

# Measurement and Prediction of the Ultrafiltration of Whey Protein

**M. Davey and K. Landman**

Dept. of Mathematics and Statistics, The University of Melbourne, Victoria 3010, Australia

**J. M. Perera and G. W. Stevens**

Dept. of Chemical Engineering, The University of Melbourne, Victoria 3010, Australia

**N. D. Lawrence**

Gilbert Chandler College and Dept. of Chemical Engineering, The University of Melbourne, Victoria 3010, Australia

**M. Iyer**

Gilbert Chandler College, The University of Melbourne, Victoria 3010, Australia

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*Ultrafiltration of proteins and other compressible material is characterized by a significant transmembrane pressure and is often limited by the formation of a fouling layer. This study examines the compressive rheology of such fouling layers. A model is proposed that can be used to predict the performance of filtration processes where there is significant pressure drop across the clean membrane and the filtration rate is limited by fouling during the filtration cycle. The information required for the model can in principle be obtained from simple centrifuge tests and the clean membrane pressure drop. The model can be used to optimize the cycle times, pressures, and other operating parameters of the ultrafiltration process. The model was tested on the ultrafiltration of whey protein concentrate and shown to describe the experimental data. © 2004 American Institute of Chemical Engineers AIChE J, 50: 1431–1437, 2004*

**Keywords:** whey protein concentrate, pressure filtration, ultrafiltration

## Introduction

Ultrafiltration plants in the dairy industry are becoming more common in the production of many products such as whey protein concentrate. However, a major limitation in the operation of these plants is the production of a fouling layer on the membrane that reduces throughput to such an extent that the equipment must be regularly taken offline and cleaned. Although a significant amount of work (Cheryan and Merin, 1980; Kim et al., 1992; Stoner et al., 1993) has been presented

on the structure of the fouling layer, no models are available that accurately predict the onset of fouling as a function of the system and operating conditions.

The aim of this study is to develop a model of the fouling of filtration where the cake is compressible and there is significant membrane resistance.

The simplest descriptions of the filtration process are the conventional engineering style models of Coulson and Richardson (1985), Wakeman et al. (1991), Shirato et al. (1986), Terzaghi and Peck (1948), and Sivaram and Swamee (1977). de Kretzer et al. (2003) published a detailed review of the compressive rheology approach to the modeling of solid–liquid separation processes. These models assume that the fouling layer or filter cake is incompressible. They describe the flux of

Correspondence concerning this article should be addressed to G. W. Stevens at [gstevens@unimelb.edu.au](mailto:gstevens@unimelb.edu.au).

liquid through a uniform volume fraction of solids and have been used extensively to model the effect of the filter resistance and bed resistance in filtration. In such theories, a plot of  $t/V$  vs.  $V$  (where  $t$  is time and  $V$  is filtrate per unit area) is linear. The slope of the line gives an estimate of the (constant) filter cake resistance, whereas the intercept gives an estimate of the membrane resistance.

Iritani et al. (1991, 1993) reported whey and acid whey fouling layers to be compressible. For such compressible cakes, an increase in the pressure difference or the rate of flow will result in a denser cake, which offers a higher resistance to flow; thus it is inappropriate to use conventional incompressible theory to make predictions for compressible cakes. Iritani et al. (1993) studied the structure of bovine serum albumin (BSA) solutions using ultracentrifugation data. They stated that the filtration behavior could be accurately described only by a compressible cake resistance model. Their model was based on unstirred dead-end ultrafiltration data, and assumed the membrane resistance to be negligible compared with the filter cake resistance. Although in agreement with Iritani et al. (1993), with respect to the compressibility of the filter cake, the resistance of ultrafiltration membranes is quite large, and thus should be included in the analysis.

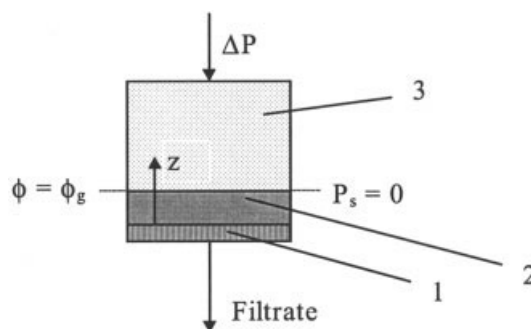
For many applications, the membrane resistance is negligible compared to the filter cake resistance. However, for the protein systems of interest here, this is not the case. Plots of  $t/V$  vs.  $V$  are definitely nonlinear and do not pass through the origin, so that a full compressional filtration theory is required. Landman et al. (1991) developed such a theory and subsequently applied the theory to optimize pressure filters in the case of no membrane resistance (Landman and White, 1997). In this article the theory is extended to ultrafiltration of proteins where there is significant membrane resistance and the fouling layer is compressible. The model allows the determination of the membrane and filter cake resistance.

We present herein an outline of the full compressional filtration theory and then apply it to ultrafiltration data for whey protein concentrate WPC80, which is a major component of the fouling layer that forms during the ultrafiltration of whey. This gives a basis for the prediction of the performance of an ultrafiltration membrane unit.

Although the theory has been developed for dead-end filtration, and thus is strictly only applicable to this case, providing the major resistance to flow is offered by the membrane and the compressed fouling layer, the theory should also be applicable to cross-flow filtration cases. This is because in cross-flow filtration the structure and rate of accumulation of the fouling layer will be different from that of dead-end filtration. However, the rearrangement of the particle networks that form the fouling layer is a function of the interparticle forces and network pressure and is thus independent of the method of build up. In applying the theory to cross-flow filtration, however, care must be taken in constructing the mass balance to ensure correct account is taken of the solid and fluid flows.

## Theory

The compressional filtration theory is based on that developed for flocculated suspensions. The key to an understanding of pressure filtration is the recognition of a solids or particle stress in addition to the hydrostatic or fluid stress. These two



**Figure 1. Region 1: membrane  $z = 0$ ; Region 2: filter cake or fouling layer  $0 < z < z_c(t)$ ; Region 3: initial concentrate  $z_c(t) < z < h(t)$  for a simple pressure filtration process.**

At  $z_c(t)$ ,  $P_s = 0$  and  $\phi = \phi_g$ .

stresses add to constitute the applied piston pressure and their contributions vary throughout the filter cell and are a function of time.

Buscall and White (1987) postulated that the network of particles could behave as a solid at a volume fraction  $\phi$  until the applied particle stress exceeded the compressive yield stress,  $P_y(\phi)$ , for that volume fraction. When this occurs the structure collapses and there is a local volume fraction increase. The further assumption that the collapse process is rapid compared to typical process times implies that the particle stress at a point in the suspension is always only infinitesimally above the compressive yield stress at the local volume fraction. For volume fractions below a particular value  $\phi_g$ , known as the gel point, no such network structure exists, and no solids stress is transmitted.

Buscall and White (1987) identified the other significant rheological property in solid/liquid separation: the hydraulic resistance  $r(\phi)$ , which is a monotonically increasing function of local volume fraction reflecting the hydrodynamic interference on any given particle by its neighbors.

Methods for measuring  $P_y(\phi)$  and  $r(\phi)$  are discussed in Landman and White (1994). These methods have been used successfully by a number of investigators. The resulting yield stress estimates were fitted with power law and exponential functions (Channell and Zukoski, 1997; Eberl et al., 1995; Eckert et al., 1996; Green, 1997; Miller et al., 1996). The hydraulic resistance was also fitted with various empirical relationships (Auzerais et al., 1990; Eberl et al., 1995; Green, 1997); the models are fairly insensitive to the form of  $r(\phi)$ .

Consider a one-dimensional treatment of the pressure filtration process, as shown in Figure 1. Let  $\phi(z, t)$  be the solids volume fraction at height  $z$  above the filter membrane as a function of time. The filtration process starts at zero time with a constant piston pressure  $\Delta P$  applied to the suspension in which the initial volume fraction  $\phi_0$  is uniform, with an initial piston height  $h_0$ . In any section of this system the solid and the fluid pressure are equal to the total pressure exerted on the system, that is,  $P_s + P_f = \Delta P$ , where  $P_s$  is the pressure exerted by the solid network and  $P_f$  is the fluid pressure. From a process perspective, the filtrate volume per unit area  $V(t)$  with time  $t$  is monitored and this study seeks to determine how the control factor  $\Delta P$  may affect the throughput of the pressure filtration process.

For ultrafiltration,  $\phi_0$  is smaller than  $\phi_g$ , and at this low volume fraction the solids cannot transmit stress. Thus in a region near the piston the volume fraction remains at this  $\phi_0$ , so there is no network stress, no compression, and the entire applied stress is carried by the fluid pressure. In the absence of gravity (which may be neglected where sedimentation times are large compared to filtration times) there is no physical mechanism to change the local concentration of particles in this unnetworked region.

Of course the fluid flux through the membrane leads to the buildup of particle concentration there and the establishment of the particle network. With time, a bed of networked particles grows from the membrane toward the piston. Thus for  $\phi_0 < \phi_g$  the initial stage of filtration is characterized by the growth of the compact bed above which there is a region extending to the piston with the initial volume fraction of particles  $\phi_0$ . Note that the volume fraction of particles in the compact bed is not uniform, varying from  $\phi(0, t)$  to  $\phi_g$  at the top of the bed because the particle stress falls to zero at this point. Note that in the absence of a membrane resistance,  $\phi(0, t)$  equals its final value of  $\phi_\infty$ , but this is not the case here.

At a certain time  $t_c$  the compact bed height reaches the piston, and thereafter the piston pressure is shared by the fluid and particles at all points in the filter press, and the network spans the whole vessel and is compressed. After a very long time, a uniform volume fraction  $\phi_\infty$  is established everywhere in the press. The network pressure at infinite time is everywhere  $\Delta P$  and the fluid pressure is zero everywhere. This study is concerned only with times such that  $t < t_c$ .

The fluid flux per unit area through the filter membrane is  $dV/dt$  at time  $t$  and the corresponding fluid pressure drop across the membrane is  $R_m dV/dt$  where  $R_m$  is the membrane hydraulic resistance. At the membrane, the sum of particle and fluid stresses is then given by

$$P_y(\phi(0, t)) + R_m \frac{dV}{dt} = \Delta P \quad (1)$$

As noted above, if the membrane resistance is neglected in comparison with the resistance of the filter bed, then from Eq. 1 the volume fraction of solids at the membrane is determined by  $P_y[\phi(0, t)] = \Delta P$ , and in the case of constant  $\Delta P$ , the volume fraction at the membrane  $\phi(0, t) = \phi_\infty$ . Because this is not the case in this study, the boundary condition (Eq. 1) does not provide a value  $\phi(0, t)$ .

The equations governing the filtration process were derived by Landman et al. (1995) and may be summarized by a diffusion equation for the local volume fraction. Let  $z_c(t)$  be the internal boundary that is the top of the growing filter cake, which exists for  $t < t_c$ , and  $h(t)$  is the position of the piston. Both of these boundaries are moving with time and must be determined in solving the model equations

$$\frac{\partial \phi}{\partial t} = \frac{\partial}{\partial t} \left[ D(\phi) \frac{\partial \phi}{\partial z} - \phi \frac{dV}{dt} \right] \quad 0 < z < h(t), t > 0 \quad (2)$$

where

$$D(\phi) = \frac{V_p}{\lambda} \left[ \frac{dP_y/d\phi}{r(\phi)} \right] (1 - \phi)^2 \quad (3)$$

where  $V_p$  is the floc volume and  $\lambda$  is the Stokes drag coefficient, which for spheres is  $6\pi$ . These terms are associated with the hydraulic bed resistance term as  $\lambda r(\phi)/V_p$ . Later  $P_y(\phi)$  and  $r(\phi)$  are specified and thus the coefficient  $V_p/\lambda$  is the only unknown part in the function  $D(\phi)$ . In addition to Eq. 1, further conditions associated with Eq. 2 are

$$\phi[z_c(t), t] = \phi_g \quad (4)$$

$$(\phi_g - \phi_0) \left( \frac{dh}{dt} - \frac{dz_c}{dt} \right) = D(\phi_g) \frac{\partial \phi}{\partial z} [z_c(t), t] \quad (5)$$

$$\frac{\partial \phi}{\partial z} [h(t), t] = 0 \quad (6)$$

$$\int_0^{h(t)} \phi(z, t) dz = \phi_0 h_0 \quad (7)$$

$$\phi(z, 0) = \phi_0 \quad (8)$$

The conditions expressed in Eqs. 4 and 5 express, respectively, the volume fraction and mass conservation across the discontinuity at the top of the filter cake. Equation 6 is a consequence of the fluid and particle velocities at the piston being equal to the flux per unit area through the membrane, whereas Eq. 7 expresses conservation of solids mass and Eq. 8 is an initial condition.

It is convenient to introduce various scalings and transformations to these equations to solve them. The scalings used by Landman et al. (1997) are adapted for this case where  $R_m$  is nonnegligible. Both the length and time scales are made dimensionless (details are outlined in Appendix) using

$$Z = \frac{z}{L} \quad T = \frac{t}{S} \quad (9)$$

where the chosen length and time scales  $L$  and  $S$ , respectively, are

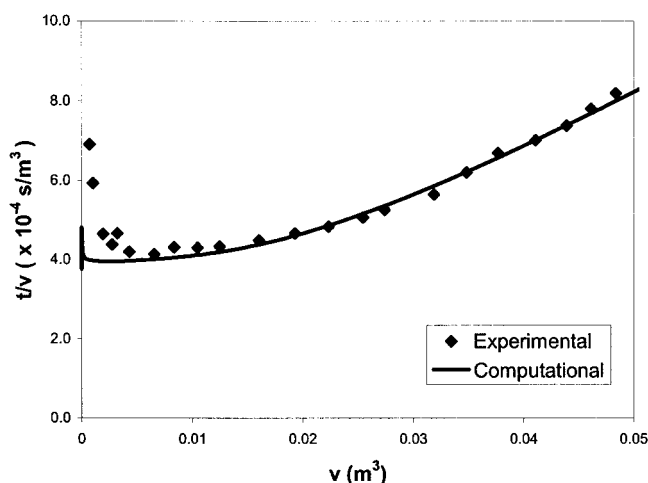
$$L = \frac{R_m D_\infty \phi_\infty^2}{\Delta P \phi_0} \quad S = \frac{l^2}{D_\infty} \left( \frac{\phi_\infty}{\phi_0} \right)^2 \quad (10)$$

where  $D_\infty = D(\phi_\infty)$ . Because  $R_m$  and  $(V_p/\lambda)D_\infty$  (and thus  $D_\infty$ ) are unknown, neither of these can be set. When the experimental data are fitted to the results of the theoretical model, these two scales will be determined.

In solving for the buildup of the filtercake, the final equations to be solved are

$$w_c^2 \frac{\partial e}{\partial \Gamma} = \frac{\partial}{\partial X} \left[ \Delta(e) \frac{\partial e}{\partial X} \right] + w_c X \frac{dw_c}{dT} \frac{\partial e}{\partial X} \quad 0 < X < 1, T > 0 \quad (11)$$

$$\frac{P_y[e(0, T)]}{\Delta P} + \frac{1}{w_c} \Delta[e(0, T)] \frac{\partial e}{\partial X}(0, T) = 1 \quad (12)$$



**Figure 2.** Comparison of experimental data and theory at  $\Delta P = 1.75 \times 10^5$  Pa,  $\phi_\infty = 0.733$ .

$$e(1, T) = e_g \quad (13)$$

$$e(w, 0) = e_0 \quad (14)$$

$$w_c \frac{dw_c}{dT} (e_0 - e_g) = \Delta(e_g) \frac{\partial e}{\partial X} (1, T) \quad (15)$$

where  $e$  is the void ratio and  $w$  is the position of the top of the filter cake in terms of material coordinates (Kirby and Smiles, 1988) as

$$e = \frac{1 - \phi}{\phi} \quad w_c(Z, T) = \frac{1}{\phi_0} \int_0^{z_c} dZ \phi(Z, T) \quad (16)$$

This system is solved numerically using Mathematica code. To determine the flux per unit area  $V(t)$ , it is necessary to evaluate

$$\frac{V(T)}{L} = w_c \phi_0 \int_0^1 (e_0 - e) dX \quad (17)$$

To compare the computed dimensionless quantities with the experimental data of  $t/V$  vs.  $V$ , it must be noted that  $t/V$  and  $V$  have the dimensions of  $S/L$  and  $L$ , respectively. The two scales must be chosen, one for the coordinate and the other for the ordinate, to best fit the experimental data to the model results. In effect values are assigned to the length scale  $L$  and the time scale  $S$ . With the most appropriate choice, estimates of the two unknown quantities can be made, that is, the final diffusivity, which relates to the bed permeability and compressibility, and membrane resistance as

$$D_\infty = \frac{L^2}{S} \left( \frac{\phi_0}{\phi_\infty} \right)^2 \quad (18)$$

$$R_m = \frac{L \Delta P \phi_0}{D_\infty \phi_\infty^2} \quad (19)$$

The results of such fitting are considered in the Results and Discussion section.

## Experimental Details

### Materials

Whey protein concentrate 80 (WPC80) powder was donated by United Milk Tasmania Ltd. (UMT) Wynyard and The New Zealand Dairy Board. De Danske Sukkerfabrikker (DDS) GR60PP 25,000 molecular weight cutoff (MWCO) polysulfone membranes were used for the filtration experiments.

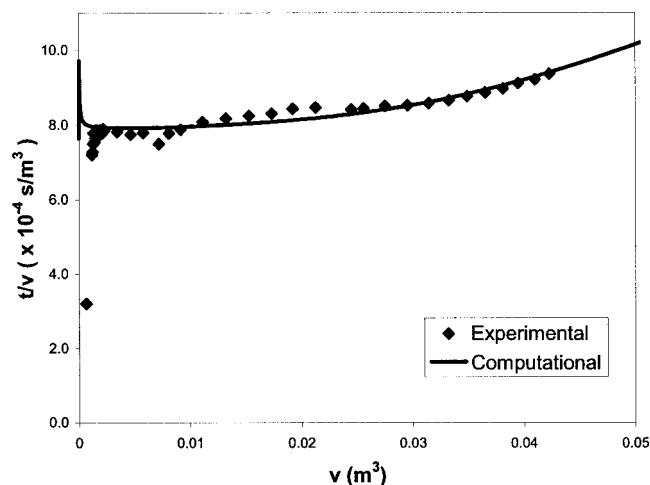
### Methods

Centrifuge experiments were carried out at 5°C in cylindrical vessels containing 30% (w/w) solutions of WPC80 in distilled water at speeds of 2500, 3000, 3500, 4000, 4500, 5000, and 5200 rpm using a Spintron Pty. Ltd. GT-155 refrigerated centrifuge. The equilibrium height of the solid component was measured after the vessels had been centrifuged for 120–250 h.

Ultrafiltration experiments with 15% (w/w) WPC80 feed solutions were conducted at 14–16°C using a DDS Mini-Lab 10 flat-sheet cross-flow ultrafiltration unit. The unit consisted of four flat-sheet membranes in series, providing a total membrane area of 0.0336 m<sup>2</sup>. The flow rate was approximately 4.2 L/min. The inlet pressure was maintained at 3.00–3.20 bar, and the outlet pressure was maintained at 2.00 bar. A back-pressure valve was manipulated to maintain these conditions. The trans-membrane pressure was in the range 1.75–3.00 bar. The trials were conducted for 11 h.

## Results and Discussion

The batch centrifuge technique of Buscall and White (1987) and Green et al. (1996) were used to estimate  $P_y(\phi)$ . The best fit was obtained with the following form



**Figure 3.** Comparison of experimental data and theory at  $\Delta P = 2.55 \times 10^5$  Pa,  $\phi_\infty = 0.804$ .

$$P_y(\phi) = k \left[ \left( \frac{\phi}{\phi_g} \right)^n - 1 \right]$$

where  $k = 220.39$  Pa,  $n = 4.069$ , and  $\phi_g = 0.142$ . We choose a typical  $r(\phi)$  (Landman and White, 1997) as

$$r(\phi) = (1 - \phi)^{-3.5}$$

Three sets of experimental data are presented in Figures 2–4. The experimental data do not extrapolate back to zero and show considerable curvature over the entire range, indicating a significant membrane resistance and compression of the fouling layer during the filtration. Thus the simple incompressible cake theory would not predict the data well. Theoretical model predictions based on the model described earlier, and generated by choosing appropriate length and time scales as described above, are also shown in Figures 2–4. The values of the parameters are as follows:  $\phi_0 = 0.118$ ,  $\phi_\infty = 0.733$  ( $\Delta P = 1.75 \times 10^5$  Pa),  $\phi_\infty = 0.804$  ( $\Delta P = 2.55 \times 10^5$  Pa),  $\phi_\infty = 0.837$  ( $\Delta P = 3.0 \times 10^5$  Pa).

As can be seen from Figures 2–4, the theory is able to accurately predict the behavior of the filter over a wide range of filtrate volumes. The measured values of volume at small times have a significant error related to the need to fill the chamber on the permeate side of the membrane before the volume of permeate can be determined and so the fit at small volumes ( $< 0.005$  m<sup>3</sup>) is not expected to be accurate. Also at small volumes the resistance of the fouling layer is not significant and other resistances may be controlling the rate of filtration.

Table 1 presents the membrane resistance  $R_m$ , and the unknown component of the hydraulic bed resistance  $V_p/\lambda$ , which is used to determine the particle diffusivity within the fouling layer.

Because in each case the same solutions are used and only the pressure is different, the parameter  $V_p/\lambda$  should be a constant. There is no consistent variation of  $V_p/\lambda$  with pressure. The value of  $R_m$ , however, varies considerably, over a range of a factor of 3.5. The same membrane materials were used in each case; however, it is known that significant variation in

**Table 1. Fitted Values of  $R_m$  and  $V_p/\lambda$**

$\Delta P$ (Pa)	$R_m$ (Pa s <sup>-1</sup> m <sup>-1</sup> )	$V_p/\lambda$ (m <sup>3</sup> )
$1.74 \times 10^5$	$6.74 \times 10^{10}$	$5.85 \times 10^{11}$
$2.55 \times 10^5$	$1.98 \times 10^{11}$	$6.35 \times 10^{11}$
$3.00 \times 10^5$	$5.65 \times 10^{10}$	$2.99 \times 10^{11}$

membrane resistance is observed from clean water experiments. For example, Lawrence (1998) obtained a variation in clean water fluxes from 131 to 155 L m<sup>-2</sup> h<sup>-1</sup> for different samples of the same membrane, before any fouling, indicating a variation in the membrane resistance. Also the previous pressure history of the membrane plays an important role in the membrane resistance. If the membrane has been exposed to higher pressures, then the membrane resistance has been shown to increase as a result of compression of the polymer membrane. Other factors that will influence both  $R_m$  and  $V_p/\lambda$  are protein intrusion into the membrane pores, causing blocking and increasing  $R_m$ , and conformational changes in the proteins, causing changes in the network structure leading to changes in  $V_p/\lambda$ . It is likely that both of these effects will increase with a pressure drop across the membrane. In this case there is no evidence to suggest that these parameters change with pressure. However, more data are required to determine whether this is more generally true.

Once these values have been established for a given solution and membrane configuration, they can be used to investigate how changes in pressure, flow rate, and filtration time influence the performance of a given system. For example, maximizing the total throughput through a filtration system by varying the cycle time—that is, how long the filter should be run before cleaning—is a common goal in any filter operation. This can easily be done through the solution of Eq. 17 with the measured parameters  $R_m$  and  $V_p/\lambda$  obtained from pilot-scale experiments.

## Conclusions

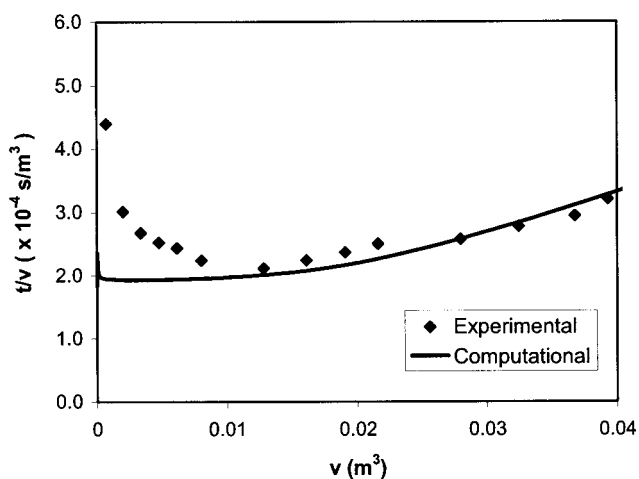
Ultrafiltration of whey protein concentrate is shown to have a significant membrane resistance and a compressible fouling layer. A new model for the interpretation of the performance of filters with compressible fouling layers and significant membrane resistance is presented and shown to be able to model the performance of one type of filter.

## Acknowledgments

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## Notation

- $D_\infty$  = diffusion coefficient at maximum volume fraction, m<sup>2</sup>/s
- $D(\phi)$  = diffusion coefficient as a function of  $(\phi)$ , m<sup>2</sup>/s
- $e$  = void fraction (see Eq. A2)
- $h$  = height of piston, m
- $h_0$  = initial piston height, m
- $h(t)$  = piston position, m
- $L$  = length scale (see Eq. 8), m
- $l$  = arbitrary length used to make variables dimensionless, m
- $P_f$  = fluid pressure, Pa
- $P_s$  = pressure exerted by the solid network, Pa



**Figure 4. Comparison of experimental data and theory at  $\Delta P = 3.0 \times 10^5$  Pa,  $\phi_\infty = 0.837$ .**

$P_y(\phi)$  = compressive yield stress for the volume fraction ( $\phi$ ), Pa  
 $\Delta P$  = constant piston pressure, Pa  
 $r(\phi)$  = hydraulic resistance,  $\text{Pa s}^{-1} \text{m}^2$   
 $R_m$  = membrane resistance,  $\text{m}^{-1}$   
 $S$  = time scale (see Eq. 8), s  
 $t_c$  = time at which the compact bed height reaches the piston, s  
 $T$  = dimensionless time of filtration  
 $t$  = time, s  
 $V$  = volume of filtrate per unit area of membrane, m  
 $V(t)$  = filtrate volume per unit area, m  
 $V_p$  = floc volume (see Eq. 3),  $\text{m}^3$   
 $w$  = top of the cake in material coordinates (Kirby and Smiles 1988), Eq. A1  
 $x$  = variable defined in Eq. A10  
 $z_c(t)$  = internal boundary, m  
 $z$  = height of filter cake, m  
 $Z$  = dimensionless height of filter cake

## Greek letters

$\lambda$  = Stokes drag coefficient (see Eq. 3)  
 $\phi$  = volume fraction of solids in a solution  
 $\phi_g$  = volume fraction at the gel point  
 $\phi_\infty$  = maximum volume fraction  
 $\phi_0$  = initial volume fraction  
 $\phi(z, t)$  = solids volume fraction at height  $z$  above the filter membrane as a function of time

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## Appendix

### Scaling and transforming the model equations

The moving boundary corresponding to the piston position  $h(t)$  can be removed from the problem by changing to material coordinates  $w$  (Kirby and Smiles, 1988), defined as

$$w(Z, T) = \frac{1}{\phi_0} \int_0^Z dZ \phi(Z, T) \quad (\text{A1})$$

Then make the transformation  $\phi(Z, T)$  to  $\phi(w, T)$  and introduce the void ratio

$$e = \frac{1 - \phi}{\phi} \quad (\text{A2})$$

In terms of the void ratio, Eqs. 1–8 can be restated as (Kirby and Smiles, 1988)

$$\frac{\partial e}{\partial T} = \frac{\partial}{\partial w} \left[ \Delta(e) \frac{\partial e}{\partial w} \right] \quad 0 < w < 1, T > 0 \quad (\text{A3})$$

$$\frac{P_y[e(0, T)]}{\Delta P} + \Delta[e(0, T)] \frac{\partial e}{\partial w}(0, T) = 1 \quad (\text{A4})$$

$$e[w_c(T), T] = e_g \quad (\text{A5})$$

$$\frac{dw_c}{dT}(e_0 - e_g) = \Delta(e_g) \frac{\partial e}{\partial w}(w_c, T) \quad (\text{A6})$$

$$\frac{\partial e}{\partial w}(1, T) = 0 \quad (\text{A7})$$

$$e(w, 0) = e_0 \quad (\text{A8})$$

where

$$\Delta(e) = \frac{\phi^2 D(\phi)}{\phi_\infty^2 D_\infty} \quad (\text{A9})$$

In the case where  $R_m = 0$ , this system can be solved using a similarity transformation. Given that  $R_m \neq 0$ , this is no longer applicable. However, the system can be transformed to that on a fixed domain by introducing

$$X = \frac{w}{w_c(T)} \quad (\text{A10})$$

Then the system, expressed in Eqs. 11–15, becomes as shown in the main text.

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