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FILTRATION BEHAVIOR OF SLURRIES WITH VARYING COMPRESSIBILITIES

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ABSTRACT

A novel filtration apparatus allows simultaneous measurements of filtrate volume, hydraulic pressure and cake thickness using slurry volumes on the order of 100 cm^3. Differences in interparticle interactions were studied by varying the barium chloride concentration of 0.38-μm polystyrene latex and filtering at pressures between 2 and 100 psi. Cakes formed from these slurries are highly compressible for concentrations between 0.01M and 0.10M, moderately compressible for the 0.005M concentration, and incompressible for the 0.001M concentration. Plots of filtrate volume versus cake thickness were linear for the incompressible cakes, whereas the compressible cakes showed significant deviations, which were pressure dependent. The pressure distribution for the incompressible cake was found to be essentially linear as predicted from the resistance plots assuming constant α and ε. For the highly compressible cakes, most of the pressure drop appears to occur near the cake/medium interface with only small changes occurring at the top of the cake.

INTRODUCTION

Understanding and predicting the behavior of slurries in cake filtrations is important to many industrial processes. Several laboratory-scale tests for measuring the filterability of a given slurry have been devised. Although many of these methods, e.g., capillary suction (1-4) and aggregate size/filterability (5-8), are valuable for determining optimum flocculant dosages for clarifying dilute dispersions and for determining the limiting low-pressure specific resistance, they cannot predict how the pressure gradient, porosity distribution, and specific resistance will vary as a function of cake thickness and applied pressure. The novel filtration cell we have developed lets us measure these variations and thus helps define the relationships between the internal cake properties, cake thickness, and applied pressure (9). Once these relationships are understood, one can determine the parameters necessary to optimize the filtrations of a given material. The apparatus, which is easy to use and clean and requires sample sizes as small as 100 cm^3, will hopefully become a more useful benchtop tool for determining the filtration behavior of slurries. In this paper, we describe the use of this apparatus in measuring the filtration behavior of uniform polystyrene and silica dispersions.

APPARATUS AND METHODS

A detailed description of the filtration apparatus is given in Ref. (9). Briefly, the apparatus consists of a pressure manifold, a filtration vessel, pressure transducers, an electronic balance, and a data acquisition system. The pressure manifold supplies compressed gas to the filtration vessel, which consists of a reinforced Plexiglas cylinder with a filter medium at the bottom. Ten ports are spaced in a helical arrangement at known heights above the filter medium. Each port is connected to a pressure transducer. The outputs from the transducers, together with those from the digital balance on which the filtrate is collected, are input to an IBM PC. This layout permits simultaneous measurements of filtrate volume, hydraulic pressure, and cake thickness. Values for cake thickness (L) as a function of filtrate volume (V) were obtained from the point at which the pressure measured at each transducer began to drop, as discussed in (9) and illustrated in Fig. 1.

The polystyrene latex dispersions used in the present study were prepared by emulsion polymerization (10,11) under the conditions given in (12). The silica dispersions were prepared according to the procedure of Stober, Fink, and Bohn (13). This procedure involves the hydrolysis of an alkyl silicate and the subsequent condensation of the resulting silicic acid in an alcoholic solution with ammonia as a morphological catalyst. Different sizes of silica particles were obtained by varying the molar ratio of ammonia to water as well as the reaction temperature.

The 0.24-µm silica dispersion was obtained with reagent concentrations of 0.4M tetraethyl orthosilicate, 1.6M ammonia, and 16.0mM water in a 1:3 solvent system of n-propanol/methanol. The reaction was carried out in a round-bottom flask equipped with an overhead stirrer and maintained in a water bath at 55°C. The resulting silica dispersion was washed with water by repeated centrifugation and resuspension. A pH of 9 was measured for the

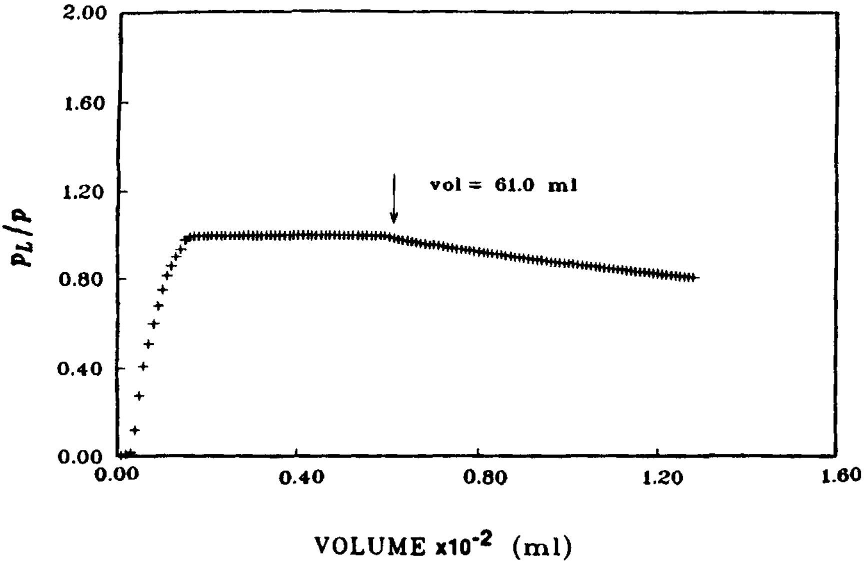

Figure 1. Hydraulic pressure (p_L/p) versus filtrate at port #4 for 0.38-μm latex ([$BaCl_2$]=0.01M; p=100 psi).

final washed dispersion and a pH of 8 was obtained for the slurry treated with barium chloride or potassium nitrate before filtration. Particle sizes were determined by transmission electron microscopy and light scattering. Number average diameters are reported for these monodisperse particles.

Stable slurries of the polystyrene and the silica were filtered in the presence of 0.01M potassium nitrate. Coagulated slurries were prepared by the addition of barium chloride. Different ionic strengths were generated by varying the barium chloride concentration between 0.001M and 0.1M. For each barium chloride concentration, a slurry volume of 150 ml and a slurry concentration of four percent for the polystyrene and eight percent for the silica was used. Both stable and coagulated slurries were allowed to sit for two days and were then resuspended by gentle agitation before filtering at pressures between 2 and 100 psi. After each filtration, the weight and thickness of the wet cake were measured before placing it in a 50°C oven for drying to constant weight.

DATA TREATMENT AND CALCULATIONS

Filtration rate data are usually analyzed according to the resistance equations using mass coordinates (14-16). However, Tiller recently noted that this practice leads to specific resistance values which depend upon the density of the particles used (17). We have adopted his suggestion and thus the rate data were analyzed according to the resistance equations based on solid cake volume per unit area (ω_c):

$$\frac{p}{\mu q} = \alpha_{av}\omega_c + R_m \tag{1}$$

and

$$\frac{pt}{\mu v} = \left(\frac{\alpha_{av}}{2}\right)\omega_c + R_m \tag{2}$$

where p is the applied pressure, μ the filtrate viscosity, q(=dv/dt) the flow rate of filtrate per unit area of cake, v the volume of filtrate per unit area of cake accumulated in time t, α_{av} the average cake resistance, and R_m the medium resistance. Note that in equations (1) and (2), ω_c has replaced the mass of dry cake per unit area ($w_c = \omega_c \rho_s$) and the mass-based average specific resistance equals α_{av}/ρ_s, where ρ_s is the density of the particles. Since Eq. (2) involves more assumptions, Eq. (1) alone was used to calculate α_{av}.

Values for ω_c were calculated from filtrate volumes using the mass balance (assuming no volume changes):

$$\omega_c = \frac{v\phi_s}{1 - \frac{\phi_s}{\varepsilon_{sav}}} \tag{3}$$

where ϕ_s and ε_{sav} are the volume fractions of solids in the slurry and the cake, respectively. ϕ_s was calculated from measured values of the mass fraction of solids in slurry(s) using:

$$\phi_s = \frac{s}{\sigma(1-s)+s} \tag{4}$$

where σ is the ratio of solid to filtrate densities (ρ_s/ρ). ε_{sav} is linked to L and v through material balance and geometric considerations, and may be calculated from any of these three relationships:

$$\varepsilon_{sav} = \frac{s_c}{\sigma(1-s_c)+s_c} \tag{5}$$

$$\varepsilon_{sav} = \frac{s(1 + \frac{v}{L})}{\sigma(1-s)+s} \tag{6}$$

$$\varepsilon_{sav} = \frac{s}{\sigma(1 - \frac{s}{s_c})}\frac{v}{L} \tag{7}$$

where s_c is the mass fraction of solid in the cake. Equation (5) uses the measured value of s_c alone to calculate ε_{sav}, Eq. (6) uses the measured values of v and L, and Eq. (7) uses all three measurements. Ideally, all three calculations should give the same answer and this is the case for relatively incompressible cakes (9). However, the more compressible cakes studied here showed signs of expression, i.e., water squeezed out of the pores. Therefore, Eq. (5), which assumes that all the pores are filled with water, gave ε_{sav} values that were too high. Consequently, Eq. (6) was used to calculate ε_{sav}. In obtaining the resistance plots, ε_{sav} was calculated from the final thickness and filtrate volume and was assumed to be constant throughout the filtration. In these calculations, the density of polystyrene is assumed to be 1.05 g/ml and that of the silica 2.20 g/ml. The average porosity of the cake (ε_{av}) is equal to $1-\varepsilon_{sav}$.

RESULTS

Our previous work focused on the filtration of flocculated and nonflocculated slurries of uniform polystyrene particles. Dispersions, stable in the presence of 0.01M potassium nitrate, behaved as incompressible materials upon filtration. Slurries of 0.67-μm particles coagulated with 0.1M barium chloride behaved like moderately compressible material. Plots of filtrate volume versus cake thickness confirmed a linear relationship for incompressible cakes as well as a linear pressure drop through the cake. Average porosity was also found to be independent of cake thickness. In the case of moderately compressible cake, both sedimentation and compaction appeared to occur during filtration, resulting in deviations from the results obtained for the incompressible cake (9); this will be the starting point for this paper. Our present work involves studying the differences in interparticle interactions. Slurries of the 0.38-μm latex were filtered in the presence of varying concentrations of barium chloride and were compared with the slurries of silica at similar ionic strengths.

Silica slurries, filtered in the presence of 0.01M potassium nitrate, formed incompressible cakes. The α_{av} values for different diameters (d) of silica are shown in Fig. 2 together with values of polystyrene and a theoretical curve from the Carman-Kozeny equation using average values for porosity and resistance (9,16)

$$\alpha_{av} = kS_o^2 \frac{(1-\varepsilon_{av})}{\varepsilon_{av}^3} \qquad (8)$$

where S_o is the area per unit volume of particles (=6/d), ε_{av} = 0.37, and the constant k = 5. By using the volume-based resistance values, the silica particles lie on the same curve as the polystyrene and their resistances are well predicted by the Carman-Kozeny equation.

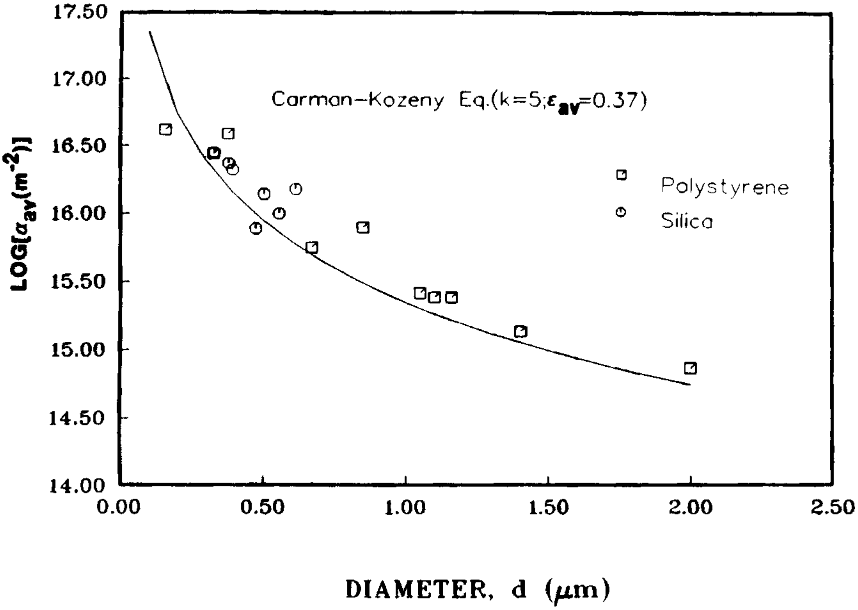

Figure 2. Filtration resistance (α_{av}) as a function of particle diameter

Figure 3 shows resistance plots obtained for the polystyrene slurries at different concentrations of $BaCl_2$. As can be seen from the resistance plots, there is a distinct curvature at the higher ionic strengths. Nevertheless, α_{av} values were obtained by linear regression excluding the first and final points. The effect of pressure on the α_{av} values is summarized in Fig. 4. For the 0.001M slurry, resistance is independent of pressure with an average value of $1.53 \pm 0.25 \times 10^{16} m^{-2}$. A constant value of $4.40 \pm 0.54 \times 10^{15} m^{-2}$ is also obtained for the slurry treated with 0.005M $BaCl_2$. In the case of the coagulated slurries having higher $[BaCl_2]$, the plot shows a linear rise in resistance with increasing pressure, indicating the highly compressible nature of these cakes. However, there does appear to be a slight curvature toward the end of the plot around 50 psi. Average porosity (ε_{av}), with a value of 0.429, is also found to be independent of pressure for the 0.001M slurry. The effect of pressure on porosity for the other slurries is not as well defined. The general trend appears to be a decrease in porosity with increasing pressure.

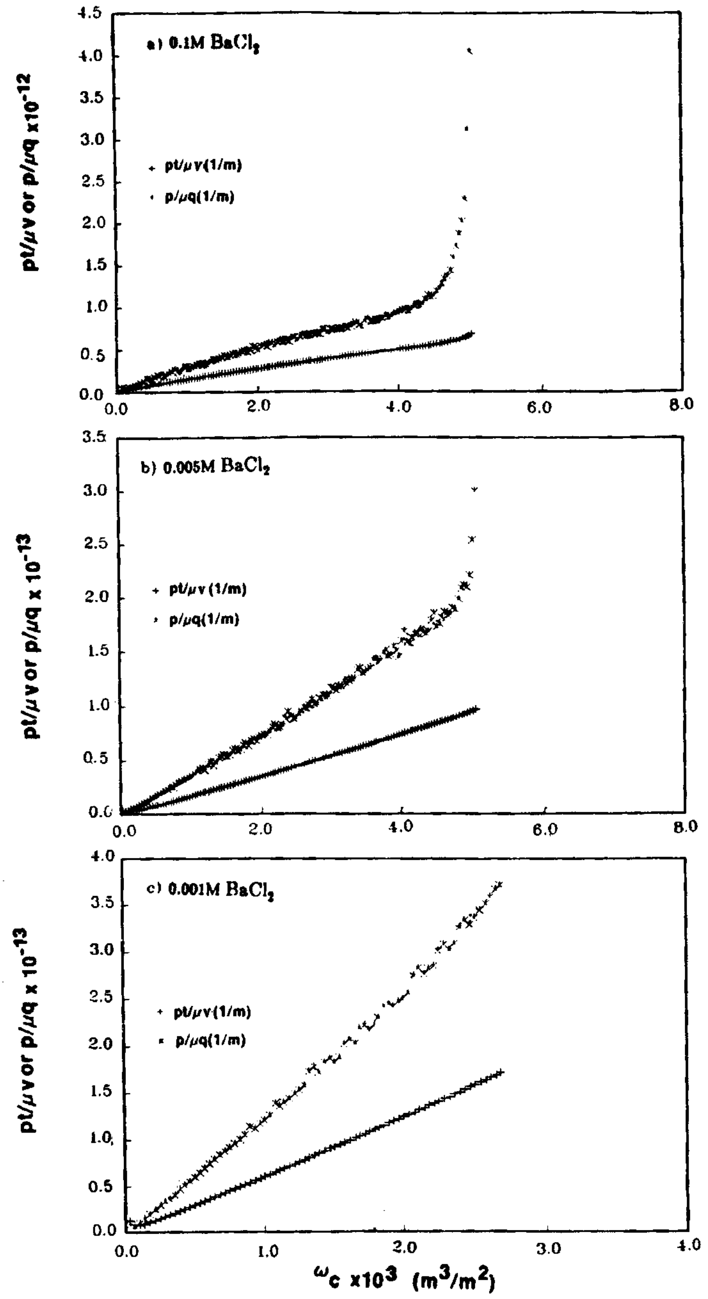

Figure 3. Resistance curves for 0.38-μm polystyrene for three BaCl$_2$ concentrations at 2 psi: +=pt/μv, x=p/μq.

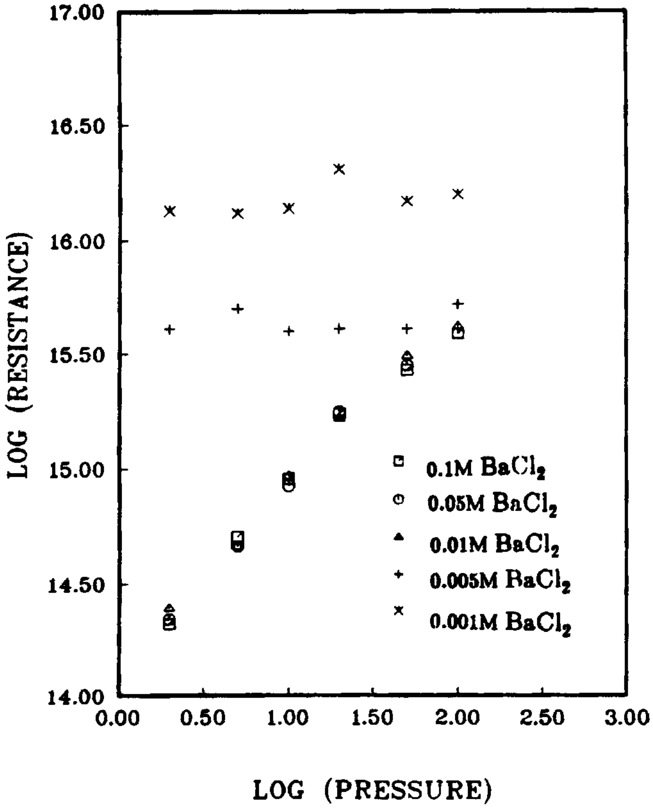

Figure 4. Plot of α_{av} as a function of pressure for the 0.38-μm latex.

Figure 5 shows filtrate volume versus cake thickness plots at 20 psi. Slurries filtered in the presence of 0.001M $BaCl_2$ give linear plots that have good agreeement between the cake dimensions and the thickness measurements during filtration. This behavior corresponds to the incompressible cakes obtained with stable dispersions. Slurries treated with 0.01, 0.05, and 0.1M $BaCl_2$, which formed highly compressible cakes, gave similar plots. These plots initially rise linearly and then flatten out to a plateau, indicating relatively large increases in cake thickness with small volumes of filtrate. The behavior of the 0.005M slurry appears to be a transition from the highly compressible cake to the incompressible. The shape of the plots for this slurry are

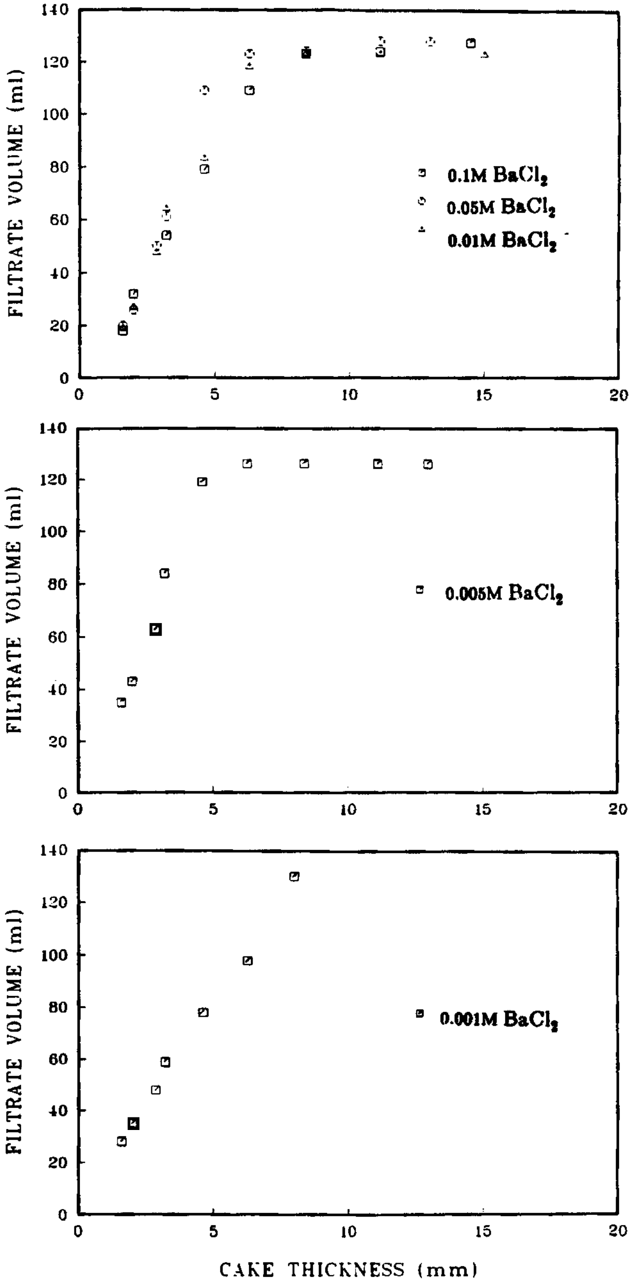

Figure 5. Filtrate volume versus cake thickness for polystyrene in the presence of five $BaCl_2$ concentrations (p=20 psi).

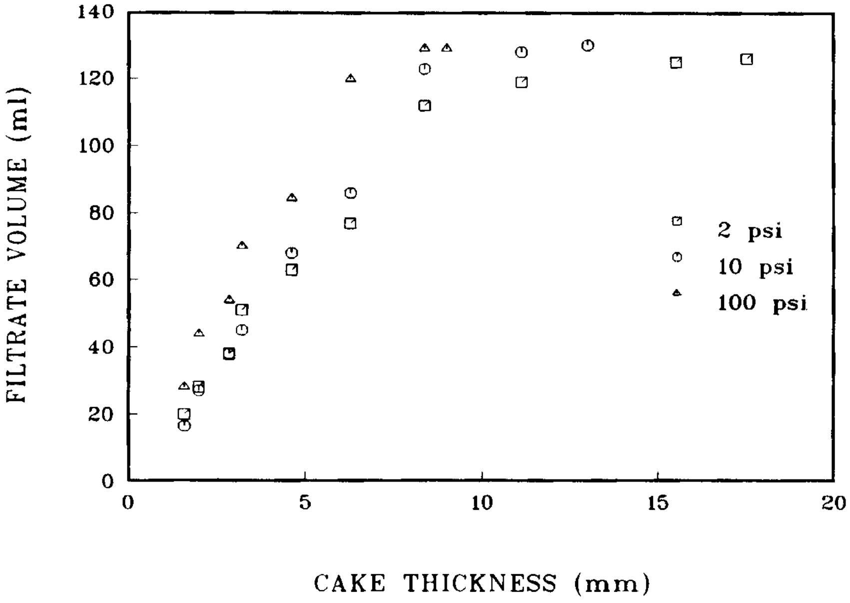

Figure 6. Filtrate volume versus cake thickness for polystyrene at different pressures ($[BaCl_2]=0.01M$).

similar to the moderately compressible cakes (9) where the breaks in the plots are not as pronounced as in the highly compressible cakes. With increasing pressure, the plateau also becomes less pronounced in the highly compressible cakes as seen in Fig. 6.

The shape of the V versus L plots for the highly compressible cakes is contrary to expectations. The sharp initial rise implies that a very tightly packed cake forms at first up to a certain volume at which a very loosely packed cake begins to form. This gives rise to the sharp rise in L while only a small amount of filtrate volume flows towards the end of the filtration. At first, this occurrence might seem reasonable, since most of the pressure drop in a highly compressible cake occurs near the cake/medium interface (14,16). One explanation is that so much of the pressure drop is borne by the medium that the height of the cake has to go above the port hole before sufficient drop occurs for the transducers to register. The plateau effect presumably is due to compaction of the very loosely packed cake

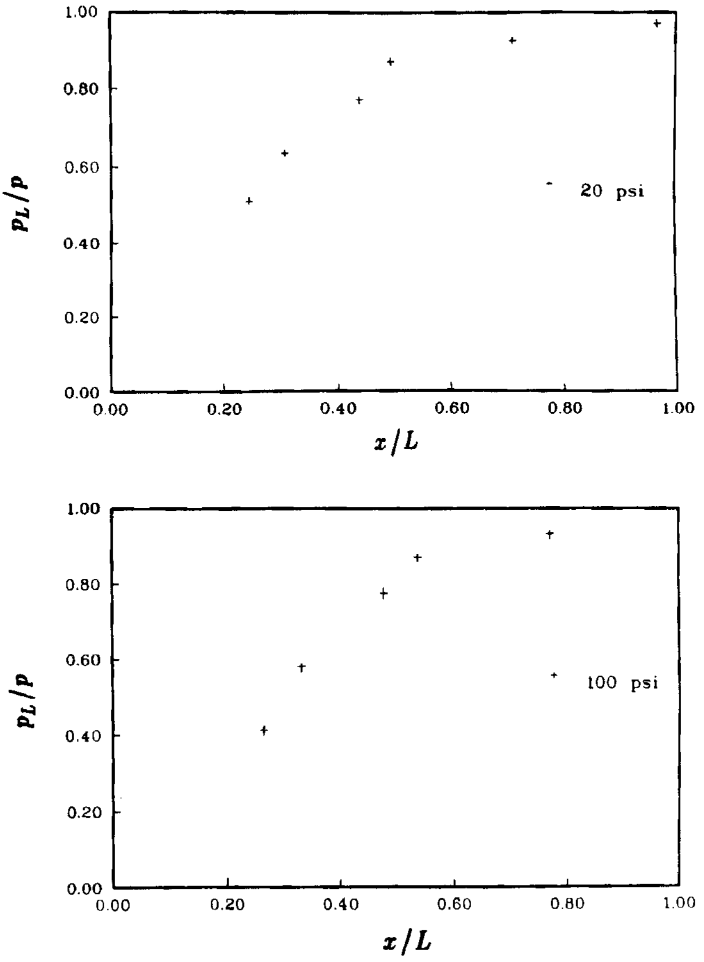

Figure 7. Hydraulic pressure (p_L/p) versus port height/cake thickness (x/L) for polystyrene ($[BaCl_2]=0.1M$).

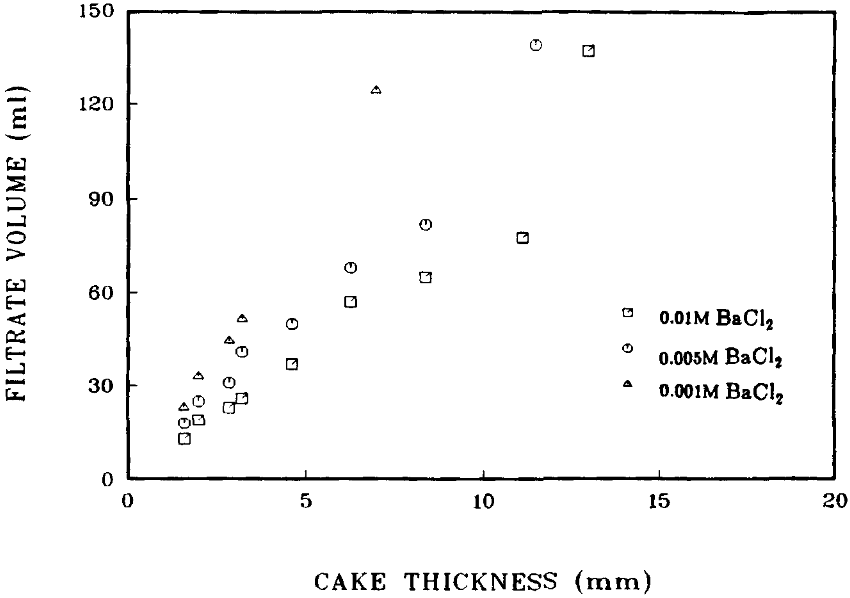

Figure 8. Filtrate volume versus cake thickness for 0.24-μm silica in the presence of different $BaCl_2$ concentrations (p=5 psi).

during the final filtration stages. That the pressure drop across most of the cake is small is seen in Fig. 7, which shows plots of p_L/p versus x/L taken toward the end of the filtration (110 ml) to avoid ambiguities in the value of L.

There is an alternative explanation. During the course of our study with polystyrene, we observed that some of the slurries coagulated with barium chloride tended to cream, i.e., float to the top. To help determine whether the plateau effect that was being obtained for the highly compressible cakes was due to this phenomenon, we studied silica dispersions. Silica particles have a higher density (ρ_s = 2.2 g/ml) than polystyrene particles (ρ_s = 1.05 g/ml) and therefore settle to the bottom upon aggregation.

The volume versus thickness plots of the 0.24-μm silica slurries filtered at 5 psi in the presence of 0.001 M, 0.005 M, and 0.01 M [$BaCl_2$] are shown in Fig. 8.

The linear plot with [$BaCl_2$] = 0.001 M is as expected. The other two plots deviate greatly from the ones obtained for the corresponding polystyrene slurries, i.e., they show a linear rise

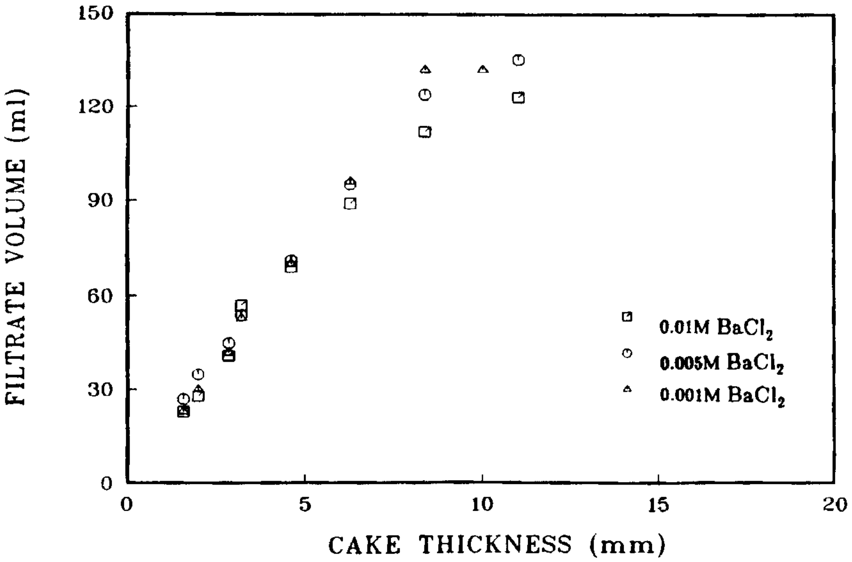

Figure 9. Filtrate volume versus cake thickness for 0.24-μm silica in the presence of three $BaCl_2$ concentrations (p=100 psi).

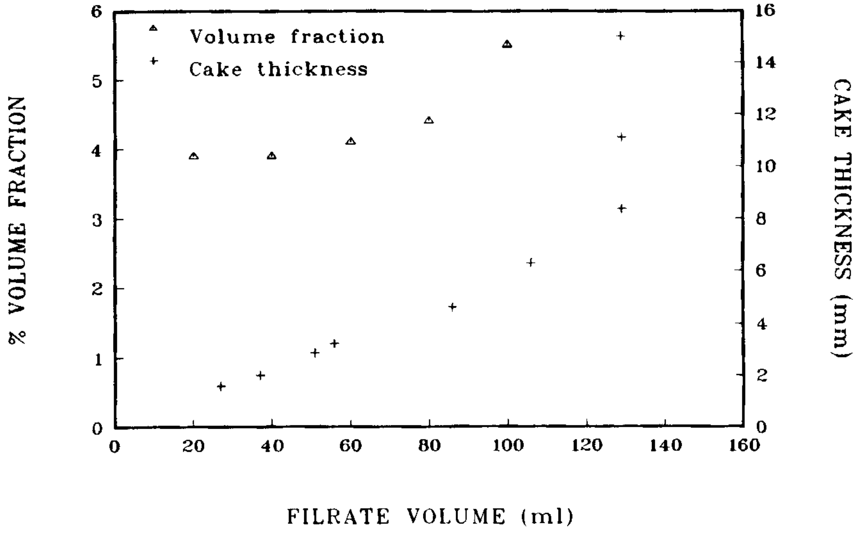

Figure 10. Slurry volume fraction (ϕ_s) and cake thickness (L) versus filtrate volume for polystyrene ($[BaCl_2]$=0.01M; p=5 psi).

with an upward turn toward the final measured cake thickness. Although these plots differ from those for the polystyrene, they are not unexpected and can be explained in terms of sedimentation. In the beginning of the filtration the silica particles settle out of the slurry, showing a large increase in cake thickness with small changes in filtrate volume. Later in the filtration, since most of the particles have already settled out, the plot shows relatively small increases in cake thickness over large filtrate volume. Volume versus thickness plots at 100 psi (Fig. 9) show the familiar linear rise to a small plateau. However, they are totally unexpected when compared to the plots obtained at 5 psi.

In one filtration, the slurry was sampled at various intervals. The results (Fig. 10) show slurry volume fraction ϕ_s and cake thickness plotted versus filtrate volume. As can be seen, ϕ_s rises from 0.039 to 0.055 during the course of the filtration. Perhaps the shape of the V versus L plots reflects the sedimentation or flotation behavior of the slurries. The disappearance of the break point in V versus L plots at higher pressures is therefore due to the shorter time span of the filtration, which allows less sedimentation to occur. Noteworthy is the fact that if sedimentation can play such an important role in slurries whose ρ is only 1.05, are there any truly pure filtrations? In answer to this question we intend to extend these studies to more concentrated slurries where sedimentation or flotation processes will be hindered.

NOMENCLATURE

d	Particle diameter (m)
k	Constant in Carman-Kozeny equation (-)
L	Cake thickness (m)
p	Applied filtration pressure (Pa)
p_L	Hydraulic pressure (Pa)
q	Rate of flow of filtrate (m/s)
R_m	Medium resistance (m^{-1})
s	Mass fraction of solids in slurry (-)
s_c	Mass fraction of solids in cake (-)
S_o	Area of particles per unit volume (m^{-1})
t	Time (s)

v	Volume of filtrate per unit area (m^3/m^2)
V	Volume of filtrate (m^3)
w_c	Mass of cake per unit area (kg/m^2)
x	Distance from the medium (m)

GREEK LETTERS

α	Local specific flow resistance (m^{-2})
α_{av}	Average flow resistance (m^{-2})
ε	Porosity (-)
ε_{av}	Average porosity (-)
ε_s	Volume fraction of solids, (solidosity) (-)
ε_{sav}	Average volume fraction of solids (-)
μ	Liquid viscosity ($Nm^{-2}s$)
ω_c	Volume of cake per unit area (m^3/m^2)
ϕ_s	Volume fraction of solids in slurry (-)
ρ	Filtrate density (kg/m^3)
ρ_s	Solid density (kg/m^3)
σ	Ratio of solid to liquid densities (-)

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REFERENCES

1. Gale, R.S. and R.C. Baskerville, Chem. Ind. (London), 355 (1967).

2. Gale, R.S. and R.C. Baskerville, Water Pollut. Control, <u>67</u>, 233 (1968).

3. Osho, O., M. Chem. Eng. Report, University of Houston, Houston, Texas (1977).

4. Leu, W., "Cake Filtration," Ph.D. Dissertation, University of Houston, Houston, Texas (1981).

5. Gregory, J., Chem. Eng. Sci., 36, 1789 (1981).

6. de Moor, A.E.L. and J. Gregory, J. Proc. Water Filtration Symp. KXIV, Antwerp (1982).

7. Eisenlauer, J. and D. Horn, Z. Wasser Abwasser Forsch., 16, 9 (1983).

8. Eisenlauer. J. and D. Horn, in Solid-Liquid Separation, Gregory J., ed., Ellis Horwood Ltd., Chichester, England (1984).

9. Harvey, M.A., K. Bridger, and F.M. Tiller, "Apparatus and Techniques for Studies of Incompressible and Moderately Compressible Cake Filtration," to be published in Filtration and Separation, January (1988).

10. Dezelic, N., J.J. Peters, and G.J. Dezelic, Macromol. Synth., 6, 85 (1977).

11. Chung-li, Y., J.W. Goodwin, R.H. Ottewill, Prog. Colloid Polym. Sci., 60, 163 (1976).

12. Tiller, F.M., "Mechanisms of Cake Filtration," DOE Report DE-AS05-81ER-10946 (1984).

13. Stober, W., A. Fink, and E. Bohn, J. Colloid Interface Sci., 26, 62 (1968).

14. Tiller, F.M., J.R. Crump and F. Ville, in Fine Particles Processing, Vol. 2, Somasundaran P., ed., Am. Inst. Min. Metall., Pet. Eng. Inc., New York, N.Y. (1980).

15. Bridger, K., M. Tadros, W. Leu, F.M. Tiller, Sep. Sci. Technol., 18, 1417 (1983).

16. Depoy, L., K. Bridger, M. Tadros, F.M. Tiller, and W. Leu, in Particulate and Multiphase Processes, T. Ariman and T.N. Veziroglu, eds., Hemisphere, N.Y. (1987) p. 11.

17. Tiller, F.M., C.S. Yeh, and W. Leu, Sep. Sci. Technol., 22, 1037 (1987).