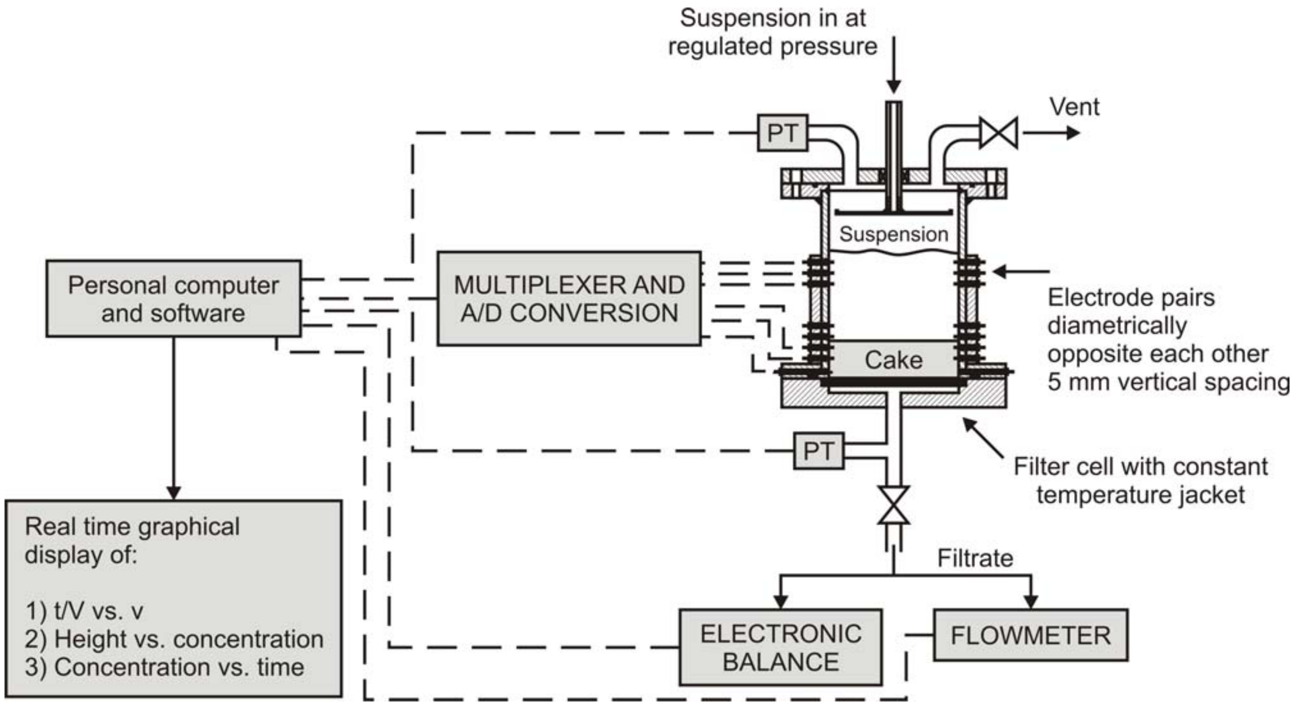

Figure 1: Schematic of pressure filtration rig.

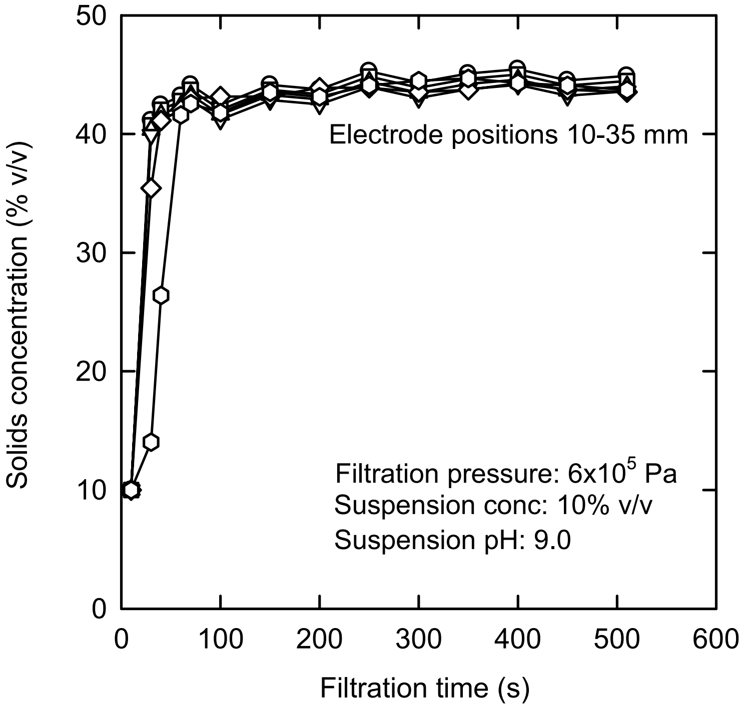

Figure 2: Transient concentration profiles for the pressure filtration of a calcite suspension.

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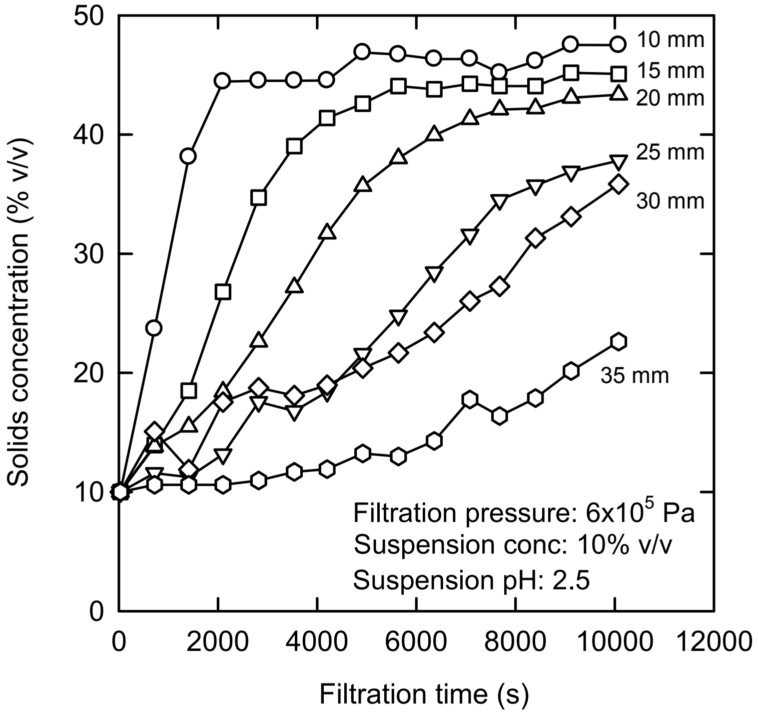

Figure 3: Transient concentration profiles for the pressure filtration of a zinc sulphide suspension.

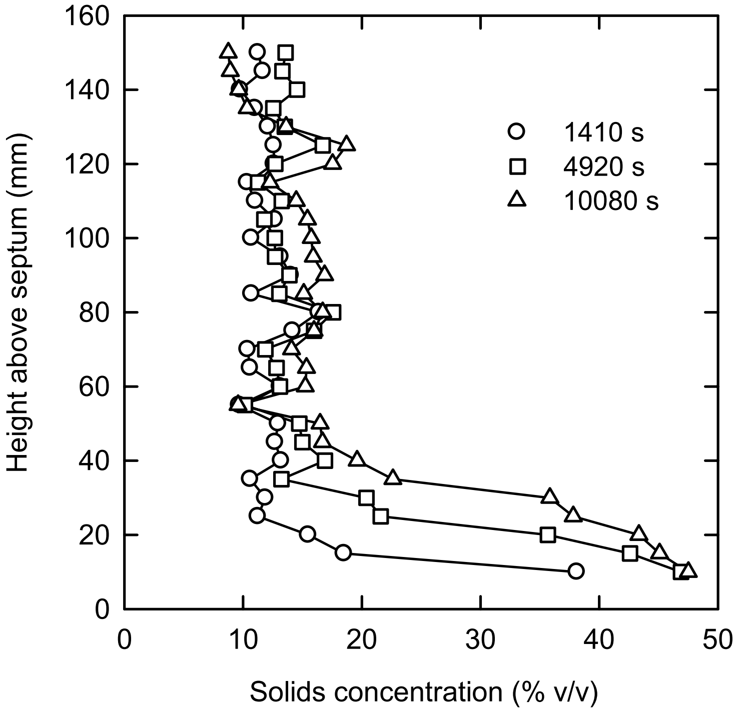

Figure 4: Concentration profile of a filtering zinc sulphide suspension/cake.

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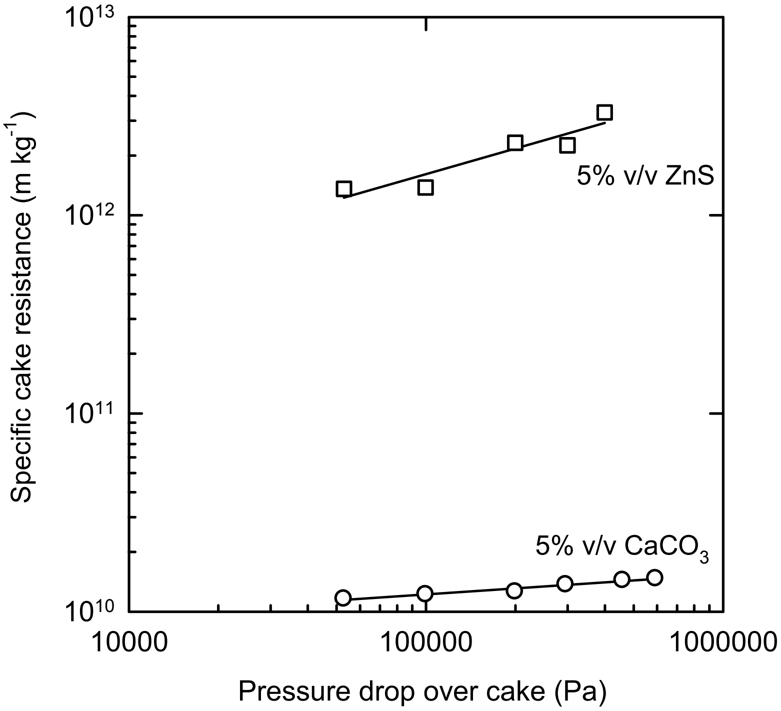

Figure 5: Effect of pressure on average specific cake resistance.

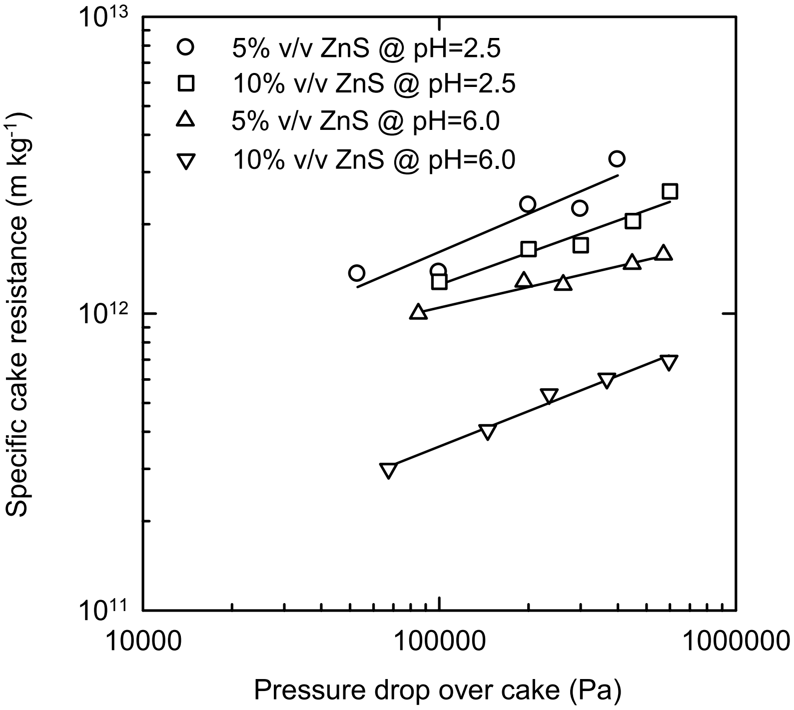

Figure 6: Effect of pressure, feed concentration and surface charge on average specific cake resistance for a zinc sulphide suspension.

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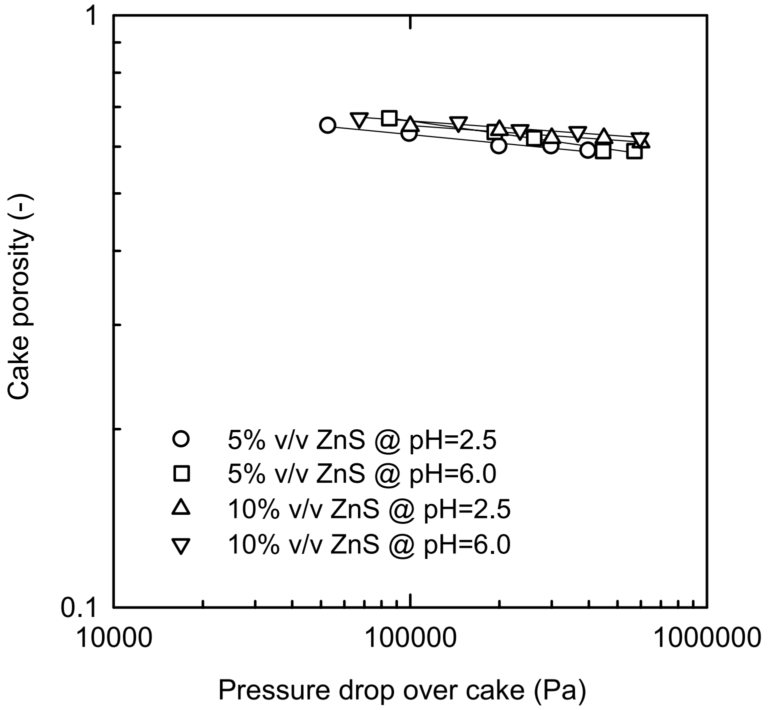

Figure 7: Effect of pressure, feed concentration and surface charge on cake porosity for a zinc sulphide suspension.

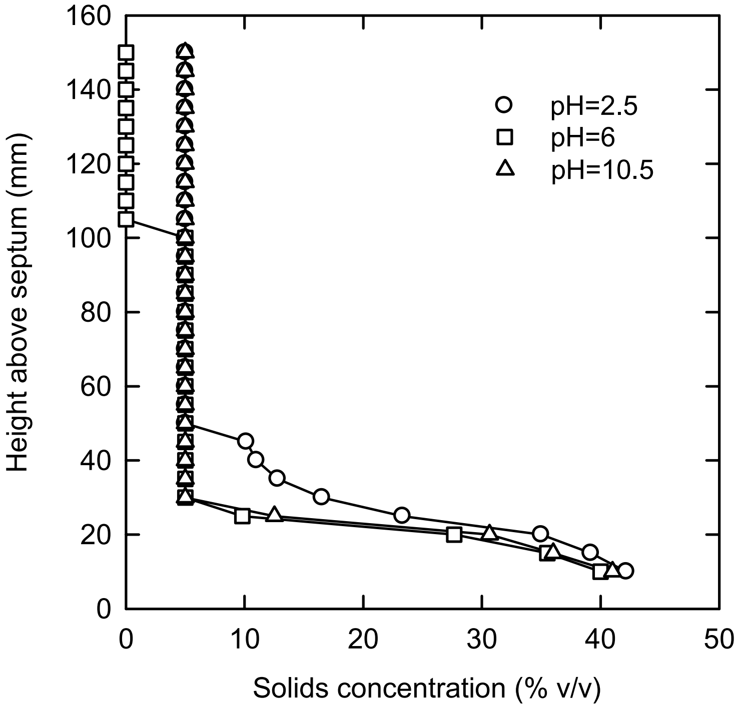

Figure 8: Effect of surface charge on the filtration of a zinc sulphide suspension, *t* = 8000 s.

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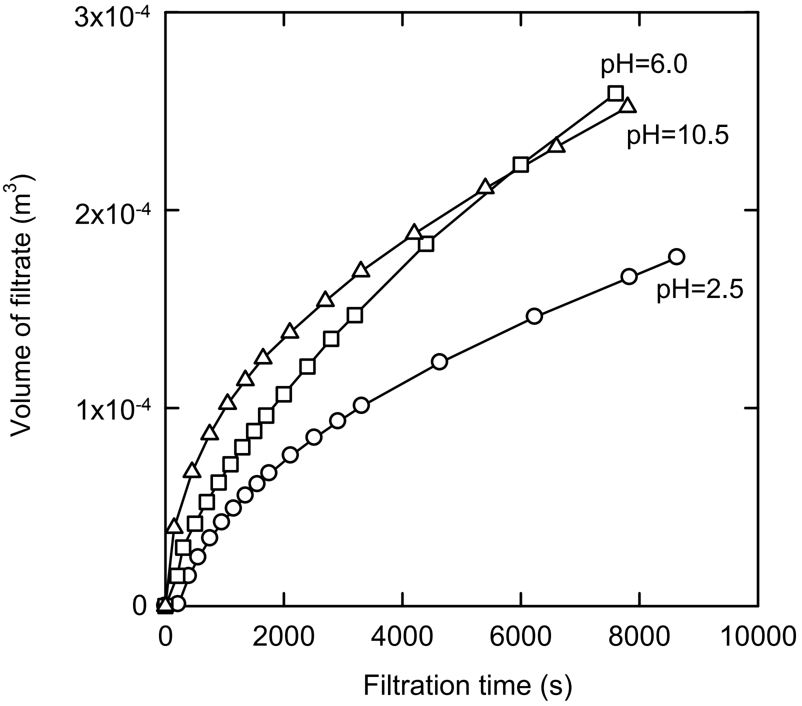

Figure 9: Effect of surface charge on the filtration rate of a zinc sulphide suspension, *t* = 8000 s.

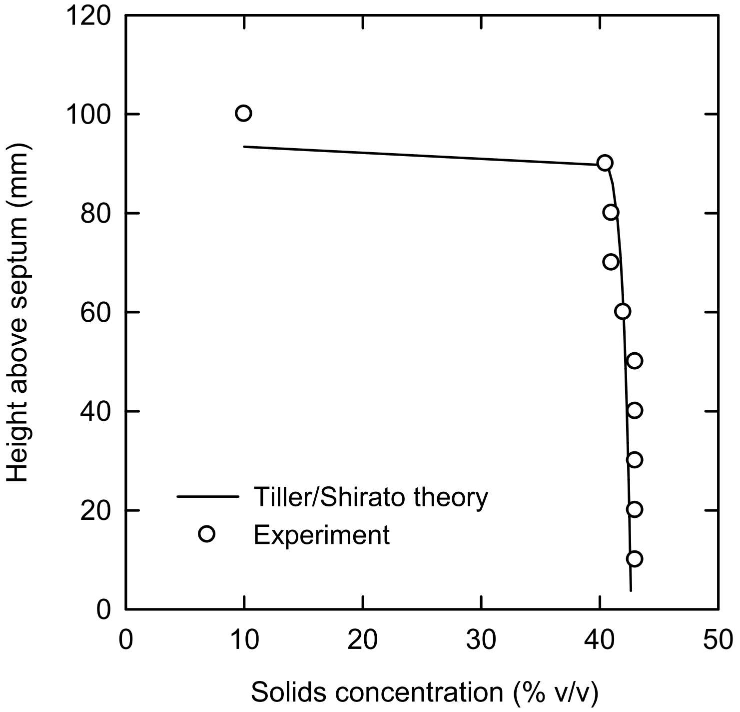

Figure 10: 'Modern' filtration theory applied to calcite data, 10% v/v, pH = 9.0.

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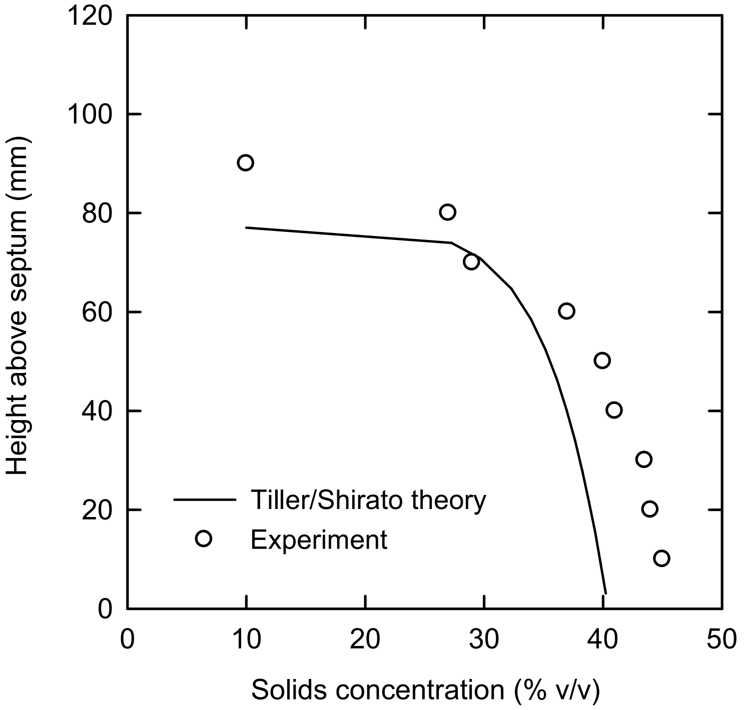

Figure 11: ‘Modern’ filtration theory applied to zinc sulphide data, 10% v/v, pH = 6.0.

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