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Filtration Behavior of Suspensions of Uniform Polystyrene Particles in Aqueous Media

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ABSTRACT

Cake filtration experiments of suspensions of polystyrene particles, of uniform morphology, through Nuclepore membranes having uniform pores were carried out. The effects of particle properties (size and surface charge), suspension properties (particle concentration and ionic strength), and applied pressure were determined. The results were analyzed in terms of the conventional Darcy-Ruth filtration equation. Plots of resistance versus weight of solids in the cake revealed two distinct regions with a transition occurring early on in the filtration process at a cake thickness of the order of 1 mm. The initial portion has a lower average slope (specific resistance) than that of the second region. It is by only plotting this second region (i.e. ignoring the initial stages of the filtration process) that apparent negative values for the medium resistance are obtained.

The specific cake resistance obtained from the slope of the second region, which spans at least 90% of the filtration time, was correlated with particle and dispersion properties. The specific filtration resistance was essentially independent of slurry concentration and of the total applied pressure. Specific cake resistance measured at constant pressure and slurry concentration showed an inverse dependence on ionic strength.

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This effect was more pronounced near the critical coagulation concentration and the filter cakes produced were more porous than those at the lower ionic strengths. These results will be discussed on the basis of variations in both the medium as well as the cake resistances during the course of filtration.

I. INTRODUCTION

Solid-liquid separations (SLS) are among the essential operations in the process industries. Two of the major challenges facing industry in this field involve increasing production rates and decreasing liquid content of the final cake. In addition to the traditional areas in the chemical and allied fields where SLS have been of long standing importance, problems of energy conservation, processing of solid fuels, coal slurry transport, environmental problems, and mineral production have been receiving attention.(1)

The area of SLS comprises four subfields including pretreatment, thickening (centrifugal or gravitation), particulate separation (filtration or sedimentation) and post treatment. This article is addressed toward particulate separation, in particular to cake filtration. Separation by sedimentation, filtration or centrifugation is dependent on the nature of the particles in the feed slurry. The size, shape, and interfacial properties along with hydrodynamical considerations determine the structure of the porous bed (cake or sediment), produced in the separation process. The porosity and permeability (flow resistance) of the porous beds are fundamental macroscopic properties which enter both theoretical and practical studies. Correlation of the macroscopic parameters used in continuum equations with particulate properties is of fundamental significance to SLS. Basic progress in the future depends upon experimentation based on the use of well characterized systems. Unfortunately, there is a paucity of published data in which the particulate properties have been carefully determined and combined with a complete and

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accurate analysis of the sedimentation and filtration behavior of the slurries. Grace provided perhaps the only comprehensive study in which particulate properties were combined with SLS data.(2) Other workers have produced useful data on clays aggregated by polymers(3-5) and polystyrene and silica(6,7) but the filtration information was limited, e.g., only for dilute slurries or the effect of pressure was not reported. The object of this paper is to extend the range of the available data.

The work reported herein represents the initial findings from a continuing program to study the synthesis, characterization and filtration behavior of monodisperse polystyrene latex dispersions over the size range 0.2-50 μm. Filtration rate experiments and pressure profile measurements within the cake will be carried out. In this study, the effects of ionic strength, slurry concentration, and pressure have been examined. The data show the initial stages of filtrations as well as those when a significant cake has developed. Results have been obtained for both stable dispersions and those which were aggregated due to the electrolyte present.

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II. EXPERIMENTAL

A. MATERIALS

The polystyrene latex dispersion was prepared by the technique of seeded emulsion polymerization,(8) using styrene monomer (Dow Chemical), potassium persulfate (Baker) and Aerosol MA (sodium dihexyl sulfosuccinate -- American Cyanamid). The resulting dispersion contained near monosized particles having median diameter 0.93 μm as measured by electron microscopy and dynamic light scattering. A transmission electron micrograph of the particles is shown in Fig. 1. The solids concentration, determined by evaporation, was 7.8% w/w. A more concentrated sample was prepared from the original using a rotary evaporator. The latex was extensively dialyzed before use.

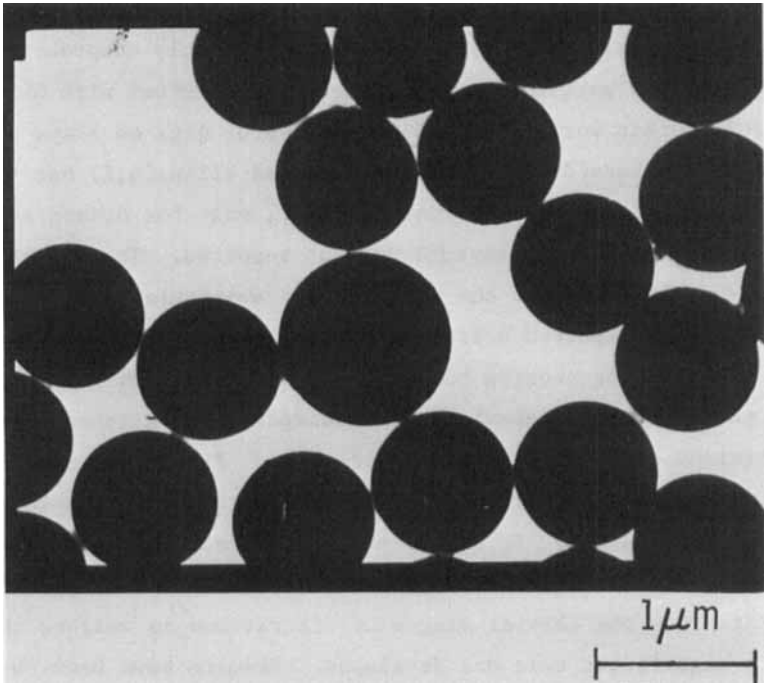

FIGURE 1. Transmission electron micrograph of the 0.93 μm polystyrene latex.

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Water was obtained from a deionizing system (Continental Water Systems, Inc.). The system includes a pretreatment for organic and particulate removal. All chemicals were analytical grade.

B. METHODS

1. Microelectrophoresis

Latex samples were diluted to 0.1% solids concentration in 0.01-0.5M potassium nitrate solutions. Electrophoretic mobilities were obtained at 25 ± 0.2°C using the cylindrical cell of a microelectrophoresis apparatus (Rank Bros.) from the time meas-

ured for a particle to travel one graticule width of 27 μm. Times were generally less than 30 s. The electrophoretic mobility was calculated from the average time for 10 measurements at the upper stationary level. Similar times were obtained at the lower level. The polarity of the cell was reversed between successive timings. Zeta potentials were calculated from the mobility data using the theory of Wiersema et. al.(9) as tabulated by Ottewill and Shaw.(10)

2. Filtration

Filtration data were accumulated using the apparatus shown in Fig. 2. A nitrogen cylinder supplies compressed gas to the system. A mercury manometer was used to calibrate the pressure gauge over the range 0-100 kPa. Above 100 kPa the gauge (Ashcroft) was used alone. It was, however, periodically checked "back-to-back" with another gauge; the discrepancy, if any, never exceeded the manufacturers specification (± 3%). After the stop cock is opened and filtration begins, the ballast volume assures an essentially constant pressure. Slurries were placed

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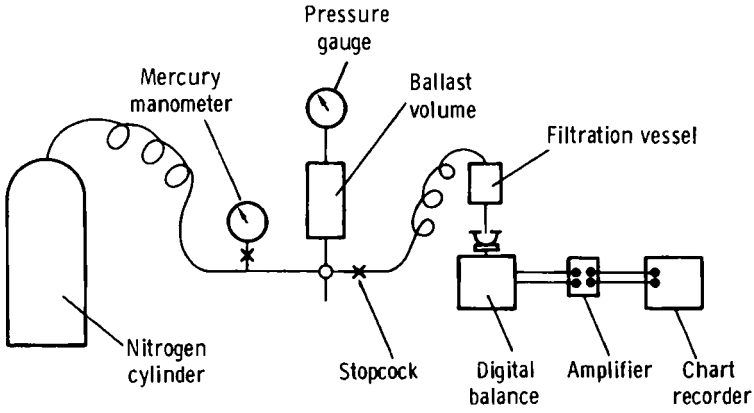

FIGURE 2. Apparatus for recording constant pressure filtration rates.

in the stainless steel vessel (Millipore Ltd.) and filtered through a polycarbonate membrane (Nuclepore) having regular straight through 0.2-μm pores. Filtrates were collected on a digital balance whose output was connected via an amplifier to a chart recorder. Plots of filtrate mass versus time were thus obtained in each experiment.

Each sample was prepared for filtration as follows. Distilled water and a measured aliquot of 1M potassium nitrate solution were mixed together in a beaker and the desired amount of latex concentrate was added. The sample was made up to the required volume with more distilled water and allowed to stand for 30 minutes. It was then poured into the assembled apparatus and the run began. At the end of each run, the wet cake was weighed and then dried to constant weight in a 50°C oven, in order to determine the mass fraction of dry solids, s_c.

Constant pressure filtrations were performed over the pressure range 1-100 psi (7-690 kPa) at ionic strengths $0.01 \leq [KNO_3] \leq 0.50$ M. Slurry concentrations varied from 4-20%. Most filtrations utilized 100 cm^3 samples but 20, 200, and 300 cm^3 volumes of slurry were also tested. Each filtration run was duplicated.

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III. DATA TREATMENT AND CALCULATIONS

Filtration data were analyzed using the traditional flow equations in resistance form (total resistance = cake + medium resistance)(11):

$$p/\mu q = \alpha_{av} w_c + R_m \qquad (1)$$

Eqn. (1) is valid [restriction discussed by Tiller and Crump(11)] for variable p, α_{av} and R_m. It provides an instantaneous view of the cake. If the pressure, α_{av} and R_m are assumed constant, integration leads to:

$$pt/\mu v = \frac{\alpha_{av}}{2} w_c + R_m \qquad (2)$$

where

p is the applied pressure (Pa)

t is time (s)

v is the volume of filtrate per unit cross sectional area of cake (m^3/m^2)

$q = dv/dt$ (m/s)

μ is the viscosity of the filtrate (Ns/m^2)

w_c is the mass of solid cake per unit area (kg/m^2)

α_{av} is the average specific cake resistance (m/kg)

R_m is the medium resistance (1/m).

Equation (2) is inadequate whenever the medium resistance R_m has a value comparable to the cake resistance $R_c = \alpha_{av} w_c$.

Values for w_c were calculated from filtrate volumes using the mass balance relationship:

$$w_c = \frac{\rho s}{1 - s/s_c} \cdot v \qquad (3)$$

where ρ is the filtrate density (kg/m^3), and s and s_c are the mass fractions of solid in the slurry and cake, respectively. Note that because s_c was only measured at the end of the filtration process it was assumed constant (valid only when Δp_c is constant) throughout this particular experiment. For each value of w_c, q was calculated from $\Delta v/\Delta t$, using 1 cm^3 increments in the raw data. These calculations were performed by a Fortran program and the data were output to a plotter (Tektronix). No smoothing of the data was attempted.

IV. RESULTS AND DISCUSSION

Figure 3 shows typical plots of $p/\mu q$ and $pt/\mu v$ versus w_c, obtained for a constant pressure filtration. The curves

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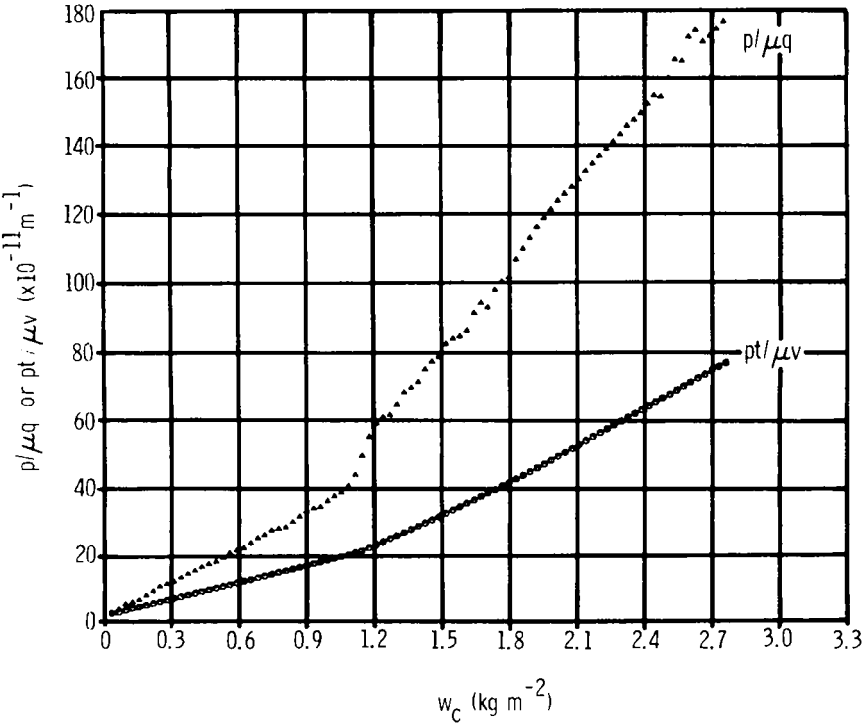

FIGURE 3. $p/\mu q$ and $p/\mu q_{av}$ versus w_c for constant pressure filtration (p = 5 psi, s = 3.9%, and I = 0.01 M).

comprise two distinct regions with a transition occurring at $w_c \approx 1$ kg/m^2. Both curves share a common intercept at $w_c = 0$ as required by equations (1) and (2). Figures 4 and 5 show that when different volumes of slurry are filtered under similar conditions the graphs are superimposable, e.g., filtration of 20 cm^3 of slurry gave the initial portion with no break-point, whereas when 300 cm^3 slurry are filtered the second linear region was extended with no curvature. The initial part of the curves, up to w_c = 1.0 kg/m^2, show slight curvature indicating increasing α_{av}. The average slope of the $p/\mu q$ curve is, however, approximately twice that of the $pt/\mu v$ plot over this range and so the values of R_m and α_{av} are not changing significantly.

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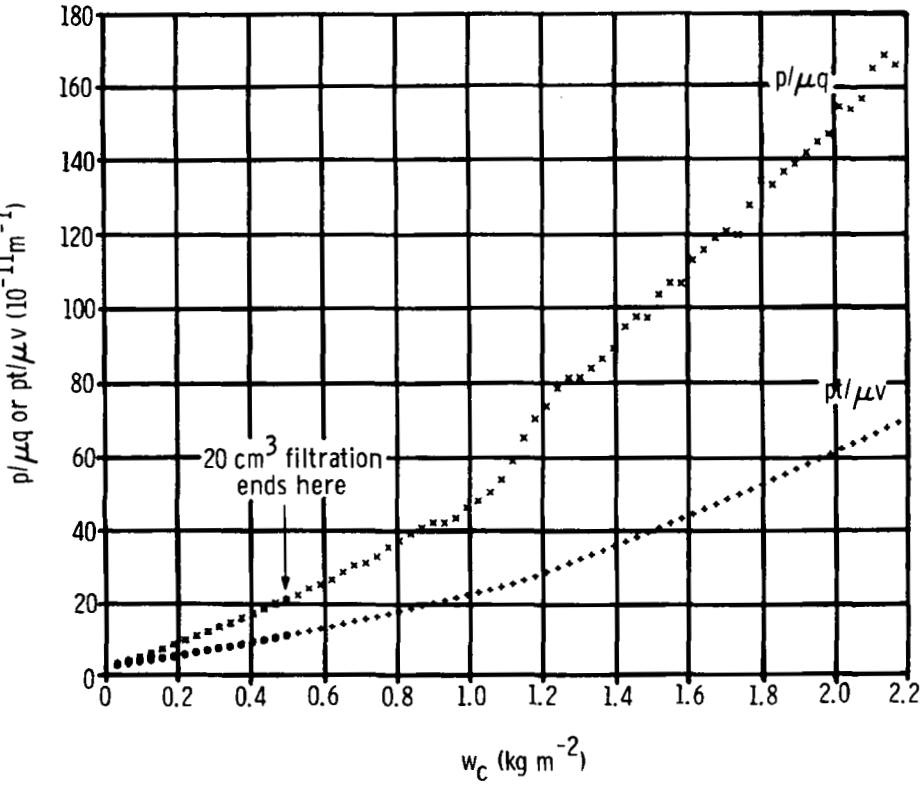

FIGURE 4. Superimposed filtration data for different volumes of slurry, 20 cm^3 and 100 cm^3 (p = 2 psi, s = 3.9%, and I = 0.01 M).

In the region of w_c = 1 kg/m^2 (cake thickness L, 1.5 mm) there is a rapid change of slope. It is postulated that the cake undergoes a transformation at this point leading to a more compact structure with higher resistance. Due to this change in α_{av}, and possibly R_m, the assumptions implicit in equation (2) no longer hold. After the transition the plot of $p/\mu q$ versus w_c yields an apparent negative medium resistance. This apparently absurd extrapolation is thought to be due to medium blinding.(1)

Further investigation into the nature of this transition was conducted by recording scanning electron micrographs of the

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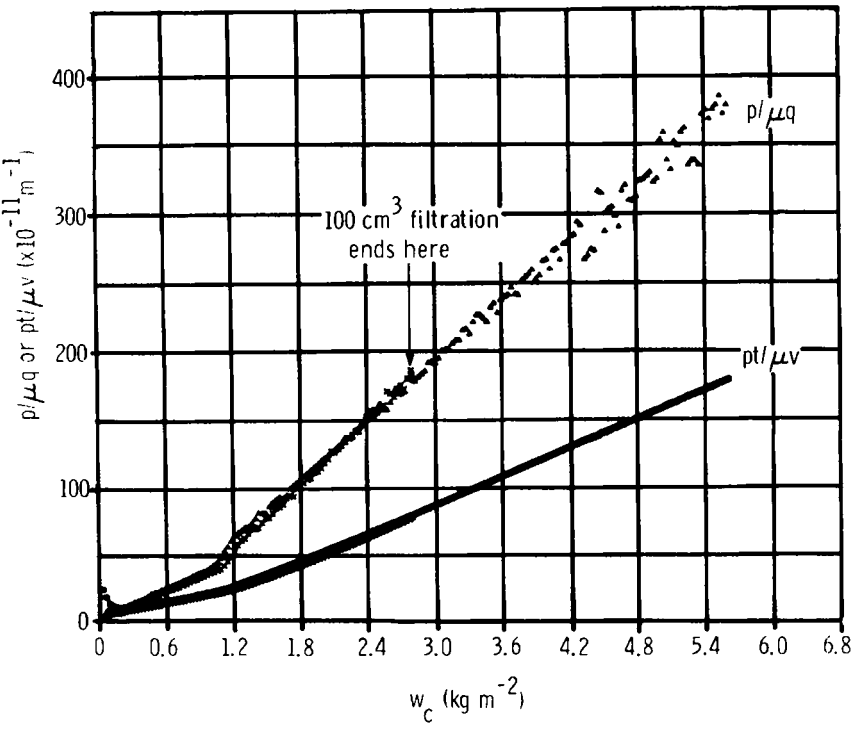

FIGURE 5. Superimposed filtration data for different volumes of slurry, 100 cm^3 and 300 cm^3 (p = 10 psi, s = 3.9%, and I = 0.01 M).

dried cakes for filtrations terminated before and after the transition region. A series of these, which were reproducible, are shown in Fig. 6. The top of the cake shows closely packed spheres in both cases; however, the bottoms of the cakes appear different before and after the transition. When 100 cm^3 of the slurry was filtered the bottom of the dried cake shows well packed spheres. When only 20 cm^3 of the slurry was filtered, the bottom of the cake appears highly porous. In fact, the value obtained for the average porosity of the former cake was (ε_{av} = 0.46) whereas the value for the latter cake was (ε_{av} = 0.37). These observations suggest that the cake

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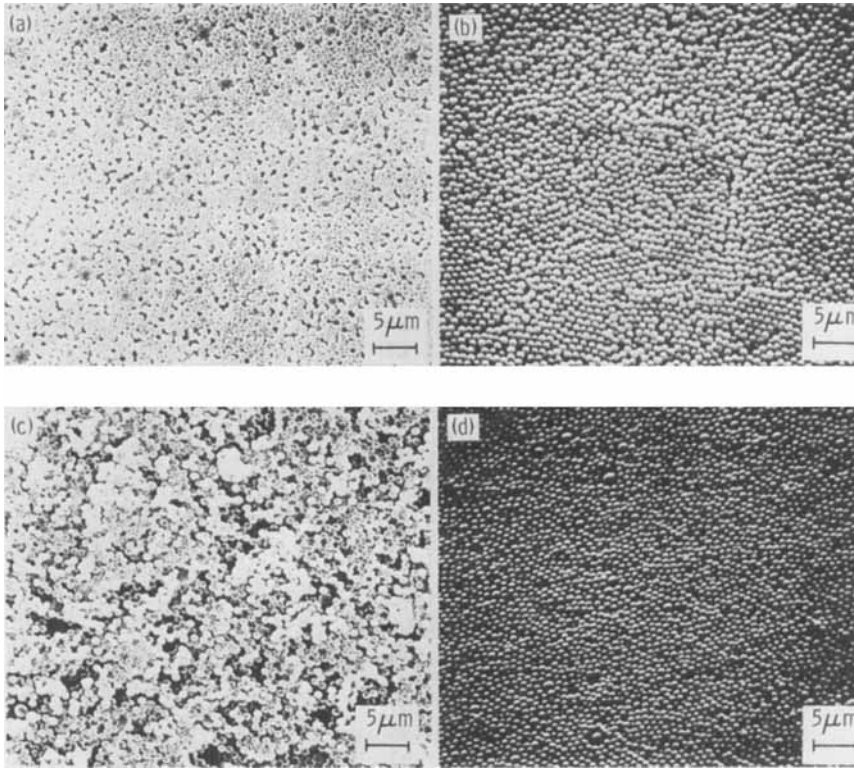

FIGURE 6. Scanning electron micrographs of cakes formed before and after the transition region: a) top before transition; b) top after transition; c) bottom before transition; and d) bottom after transition (p = 2 psi, s = 3.9%, and I = 0.01 M).

initially forms as a dendritic growth which leads to a very porous structure at the cake-medium interface, and this later collapses at some critical point. Table 1 lists the $p/\mu q$ and w_c values at which the transition occurs. Note that the values are independent of the applied pressure, which would not be the case if the transition point was dependent upon the solid compressive pressure, p_s.

The second linear region beginning at w_c values of ~ 1 kg/m^2 could be extrapolated to greater cake thicknesses (cf Fig. 5).

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TABLE 1

Values of $p/\mu q$ and w_c at Transition Points

Ionic Strength (M)	Filtration Pressure (psi)	Transition Points w_c (1.05 kg/m^2)	Transition Points $p/\mu q \pm .2(*10^{-12}$ m$^{-1})$
0.01	1	1.07	4.4
	2	1.05	5.1
	5	1.11	4.7
	10	1.14	4.5
	20	1.20	3.8
	50	1.24	4.2
	100	1.02	4.1
0.10	1	1.05	2.1
	2	1.12	3.9
	5	1.10	3.7
	10	1.17	3.7
	20	1.13	3.2
	50	1.23	3.1
	100	1.09	3.8
0.50	1	-	-
	2	.87	1.2
	5	.99	1.4
	10	.97	1.8
	20	1.00	1.9
	50	1.03	1.8
	100	.69	1.8

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In most cases this region will cover by far the greater time period of the filtration, and it is this region which is usually recorded in filtration experiments. In the following discussion, therefore, the effects of dispersion properties upon filtration behavior will be judged according to the slope of this line, which will be designated as the specific cake resistance. Note that in this analysis, the value of $p/\mu q$ at the transition point may be considered an apparent medium resistance, which is a composite value comprising the initial medium resistance, the

resistance of the cake formed before the transition, and any medium blinding occurring in the initial stages of cake build-up.

A fundamental property of colloidal dispersions is the manner in which the particles interact with each other, and this is of great importance in cake formation. In the case of charge stabilised dispersions, such as polystyrene latexes, these interactions may be varied by adjusting the ξ-potential. This is achieved by addition of an electrolyte which may either specifically adsorb on the particles or merely raise the ionic strength of the continuous phase. Figure 7 shows the variation of specific cake resistance with ionic strength (potassium nitrate concentration) for a series of different pressures. For all the pressures studied α decreases with increasing ionic strength. Figure 8 shows some more detailed data for p = 10 psi (69 kPa).

The errors depicted here ($\pm$ 13%) are much higher than would be expected from the good overlap seen in Figs. 4 and 5. They result from the time interval separating the various experiments. Runs performed on the same day reproduced each other almost exactly, whereas runs separated by a period of one month did not. In general, the specific filtration resistance appeared to increase with age of the dispersion. It is suspected that this ageing effect is due to the increasing quantities of smaller particles and a TEM of the dispersion taken 10 months after its preparation showed some broadening of the size distribution. Because this effect was not expected, it has not been studied systematically and has been included as an error.

Also plotted on Figure 8 are the measured ξ potentials for the latex under those conditions of ionic strength. At most of the ionic strengths studied, the latex showed little sign of coagulation after standing for one hour and the porosities of the cakes did not vary (ϵ_{av} = 0.37). However, after standing for one hour in the presence of 0.5M KNO_3, there was a considerable degree of coagulation, and the cakes formed were more porous (ϵ_{av} =

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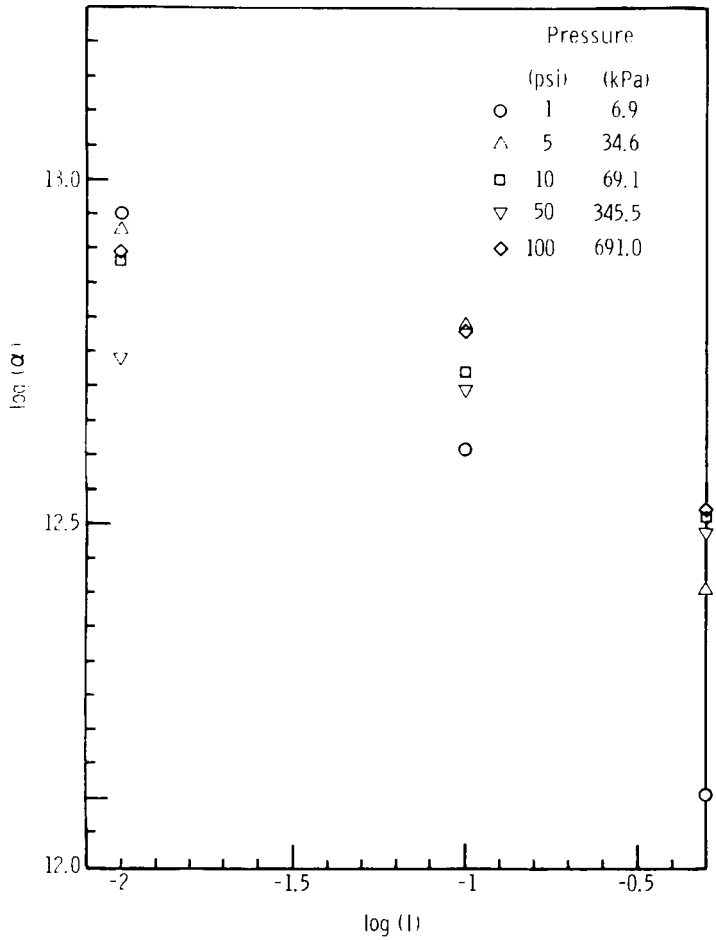

FIGURE 7. Effect of ionic strength I upon specific cake resistance α, for pressures 1, 5, 10, 50, and 100 psi.

0.43). The specific cake resistance decreases as ξ becomes less negative; and Figure 9 shows a plot of log α versus ξ. Linear regression analysis on these data gives a correlation coefficient of -0.82 and a slope of -5×10^{-3} (mV^{-1}). It is evident that the variation of α with ξ is too small compared to the experimental errors to be able to pin down the exact nature of this relation-

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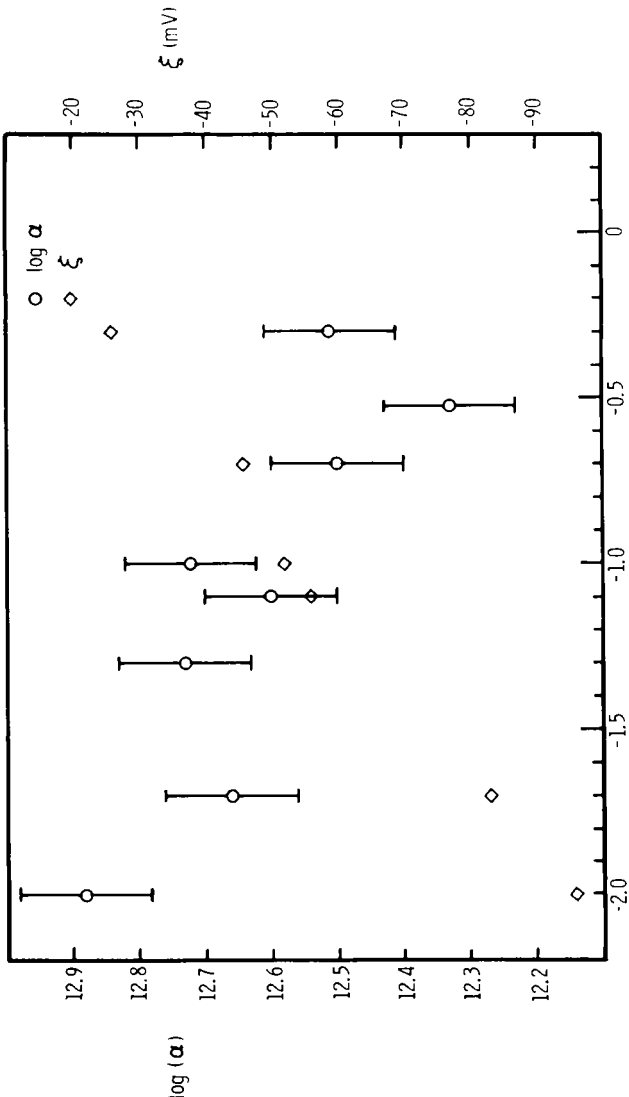

FIGURE 8. Effect of ionic strength I upon specific cake resistance α and ξ-potential (resistances are for a pressure of 10 psi).

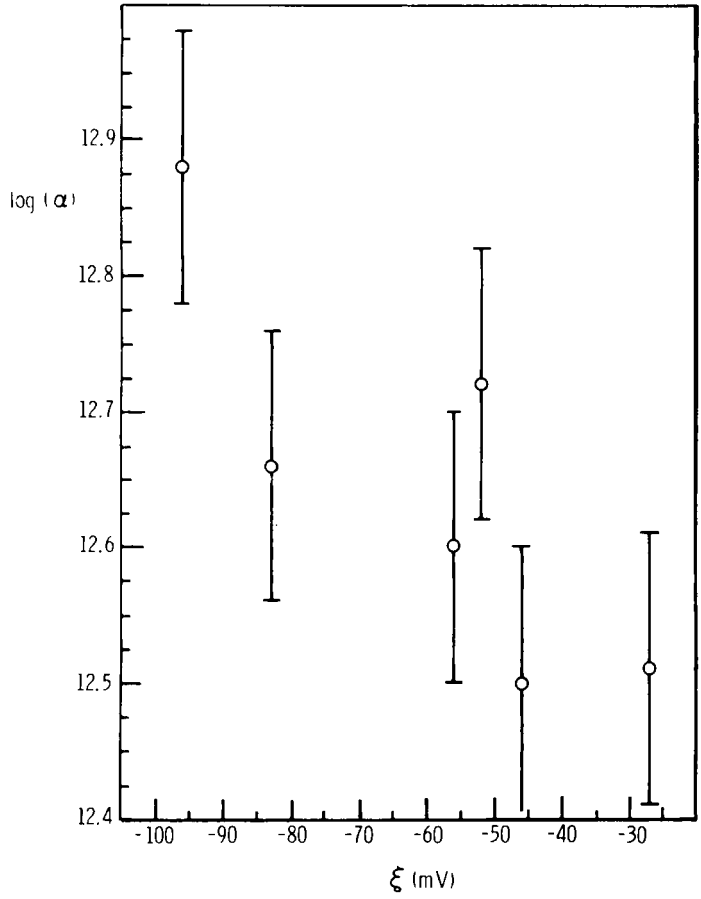

FIGURE 9. Log α versus ξ (α values for p = 10 psi).

ship at this time. The effects are more pronounced, however, when comparing the compressibilities of cakes formed from stable (high ξ) and unstable (low ξ) dispersions.

Figure 10 shows plots of α versus p for these ionic strengths on a log-log scale. Above 10 psi (69 kPa) the variation of α with p appears small for each ionic strength, i.e., the cakes are incompressible. However, between 1 and 10 psi (7-69 kPa) the cakes formed from slurries having ionic strength I = 0.5 M

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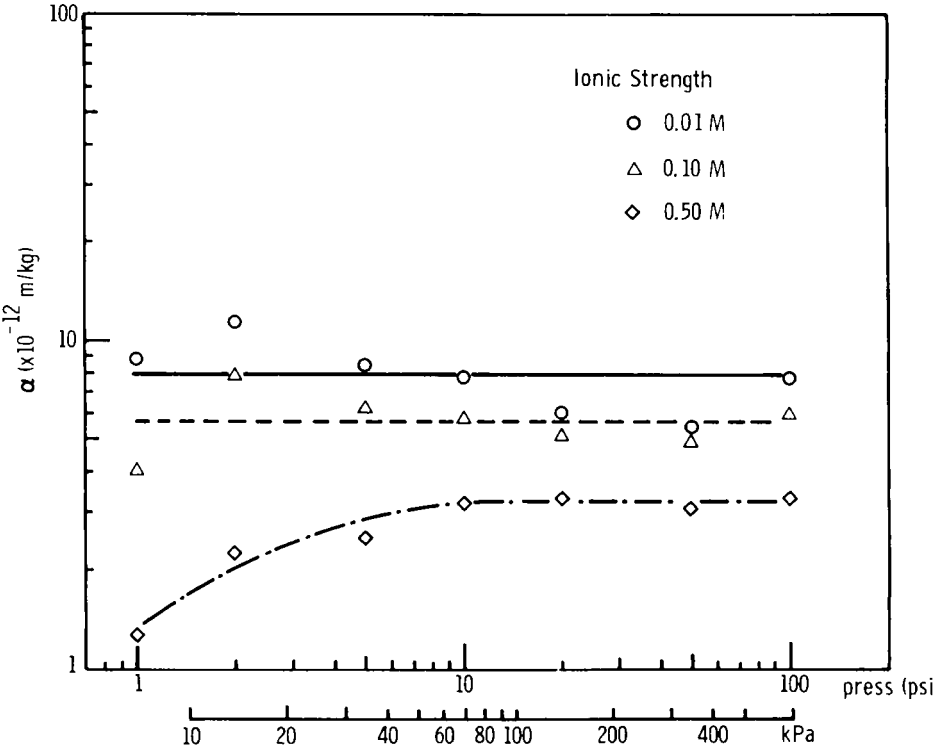

FIGURE 10. Effect of pressure on specific filtration resistance for ionic strengths of 0.01 M, 0.10 M, and 0.50 M.

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show a consistent increase in α with p. It can be seen that the increase in cake permeability gained from filtering an aggregated slurry as opposed to the unaggregated slurry is greater at low filtration pressures. The incompressibility of the cakes at pressures above 10 psi (69 kPa) was confirmed by filtration experiments during which the applied pressure was varied from 10-50 psi (69-345 kPa). The filtration was begun at a pressure of 10 psi and this was raised, stepwise, by 2 psi for every 5 g of filtrate. Figure 11 shows the resistance plot obtained for ionic strength 0.01 M; the other ionic strengths gave similar plots. The plot of pt/μv shows the steps corresponding to

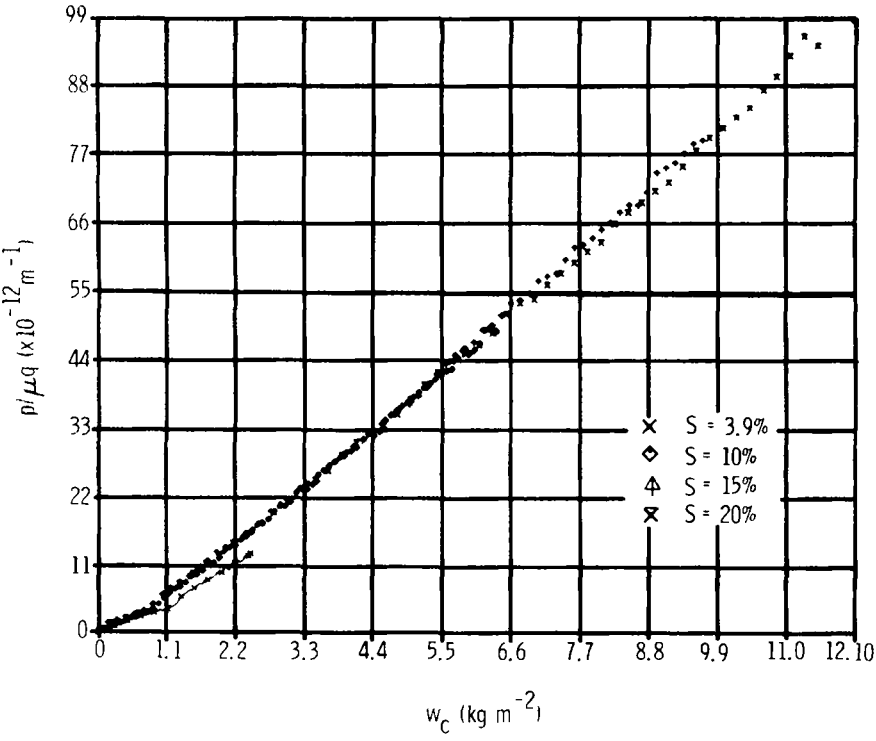

FIGURE 11. $p/\mu q$ and $p/\mu q_{av}$ for variable pressure filtration ($10 \leq p \leq 50$ psi, s = 3.9%, I = 0.01 M).

the increases in pressure. The $p/\mu q$ versus w_c graph consists of two straight lines. If the cake were compressible (i.e., α increases with p) then the graph would show a curve convex to the w_c axis. The specific cake resistances measured from the variable pressure experiments were similar to, but lower than, the average values obtained from the constant pressure filtrations. The values are as follows:

Ionic Strength (M):	0.01	0.10	0.50
α from variable pressure ($\times 10^{-12}$ m/kg):	6.5	4.6	3.0
Average α from const. pressure ($\times 10^{-12}$ m/kg):	8.0	6.7	3.3

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The nature of these data has required a re-evaluation of the basic constitutive equations(12-14) used to relate porosity ε and specific flow resistance α to the effective pressure p_s. Combined with Darcy's law and material balance, the constitutive relations provide formulas relating pressure volume and time. For cakes consisting of hard particles which do not change shape, the initial open structure laid down under a null stress undergoes compression until a minimum porosity is reached under increasing stress. While ε decreases from some value ε_o to a minimum ε_∞, the flow resistance increases from α_o to the maximum α_∞. Normally a wide range of pressure (100 atmospheres) is covered in approaching minimum values of ε and maximum values of α. Data taken with particles used in this investigation indicate that a much lower pressure (possibly 1 atmosphere) appears to yield ε_o and α_∞. Inasmuch as the power functions of p_s commonly in use do not yield limiting values of ε and α as required theoretically, new expressions must be obtained to represent the data of this investigation.

A final effect worth mentioning is that of the slurry concentration. The experiments described above were all performed on slurries having a 4% solids concentration. Figure 12 shows that, at least for I = 0.01 M and p = 10 psi (69 kPa), a unique resistance plot is obtained for slurry concentrations from 4-20%. A more detailed study of the filtration behavior of polystyrene latex at higher slurry concentrations will be the subject of a forthcoming publication.

V. SUMMARY

- Resistance plots for filtration of 1-μm polystyrene latex particles show two regions with a transition at $w_c \sim 1$ kg/m^2

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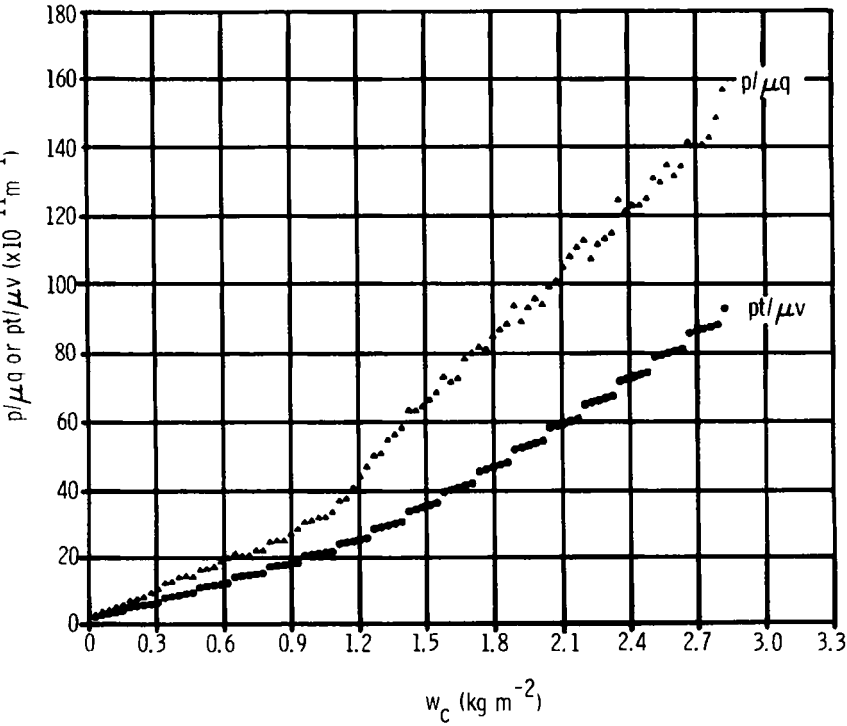

FIGURE 12. $p/\mu q$ versus w_c for constant pressure filtration of slurry concentrations: 3.9%, 10%, 15%, and 20% (p = 10 psi, I = 0.01 M).

- The cake structure before the transition is more porous than that after the transition -- especially at the cake/medium interface

- The specific cake resistance decreased as the absolute value of zeta potential decreased

- Under electrolyte conditions where the latex was stable, incompressible cakes were formed ($1 \leq p \leq 100$ psi)

- At high ionic strength when the latex was unstable, the

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cakes appeared compressible over the pressure range 1 - 10 psi

- Specific cake resistance was independent of the solid concentration in the latex up to a value of 20%

NOMENCLATURE

I	Ionic strength (mole/L^3)
p	Applied filtration pressure (F/L^2)
Ps	Solid compressive pressure at distance x from medium (F/L^2)
Δp_c	Pressure drop across the cake (F/L^2)
q	Rate of flow of liquid (L/T)
q_{av}	Average rate of flow of liquid as defined by v/t (L/T)
R_m	Medium resistance (1/L)
s	Mass fraction of solids in slurry (-)
s_c	Average mass fraction of solids in cake (-)
t	Time (T)
v	Filtrate volume per unit filtration area (L^3/L^2)
w_c	Mass of dry solids per unit area (M/L^2)
α	Local specific filtration resistance (L/M)
α_{av}	Average specific filtration resistance (L/M)
α_o	Initial specific filtration resistance at $p_s = 0$ (L/M)
α_∞	Limiting specific filtration resistance
ε	Local porosity of filter cake (-)
ε_{av}	Average porosity (-)
ε_o	Initial local porosity at $p_s = 0$ (-)
ε_∞	Limiting local porosity (-)
μ	Viscosity of liquid (M/LT)
ρ	Density of liquid (M/L^3)
ρ_s	True solid density (M/L^3)
ξ	Zeta potential (ML^2/AT^3) (V)

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VI. ACKNOWLEDGEMENTS

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