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A NEW EXPERIMENTAL TECHNIQUE FOR THE ANALYSIS OF CAKE FILTRATION

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ABSTRACT

An electrical conductivity technique for determining cake heights, and hence average concentrations, during stepped-up pressure filtrations is described. Experimental data are presented which compare the results from individual constant pressure filtrations and a filtration employing a sequence of corresponding pressures. The data are analysed in a variety of ways that show the accuracy of the time saving step-up technique when the solids concentration is measured in-situ.

INTRODUCTION

Filter scale-up parameters are usually obtained from a sequence of individual constant pressure filtration experiments. These time consuming tests give data relating parameters such as average cake resistance and cake concentration to the applied cake forming pressure. It is common, however, for the concentration data to be inconclusive, as obtaining a sample of filter cake for subsequent analysis without residual slurry (filtration stopped too early) or a portion of air blown cake (filtration stopped too late) is difficult. Compression permeability cells are not often used nowadays, as the lack of homogeneity within the cell is well known. Filtrations in which the cake forming pressure is stepped-up progressively, from the lowest to highest values, have been proposed; see, for example, Purchas and Wakeman, 1986. Obtaining the concentration with pressure relation by sampling has, however, previously been impossible during such tests and restricts the subsequent analysis to evaluating the relation between specific resistance and pressure only. This paper describes a step-up technique which enables the position of the filter cake surface as a function of time to be monitored. Average cake concentration can be calculated from a knowledge of the filtrate volume and initial slurry concentration and data acquired for both the variation of specific resistance and concentration with pressure from a single test without the need of physically sampling the filter cake.

The approach adopted is to measure the electrical conductivity using a series of electrode pairs positioned vertically within a filter cell. The electrodes are interfaced to either a computer to provide the concentration profile, or to a signal generator and bridge circuit to monitor the resistance at selected electrodes. When the cake reaches the electrode pair the cake height with time, and filtrate volume, are known. The approach used is similar to one in which pressure probes were used to monitor the cake height with time (Murase *et al.*, 1989), but the conductivity technique can be more easily adapted to the full determination of the concentration profile within the filter cake. The average filter cake concentration can, therefore, be obtained from the integrated local filter cake concentrations.

ANALYSIS TECHNIQUES

The well known reciprocal rate equation for constant pressure filtration is

$$\frac{dt}{dV} = \frac{\mu c \alpha_{av}}{A^2 \Delta P} V + \frac{\mu R_m}{A \Delta P} \quad (1)$$

where t is the time, V the filtrate volume, μ the liquid viscosity, A the filter area, ΔP the pressure, R_m the medium resistance, α_{av} the average specific cake resistance and c the mass of dry solids deposited per unit volume of filtrate given by

$$c = \frac{s\rho}{1-ms} \quad (2)$$

where s is the mass fraction of solids in the feed slurry, ρ the liquid density and m the moisture ratio of wet to dry cake. The moisture ratio is often assumed to be unity, i.e. the cake moisture is neglected, and a simplified version of equation (2) can then be used.

If the average cake concentration by volume fraction (C_{av}) and true solid density (ρ_s) are known then the dry mass of solids deposited per unit volume filtrate can be related through a constant of proportionality (w) to the cake height (L) by

$$L = \frac{c}{\rho_s A C_{av}} V = wV \quad (3)$$

Combining equation (3) with liquid and solid mass balances over the filter cake gives

$$c = \left(\frac{Aw + 1}{1 + (1 - C_i)/C_i} \right) \rho_s \quad (4)$$

where C_i is the feed slurry concentration by volume fraction. Average specific cake resistance can then be calculated from the gradient of a plot of reciprocal filtrate rate with filtrate volume, in accordance with equation (1).

During step-up filtration the gradient of the line between cake height and filtrate volume will change, in-step with the pressure change, hence the value of w to be used in equation (4) will need to be calculated at each pressure increment. The above demonstrates the importance of knowledge of the cake concentration, or dry mass per unit filtrate volume, to accurately calculate specific cake resistance. The cake concentration has not been available in most of the previously published analyses using stepped-up filtration pressure and have relied on the use of $m = 1$ in equation (2). This implicitly assumes an incompressible filter cake which is inconsistent with the aim of determining the variation of specific resistance with pressure.

The average values of specific resistance and concentration are scale-up parameters which can be related to cake forming pressure through the constitutive equations

$$C_{av} = C_0 (1 - \beta) \Delta P^\beta \quad (5)$$

and

$$\alpha_{av} = \alpha_0 (1 - n) \Delta P^n \quad (6)$$

respectively, where C_0 , α_0 , m and β are constants. It has been shown (Tiller, 1975) that if these constituent equations are known it is possible to predict the filter cake concentration profile from

$$C = C_0 \Delta P^\beta \left(\frac{y}{L} \right)^{\beta/(1-\beta-n)} \quad (7)$$

where y is the fractional cake depth and y/L the dimensionless distance into the cake. Cake depth can be deduced from

$$L = \frac{\Delta P^{(1-\beta-n)}}{\mu \alpha_0 C_0 \rho_s (1-\beta-n)} \frac{A}{Q} \quad (8)$$

where Q is the instantaneous filtrate rate. This approach is generally attributed to Tiller and Shirato.

EXPERIMENTAL

The experimental technique for in-situ concentration analysis using electrical conductivity has been described previously (Holdich and Sinclair, 1992). In this study the filter cell was 44 mm in diameter and constructed out of PVC with an inner cylinder of perspex. A small viewing window was cut in the PVC and pressures of up to 3 bar could be used. The experimental equipment is shown schematically in Figure 1.

Kaolin with a density of 2650 kg m^{-3} (ECC china clay, grade E) was used in all the tests. The size distribution of this material was measured by a Micromeritics Sedigraph and is given in Table 1.

All the slurries were made up to 16.3% by volume (34% by weight) in 0.31 g l^{-1} potassium chloride solution. The pH of the slurries was 5.04. The filter surface faced upwards, but significant sedimentation in the filter cell was not observed during the test. A new Gelman Scientific Versapor $0.2 \text{ }\mu\text{m}$ membrane was used as a filter medium in each test. This media was chosen in an attempt to limit media clogging and to provide a stable and uniform media.

Four individual constant pressure filtrations were conducted at 0.55, 1.0, 2.0 and 2.8 bar, and one filtration employed all four of these pressures 'stepping-up' during the experiment. In all the experiments the solid concentration with time at each electrode was monitored via the computer interface. The pressure was stepped-up during the test when the cake surface, assumed to be a concentration of 18% by volume, reached the 20, 40 and 60 mm electrode pairs positioned above the medium.

RESULTS

Figure 2 is an example of the output provided by the technique, where concentration is in terms of solids volume fraction (v.f.). The first check of reproducibility between the individual and step-up tests is to investigate the reciprocal filtrate rate. Equation (1) implies that reciprocal rate should be a function of filtrate volume collected, regardless of the test's history, and Figure 3 shows the relevant data obtained from the individual and step-up filtrations.

In Figure 3 slight deviation is noticeable when filtering at 2.0 bar, and considerable deviation is apparent at 2.8 bar between the individual and stepped-up data. The dry mass per unit volume of filtrate is calculated from equation (4) using the constant of proportionality between the cake height and filtrate volume (w). The assumption is again made that stepping-up the pressure does not alter this relation. The measured height-volume relation is shown in Figure 4. This relation is apparently identical for the two individual filtrations at 2.0 and 2.8 bar.

An alternative method to determine the dry weight of cake per filtrate volume is to use the knowledge of the cake concentration profile to provide an integrated value for the average cake concentration giving

$$c = \frac{A\rho_s}{V} \int C dx \quad (9)$$

This last approach is only applicable if the conductivity, and hence concentration, at each electrode pair is continually monitored during a test. If a single bridge circuit was used to ascertain the cake position at a known time, and filtrate volume, then this option would not be available. The times chosen to investigate the step-up filtration data were: 867, 2710, 5090 and 6710 s. These times correspond to cake heights at electrodes 20, 40, 60 and 70 mm from the filter medium and the corresponding cake forming pressures were 0.55, 1.0, 2.0 and 2.8 bar. The cake concentration profiles at these times and pressures are shown in Figure 5.

The values for dry weight solids deposited per unit volume of filtrate using the various approaches are given in Table 2. Equation (4) was used for the individual and step-up data, integration by equation (9) was used for the second column of step-up data. Finally, equation (2) with $m = 1$ was also used for comparison which neglects any moisture retained in the filter cake and does not, therefore, show any effect due to cake compressibility.

Average specific cake resistances for the individual and the separate parts of the step-up filtrations, were calculated from the gradients shown on Figure 3. Four different approaches were used to calculate dry weight of cake per unit volume of filtrate, hence four different values of alpha are also found, depending on which approach has been followed. The results are shown in Table 3.

The average cake concentrations were calculated by mass balance using the filtrate volume and initial concentration for all the filtrations. Thus, both the variation of concentration and specific resistance with pressure have been determined, the results are presented in Figures 6 and 7.

The regressions employed to find the best fit between the specific resistance and pressure, and the concentration and pressure ignored the individual test data at 2.8 bar as the resistance and cake concentration were apparently greater at 2.0 bar than at 2.8 bar. Such a result would seem unreasonable and the effect is attributed to medium clogging during the higher pressure filtration, leading to significant medium resistance, despite the use of the membrane. A significant intercept is shown in Figure 3 for the 2.8 bar individual test filtration. It can be seen from equation (1) that the medium resistance is proportional to the product of this intercept and filtration pressure. Thus, it is concluded that a significant resistance due to the medium occurred during the 2.8 bar individual filtration test, leading to an apparently lower cake specific resistance than during the 2.0 bar filtration and explaining the apparently similar cake height to filtrate volume ratio given in Figure 4. Clogging was not an apparent problem during the step-up filtration, possibly because the filtration started at low pressures and gradually built up to the higher values; a layer of cake protected the media by the time high pressures were employed.

Values for the constants used in equations (5) and (6) can be obtained from Figures 6 and 7. These values depend on the approach adopted for the determination of the dry weight of cake mass per unit filtrate volume.

Table 4 provides a range of data. Assuming the individual test data to be the most representative of this material the compressibility coefficient (n) was 0.55. Step-up filtration employing equation (4) for data analysis appears to be the best method to reproduce this value. The coefficient and constant in the concentration power law expression is, however, more accurately represented by step-up filtration using equation (9). The significance, or otherwise, of the variation in the values shown in Table 4 can be assessed by modelling the cake concentration profile.

The profiles can be calculated using equations (7) and (8), i.e. following the approach adopted by Tiller and Shirato. Again the calculations depend on the approach used to provide the values

given in Table 4. The predicted profiles are shown in Figure 8 together with the measured profile obtained during step-up filtration at 5090 s, i.e. during the 2.0 bar filtration.

All the different approaches provide a reasonable estimate of the cake height (about 60 mm), but show a poor representation of the concentration profile. The approach that gives the closest to the measured concentration at the base of the filter cell is the one using equation (9), the technique based on integration of the measured profile. The agreement should not, therefore, be surprising. Even this approach cannot accurately model the full profile, however. The disagreement must, therefore, be due to the oversimplifications required in deriving equations (7) and (8): neglect of solids weight and variation of the liquid flow within the cake. It is noticeable that the profiles obtained using the individual filtration and the integrated cake concentration data are very similar. The time saving step-up technique is supported by this result.

CONCLUSIONS

The technique based on electrical conductivity to measure the time at which the cake surface reaches a fixed, and known, point provides a means of investigating cake properties. This is especially useful during filtrations employing stepped-up pressures. Average cake concentration can be calculated if the initial slurry concentration and filtrate volume are known. The technique can be extended to provide in-situ monitoring of solid concentration at the position of the electrodes, thus providing the concentration profile of the cake. Average cake concentration can be deduced by integrating the local concentrations with respect to distance up the filter cell.

Pressure can be stepped-up during filtration after the cake height has been measured. Thus, an incremental scheme for the calculation of cake concentration, average specific cake resistance (from the filtrate rate data) and their variation with incremented cake forming pressure can be deduced from a single experimental test. Integration of the local cake concentration for average cake concentration appears to be superior to the approach employing a mass balance using a knowledge of the cake height, filtrate volume and initial slurry concentration.

Neglecting cake moisture content when calculating the dry mass of cake per unit volume of filtrate led to an error of approximately 33% for the kaolin tested. It also precludes the calculation of the constant and exponent in the power law function between concentration and pressure. The exponent in the similar function between specific resistance and pressure appeared to be unaffected by the neglect of cake moisture. The modelling approach of Tiller and Shirato to provide the cake concentration profile is, however, invalid if the pressure dependent variation in cake moisture is ignored. It is, therefore, essential to have an in-situ experimental means of deducing cake height with time if stepped-up pressure filtrations are to be employed.

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FIGURES AND TABLES

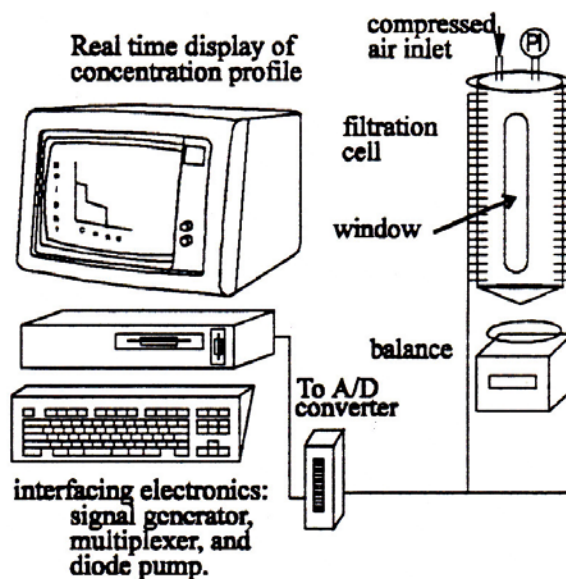


Figure 1: Experimental equipment.

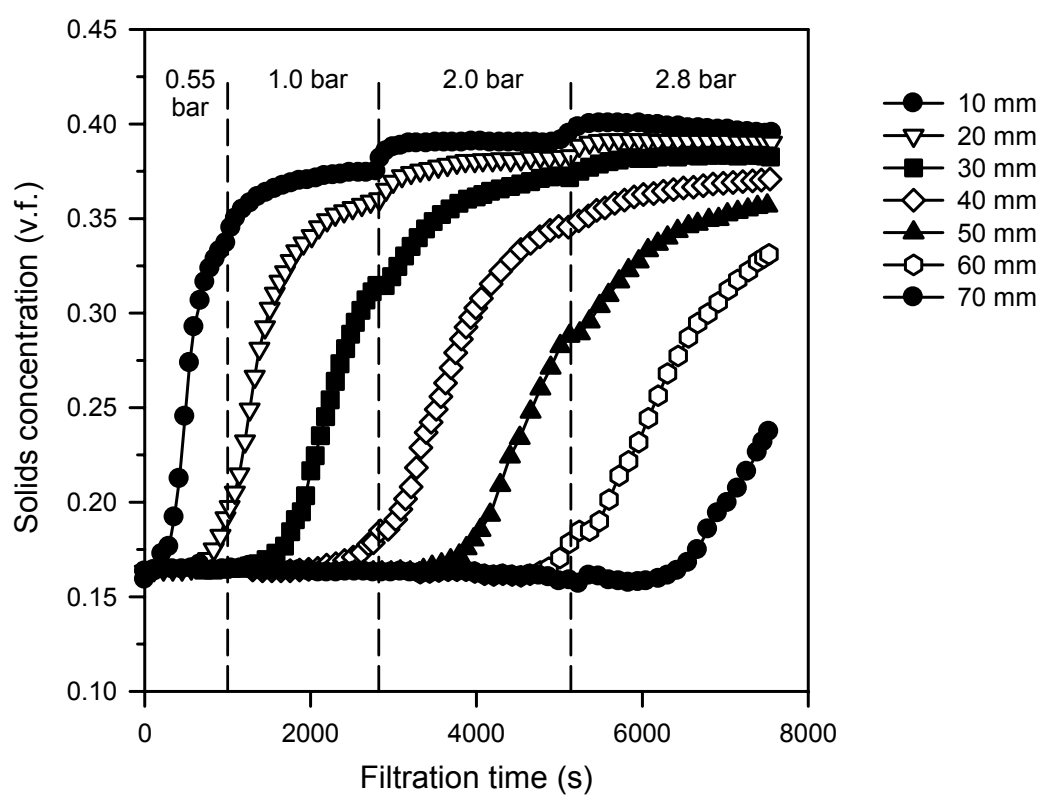


Figure 2: Solids concentration with time during the step-up filtration.

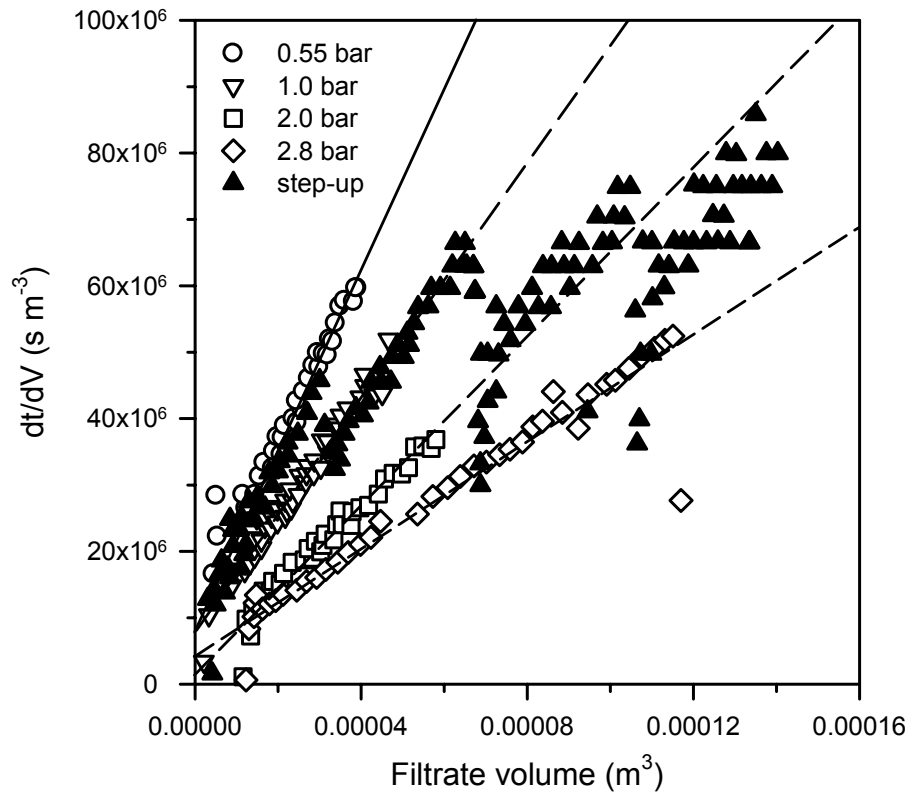


Figure 3: Reciprocal rate with filtrate volume; individual and step-up filtrations.

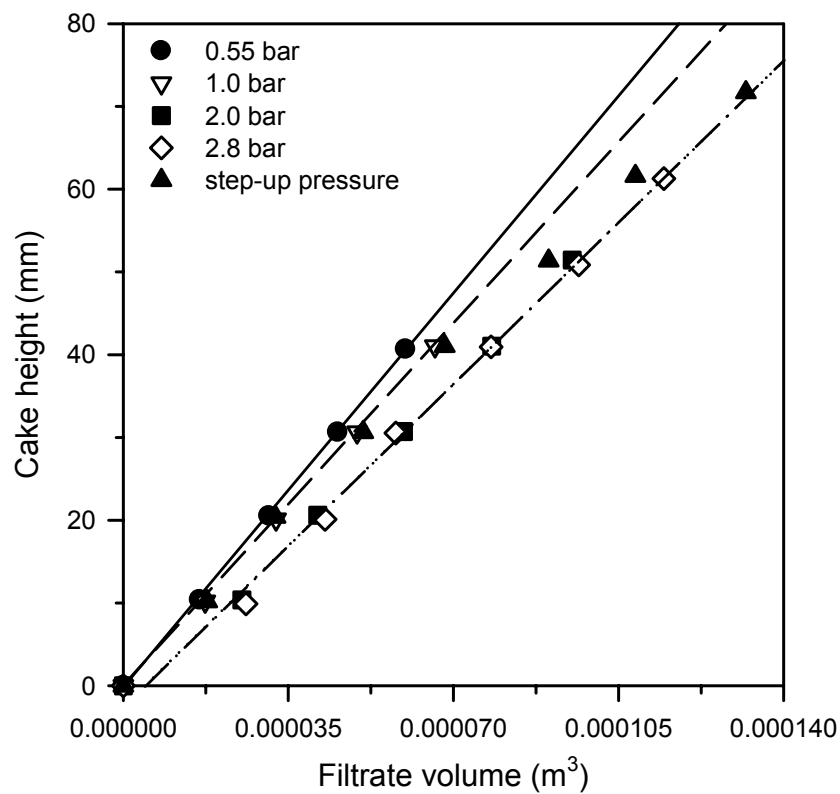


Figure 4: Filter cake height with filtrate volume; individual and step-up filtration.

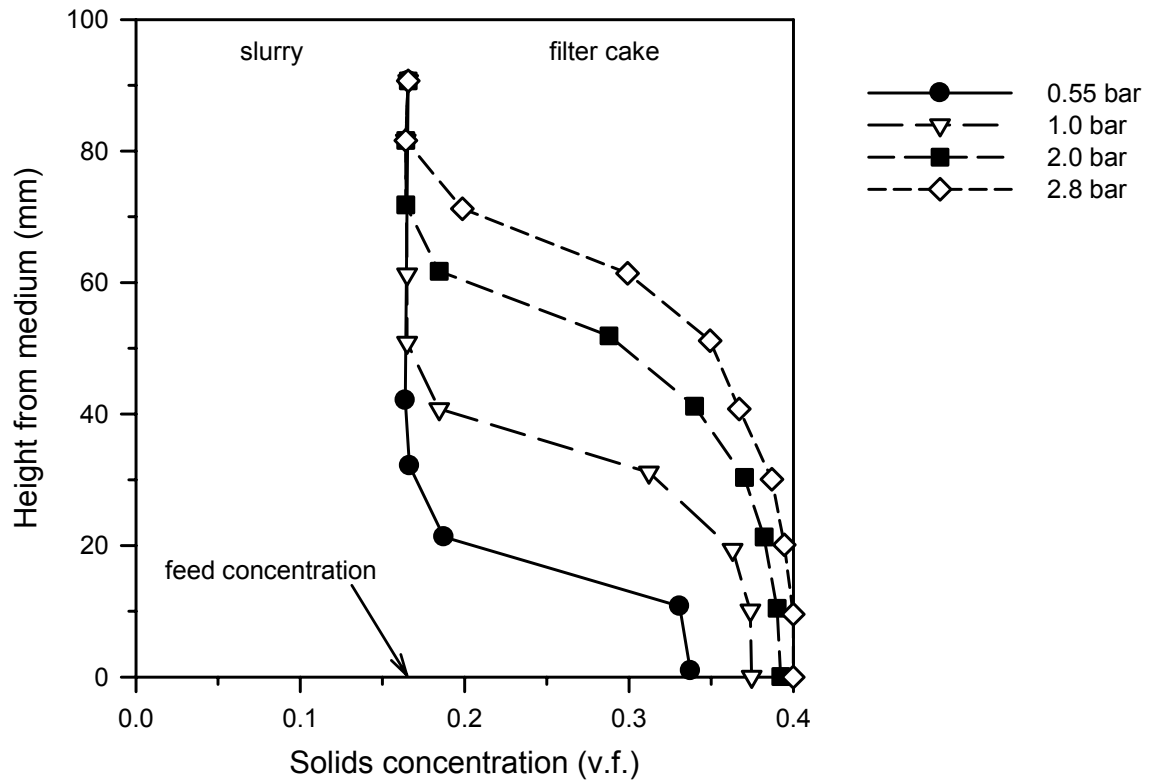


Figure 5: Solids concentration profiles inside the filter cake during step-up filtration.

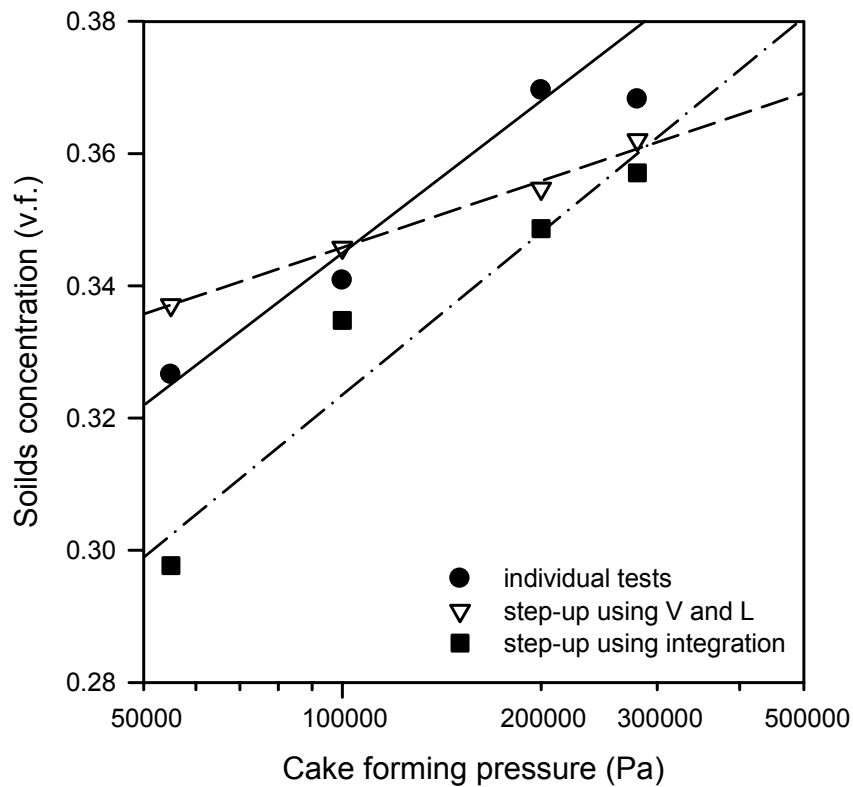


Figure 6: Average cake concentration with pressure; individual filtration and step-up filtration.

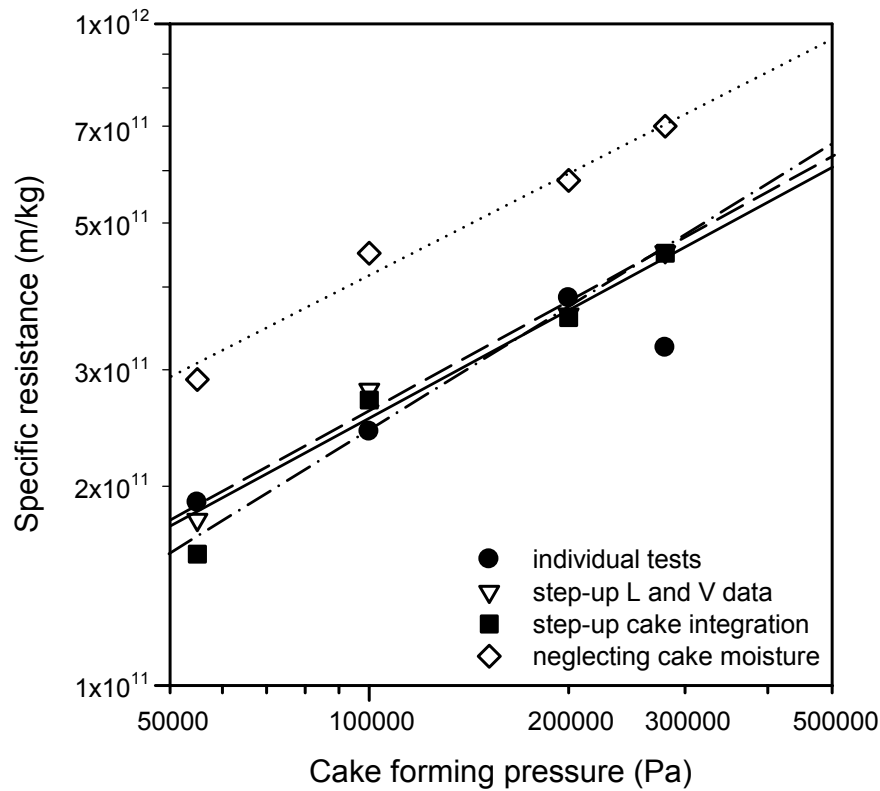


Figure 7: Average specific resistance with pressure; individual filtration and step-up filtration data.

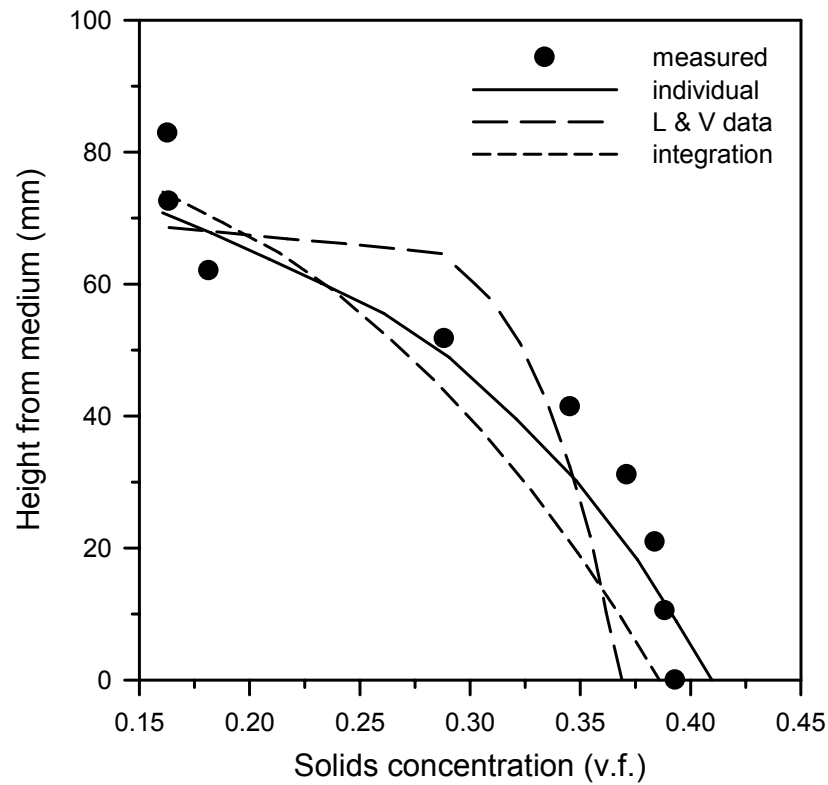


Figure 8: Solids concentration profile inside the filter cake during step-up filtration by equations (7) and (8).

Particle diameter (μm)	30	10	7.5	3.4	2.0	1.5	0.7	0.3
Cumulative mass less than (%)	100	90	80	50	35	30	20	10

Table 1: Particle size distribution of kaolin.

Filtration pressure (bar)	Individual tests (kg m^{-3})	Step-up equation (4) (kg m^{-3})	Step-up equation (9) (kg m^{-3})	Neglecting cake moisture (kg m^{-3})
0.55	873	845	961	516
1.0	836	824	851	516
2.0	776	804	819	516
2.8	778	790	801	516

Table 2: Values of dry weight of cake deposited per unit volume of filtrate used in the analysis of specific resistance using different approaches.

Filtration pressure (bar)	Individual tests (m kg^{-1})	Step-up via eqn. (4) (m kg^{-1})	Step-up via eqn. (9) (m kg^{-1})	Neglecting cake moisture (m kg^{-1})
0.55	1.89×10^{11}	1.81×10^{11}	1.59×10^{11}	2.96×10^{11}
1.0	2.43×10^{11}	2.81×10^{11}	2.72×10^{11}	4.48×10^{11}
2.0	3.81×10^{11}	3.74×10^{11}	3.67×10^{11}	5.82×10^{11}
2.8	3.25×10^{11}	4.59×10^{11}	4.53×10^{11}	7.03×10^{11}

Table 3: Values of specific resistance using different approaches.

	C_0	β	α_0	n
Individual tests	0.12	0.102	4.8×10^8	0.55
By equation (4)	0.22	0.044	4.5×10^8	0.55
By equation (9)	0.11	0.105	2×10^8	0.62
Using $m = 1$	N/A	N/A	11×10^8	0.52

Table 4: Values of constants in equations (10) and (11) using different approaches.