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The Vacuum Filtration of Iron Ore Ultrafines

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ABSTRACT

The dewatering of fine particles ($-0.5 + 0$ mm) derived from iron ore processing is a topic of increasing importance to Australian producers. With little previous published work in this area, it is difficult to predict how these materials are going to dewater, in particular during vacuum filtration. The work described in this paper was therefore undertaken in order to help overcome this paucity of background information. The sample of iron ore that was tested responded well to vacuum filtration provided that anionic flocculants were used as a filter aid. In the presence of these polymers, cake formation times were very short (mainly below 10 seconds) and the moisture levels of 18 mm thick filter cakes were reduced to 10–11 wt% after dewatering times of only 40 seconds. By comparison, the cationic and nonionic flocculants tested were much less effective. The mathematical model devised by Wakeman shows considerable promise as a tool for predicting the kinetics of desaturation.

Key Words. Iron ore; Filtration; Flocculation; Modeling

INTRODUCTION

The dewatering of ultrafine particles (below ca. 0.5 mm) from iron ore processing is becoming increasingly important with Australian producers. Issues such as minimizing costs associated with downstream transport and handling operations, maximizing plant efficiency, and ensuring high product consistency are receiving increasingly greater emphasis. Even relatively mod-

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est reductions in product moisture can lead to large financial savings in an industry which has an output of many tens of millions of tonnes per annum. The problem is especially acute for the finest size ranges, below ca. 0.5 mm, where product moisture will be highest.

The subject of iron ore dewatering has, however, not previously been widely reported, and several of those papers that have appeared are sparse on the details. One of the more in-depth studies was by Hosten et al. (1–3) who investigated the behavior during vacuum filtration of different iron ore samples under a range of conditions. In particular (1), they investigated the effect of particle size on dewatering of a hematite concentrate using a range of flocculants and surfactants. In the presence of a nonionic flocculant, product moisture contents as low as 8–9 wt% were reported for dewatering times of up to 120 seconds using feeds of ca. 80 wt% < 37 μm, although no data were given on cake thickness and equivalent product throughput.

Hosten et al. reported using nonionic or cationic polyacrylamide flocculants. Rayner (4) investigated the effect of a range of polyacrylamides (including anionics) on the rate and extent of filtration of a deslimed iron ore concentrate with a top size of 250 μm. He found that the polymer dose required for optimum product moisture was up to three times that for optimal filtration rate. Product moisture contents down to 3.5–4.0 wt% were quoted although no equivalent throughput figures were given. A recent paper by Hu et al. (5) investigated the vacuum filtration of a hematite concentrate of mean particle size 6.5 μm. Perhaps not surprisingly, at such fine sizes there did not appear to be any significant desaturation.

Thus there appears to be a need for more work on the dewatering behavior of iron ore fines. The results presented in this paper were aimed at establishing and, for the first time, modeling the vacuum filtration characteristics of a sample of iron ore from Western Australia, where the issue of dewatering will become of major importance over the next few years.

THEORY

Vacuum filtration consists of several stages, the most important of which are:

- Initial bridging of the particles across the filter medium as the first few layers of the filter cake start to form.
- Cake formation characterized by a continuously increasing cake thickness and single phase (water) flow through the forming cake.
- Cake desaturation characterized by the flow of two phases (air and water) through the cake during which the largest pores are emptied of water.

In the cake formation stage, the flow of wetting fluid through the bed is described by Darcy's law;

$$\frac{dV}{dt} = \frac{KA\Delta P}{\eta L} \tag{1}$$

where dV/dt is the rate of flow of filtrate, K is the cake permeability, ΔP is the pressure difference across the cake, A is the filter cake area, L is the cake thickness, and η is the fluid viscosity. Integrating Eq. (1) and rearranging yields

$$\frac{t}{V} = \frac{K\eta L}{2A^2\Delta P} V + \frac{R_m \eta L}{A\Delta P} \tag{2}$$

where R_m is the medium resistance. Provided that the cake is incompressible (which is, to a first approximation, the case for iron ore fines), a graph of t/V vs V will give a straight line, from which the cake permeability can be calculated.

Many models have been developed over the years, since the work of Ruth (6) in the early 1930s, for predicting the desaturation of filter cakes during vacuum filtration. In this present study the model devised by Wakeman (7, 8) was used since it had rated highly in a comparative assessment of several models undertaken by Carleton and Mackay (9) and had been used successfully by the authors in a previous study on modeling the vacuum filtration of coal (10). The model provides, inter alia, a prediction of the desaturation profile with time. Parameters associated with equilibrium dewatering conditions such as breakthrough pressure, equilibrium saturation, and pore size distribution index are used to set up and solve equations for the flow of both air and water through elemental slices of the filter cake. The original Wakeman paper used graphical methods to predict air and water flows. The authors had previously transformed Wakeman's graphs into a series of lookup tables in a computer spreadsheet (10).

MATERIALS

Iron Ore Sample

The iron ore fines used in this study were from a mine in Western Australia and were nominally 100 wt% < 600 μm. The material was supplied as an aqueous slurry with a solids content of 55 wt%. The solids were stored wet at all times in case drying out affected dewatering, as is the case with coal (11). The particle size distribution measured by wet screening was as expected, with

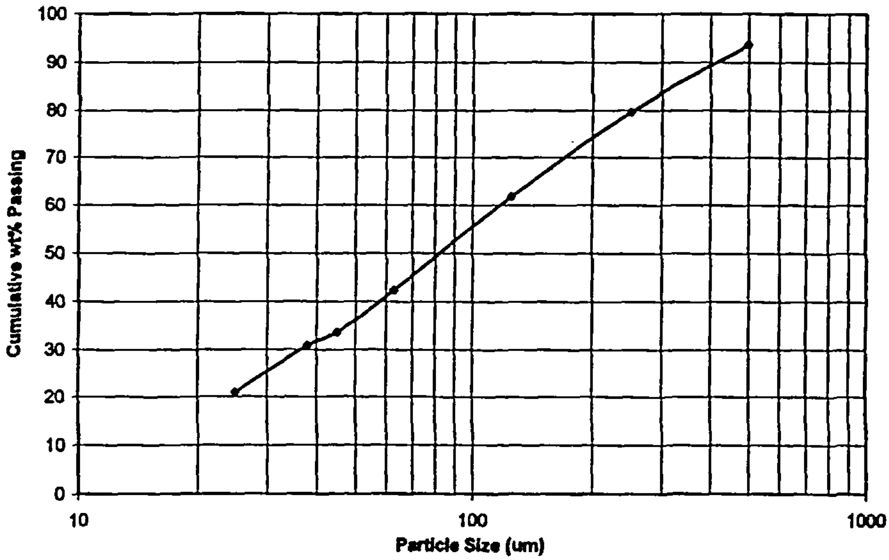

FIG. 1 Size analysis by screening of the iron ore fines.

> 90 wt% being below 500 μm (Fig. 1). The density of the solids as measured by helium pycnometry was 4710 kg/m^3.

Flocculants and Flocculant Solution Preparation

At the outset of the study there was no way of predicting what might be a good flocculant for the iron ore sample. Four different flocculants, all supplied by Allied Colloids Pty Ltd., were therefore evaluated. The details are given in Table 1. Magnafloc 156 was used in most of the tests in order to establish a baseline of performance and optimum dose. The other polymers

TABLE 1
Summary of Flocculants

Flocculant	Charge	Molecular weight
Magnafloc 333	Nonionic	High
Magnafloc 156	Moderately anionic	Medium
Magnafloc 919	Highly anionic	Very high
Zetag 53	Moderately cationic	High–very high

were tested to establish the effect of flocculant ionicity on dewatering. All polymers were added as a 0.1 wt% aqueous solution, which was stored in the refrigerator and prepared fresh every 3 days.

EXPERIMENTAL

The coarser particles of iron ore settle extremely quickly in aqueous suspension. A procedure was therefore devised for taking subsamples as representatively as possible for the filtration experiments. Repeat filtration tests confirmed that the split was reasonably accurate.

For each filtration test, the slurry subsample was conditioned with flocculant in a 250-mL baffled beaker. The required amount of flocculant solution was added from a syringe over a period of 10 seconds and stirred for another 30 seconds at a mixing power input of 10 W/m^3 of slurry prior to pouring the slurry into the filtration cell described below.

The filtration tests were performed in a 50-mm diameter top-fed, brass, vacuum filtration cell fitted with a monofilament polypropylene filter medium (pore size 44 μm). A fresh cloth was used for each experiment. The filtrate removed during each test was collected in a flask mounted on a balance, the weight was recorded every 1 second by a computer connected to the balance via an RS232-interface. After pouring the conditioned slurry into the filter cell, a vacuum of 70 kPa was applied immediately and remained constant throughout the test. All experiments were conducted for a total dewatering time (formation and desaturation) of 4 minutes, which in practice was sufficient for almost complete desaturation since formation was extremely rapid. Many tests were carried out in duplicate.

After completion of each test, the cake was removed from the cell. Cake thickness was determined by piercing it in four widely spaced locations with a vernier caliper, the quoted values being the arithmetic mean of the four determinations. The cake was then placed in a forced draught oven at 110°C overnight for moisture content determination.

RESULTS

Effect of Flocculant Dose on Cake Formation

As shown in Fig. 2, the rate of cake formation in the presence of only a small amount of flocculant was extremely high. At a dose of $\geq$ 5 g/tonne, flocculation was both rapid and comprehensive with a clear supernatant. This high efficiency of flocculation together with the high density of the iron ore particles caused very rapid settling, leading to very low ($\leq$ 10 seconds) cake formation times. By contrast, at doses below 5 g/tonne, cake formation be-

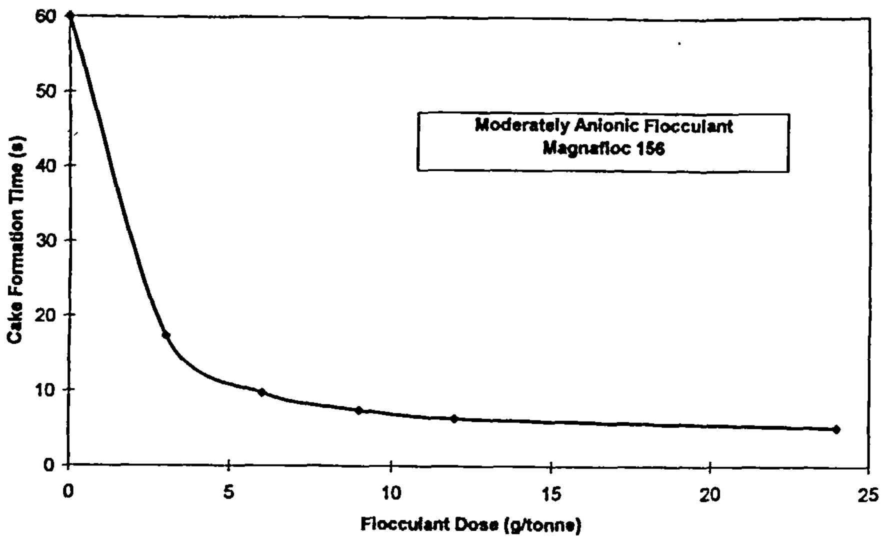

FIG. 2 Effect of flocculant dose on cake formation time.

came much slower. In the absence of flocculant, the filter cake took a minute to form.

Equation (2) was used to determine filter cake permeability. Plots of t/V against V during cake formation were prepared, and the gradients of the resultant lines were used to calculate the values of permeability, as shown in Table 2.

The effect of adding the flocculant was to cause a massive increase in cake permeability from just below 10×10^{-14} up to 370×10^{-14} m^2. The relation between permeability and flocculant dose is not smooth, probably due to the difficulties in obtaining reliable t/V vs V profiles caused by the very short

TABLE 2
Effect of Polymer Dose on Filter Cake Permeability

Flocculant dose (g/tonne)	Cake permeability ($m^2 \times 10^{-14}$)
0	8.9
3	182
6	110
10	240
12	370

formation times. Permeabilities determined from cakes with long form times are likely to be more accurate than those where the form time is very short.

Effect of Flocculant Dose on Cake Desaturation

The effect of dose of Magnafloc 156, between 0 and 24 g/tonne, on the desaturation of iron ore fines is shown in Fig. 3. In the absence of flocculant, filtration was slow. Even after 4 minutes the cake was still effectively saturated with a moisture content of 19.2 wt%. The presence of 6 g/tonne of flocculant caused both a big increase in filtration rate and a considerable reduction in final cake moisture at all filtration times, which is entirely consistent with the increase in cake permeability shown in Table 2. Increasing the flocculant dose beyond 6 g/tonne gave modest increases in filtration rates that were particularly noticeable in the early stages of desaturation, but no significant benefit in final moisture after 4 minutes. Interestingly, there was no deterioration in performance, as can frequently be the case when flocculant is added in excess. Reducing the flocculant dose to 3 g/tonne did have a significant detrimental effect, reducing both the rate and extent of filtration.

The data in Fig. 3 were interpolated to provide values of filter cake moisture after filtration times of 20, 40, and 60 seconds. These values are shown to-

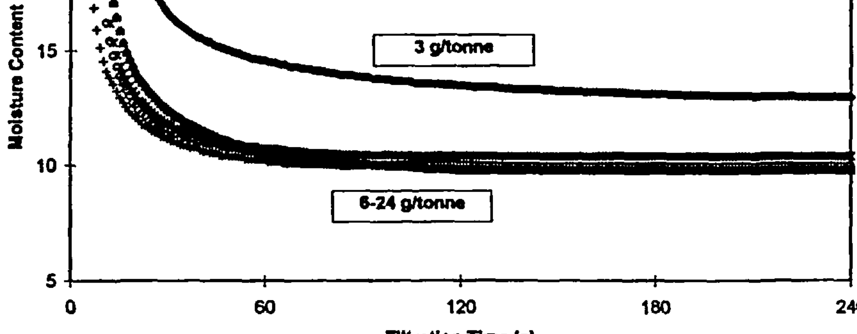

FIG. 3 Effect of flocculant dose on desaturation kinetics.

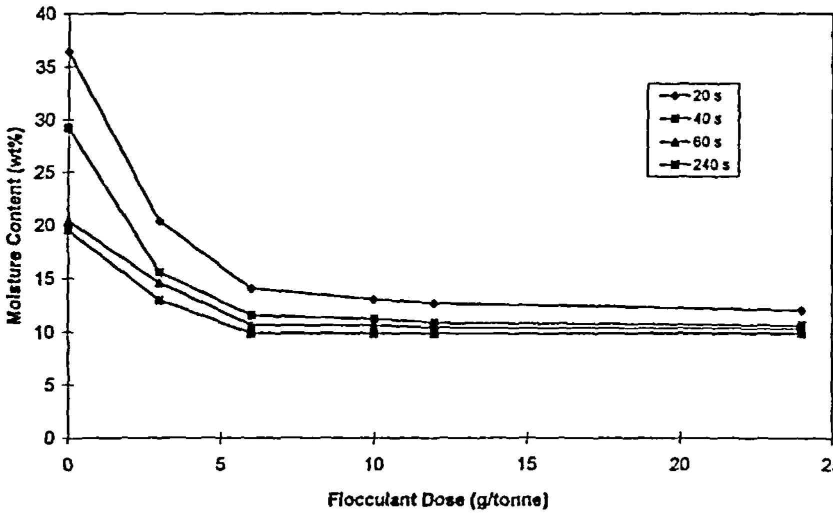

FIG. 4 Effect of flocculant dose and filtration time on product moisture.

gether with the determined values of cake moisture after 240 seconds as a function of floc dose in Fig. 4.

Only 40 seconds of filtration time and a flocculant dose of 10 g/tonne were needed to reduce filter cake moisture to 10–11 wt%. In all these experiments the filter cake thickness had a mean value of ca. 18 ± 1 mm (35.6 ± 1.8 kg/m^2). All values of cake porosity were in the 0.58 ± 0.1 range, which was higher than expected—for example, flocculated cakes from the vacuum filtration of −0.5 + 0 mm coal typically have porosity values around 0.4. Using the determined cake properties, extrapolation of the bench scale filtration results gave a predicted throughput of 3200 kg/m^2/h.

The above results differ considerably from those of Hosten et al. (1) and Rayner (4). The feedstock used in these tests (ca. 30 wt% < 37 μm) was considerably coarser than Hosten et al.'s (ca. 80 wt% < 37 μm) and yet the final product moisture contents were higher by at least 1 wt%. In addition, Rayner found that the flocculant dose for optimum cake formation was three times that for optimum dewatering. In our experiments, increasing the flocculant dose steadily increased cake formation rates but had little effect on final cake moisture. The reasons for these differences are unclear, but seem to highlight the dangers in extrapolating or generalizing behavior from one sample to another.

Effect of Flocculant Type on Cake Formation

Four flocculants were investigated for their influence on the dewatering of iron ore fines, ranging from Magnafloc 919 (highly anionic) to Zetag 53 (moderately cationic). All flocculants were examined at a low dose of 5 g/tonne in order to accentuate any differences between their effects.

The data in Fig. 5 show clearly the significant effect of flocculant type on cake formation time. As the anionic character of the polymer increases, so does the rate of cake formation, with Magnafloc 919 performing the best, although the difference between it and Magnafloc 156 was small.

The differences in cake formation time are reflected in the effect of polymer type on permeability, the values of which were determined from Eq. (2) and are shown in Table 3.

Both the anionic polymers, and particularly the Magnafloc 919, gave massive increases in permeability compared with no polymer present, whereas the increases caused by the nonionic and cationic polymers were much more modest.

Effect of Flocculant Type on Cake Desaturation

The effect of flocculant type added at a dose of 5 g/tonne on the kinetics of desaturation of iron ore fines is shown in Fig. 6. The two anionic flocculants

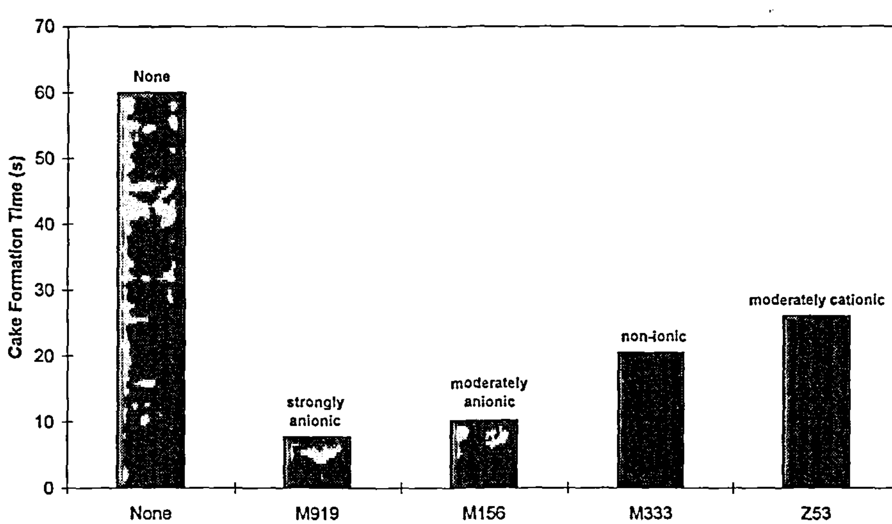

FIG. 5 Effect of flocculant type on cake formation time.

TABLE 3
Effect of Flocculant Type on Filter Cake Permeability

Flocculant type (5 g/tonne)	Cake permeability ($m^2 \times 10^{-14}$)
None	8.9
Magnafloc 919	630
Magnafloc 156	110
Magnafloc 333	19
Zetag 53	44

gave virtually identical dewatering profiles and values of final moisture between 9.9 and 10.1 wt%. It was surprising that the rate of desaturation with the Magnafloc 919 wasn't somewhat higher than for the Magnafloc 156, since the cake permeability with the former was much higher. By comparison, and as expected from the permeability data, the nonionic Magnafloc 333 and cationic Zetag 53 produced significantly slower dewatering, leading to much higher values of residual moisture after 4 minutes of 14.7 and 15.2 wt%, respectively.

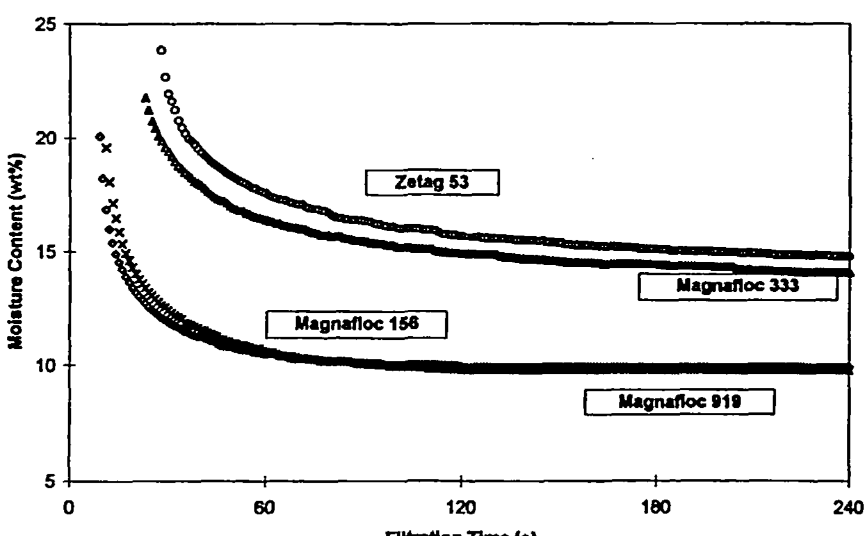

FIG. 6 Effect of flocculant type on desaturation kinetics.

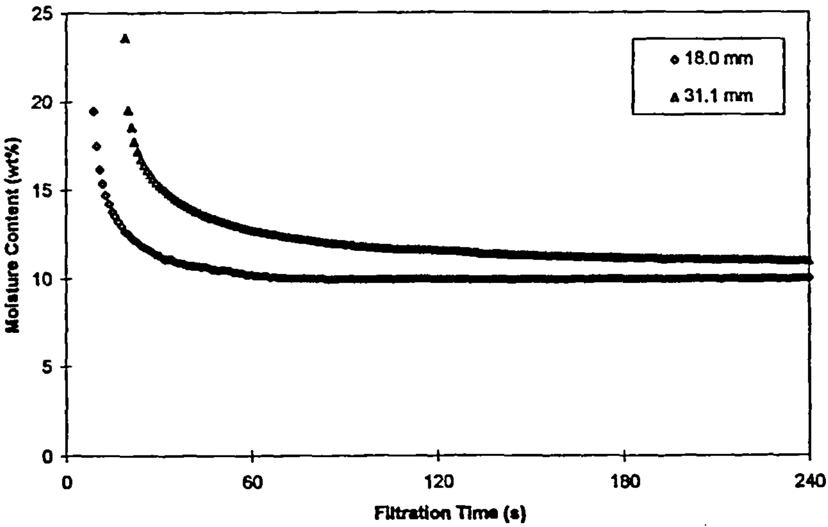

FIG. 7 Effect of cake thickness on kinetics of filtration (12 g/tonne Magnafloc 156).

Effect of Cake Thickness on Desaturation

In this last experiment, cake thickness was increased from 18 to 31 mm (35.6–61.3 kg/m^2). The dose of flocculant (Magnafloc 156) was relatively high (12 g/tonne), and it was chosen to ensure that flocculation would be complete and the observed trends would be solely dependent on the change in cake thickness.

The results (Fig. 7) show, as expected, that the thicker cake dewaters more slowly. The difference in residual moisture after 4 minutes is only 1.0 wt%. However, after the more realistic dewatering time of 60 seconds, the difference in cake moisture is 2.7 wt%, the thinner cake containing 10.1 wt% moisture and the thicker cake 12.8 wt%. By interpolation in Fig. 7, the equivalent filtration times and throughputs at a moisture level of 12.8 wt% are as shown in Table 4. Clearly, in this example the throughput of the filter will be greater when dewatering the thinner cake.

MODELING OF DESATURATION KINETICS

The results described above suggest that there were no basic problems in the vacuum filtration of the particular sample of iron ore under investigation.

TABLE 4
Effect of Cake Thickness on Throughput

Cake thickness, mm (kg/m^2)	Filtration time for 12.8 wt% moisture (seconds)	Equivalent throughput ($kg/m^2/h$)
18 (35.6)	18	7124
31 (61.3)	60	3681

Flocculation with an anionic flocculant was good and tolerant to changes in floc dose. Both cake formation and cake desaturation were rapid, leading to optimum product moisture levels in the region of 10 wt%.

Three diverse cases were investigated for simulating the desaturation phase of the dewatering cycle using Wakeman's model (7, 8) as described below.

Case 1

Case 1 used 10 g/tonne of Magnafloc 156 and corresponded to one of the desaturation curves shown in Fig. 3. The input data for the model, which were either determined experimentally or are material constants, are shown in Table 5. The value of the pore size distribution index of 0.4 was determined from pseudocapillary pressure curves on flocculated fine coal of similar size distribution to the iron ore sample.

The solid/liquid contact angle was assumed to be zero, since it would be reasonable to expect the mixture of iron oxides which comprise the iron ore

TABLE 5
Parameters Used as Input to Wakeman's Model

Input parameter	Symbol	Case 1	Case 2	Case 3
Cake permeability (m^2)	K	2.4×10^{-12}	7.6×10^{-14}	1.3×10^{-13}
Cake porosity (—)	ϵ	0.59	0.56	0.59
Cake thickness (m)	L	0.0182	0.174	0.0196
(kg/m^2)		(36.0)	(34.4)	(38.8)
Cake area (m^2)	A	0.00196	0.00196	0.00196
Solids density (kg/m^3)	ρ_s	4706	4706	4706
Liquid viscosity (Pa·s)	η_l	0.001	0.001	0.001
Liquid surface tension (N/m)	γ	0.072	0.072	0.072
Gas viscosity (Pa·s)	η_a	1.9×10^{-5}	1.9×10^{-5}	1.9×10^{-5}
Solid/liquid contact angle (°)	β	0	0	0
Applied vacuum (kPa)	ΔP	70	70	70
Pore size distribution index	λ	0.4	0.4	0.4

TABLE 6
Derived Parameters for Input into Wakeman's Model

Parameter	Symbol	Case 1	Case 2	Case 3
Breakthrough pressure (kPa)	p_b	2.42	13.2	10.4
Kozeny mean diameter (m × 10^{-6})	d_k	17.2	3.61	4.1
Equilibrium saturation (—)	S_e	0.371	0.586	0.546

to be completely hydrophilic. The values in Table 5 were used to calculate the parameters in Table 6 according to the following equations.

- Breakthrough Pressure:

$$p_b = \frac{\alpha\gamma \cos \beta(1 - \epsilon)}{d_k \epsilon} \tag{3}$$

  where α is a constant and d_k is the Kozeny diameter. A value of α = 0.83 as determined by Condie et al. (10) was used to calculate p_b.
- Kozeny Mean Diameter (d_k):

$$K = \frac{\epsilon^3 d_k^2}{150(1 - \epsilon)^2} \tag{4}$$

- Equilibrium Saturation (S_e) is the filter cake saturation at infinite time for the dewatering conditions being used and was calculated using

$$S_e = S_\infty + (1 - S_\infty)\left(\frac{p_b}{\Delta P}\right)^\lambda \tag{5}$$

  A value of 0.155 was assumed for the irreducible saturation (S_∞) as suggested by Wakeman (7). The value of pore size distribution index (λ) of 0.4 used to calculate S_e was different from the value of λ = 5 used by Wakeman to calculate his curves for interpolating reduced saturation.

A comparison of the experimentally determined desaturation kinetics and those predicted by the Wakeman model based on the above parameters is given in Fig. 8 for Case 1. The fit of the model to the data is very good, with agreement within 1 wt% moisture throughout.

Case 2

The second application of the model was carried out on the data from the filtration experiment performed without flocculant (see Fig. 3). The input filtration data are shown in Tables 5 and 6. The comparison of model and experiment in Fig. 9 shows there is a significant difference between the two

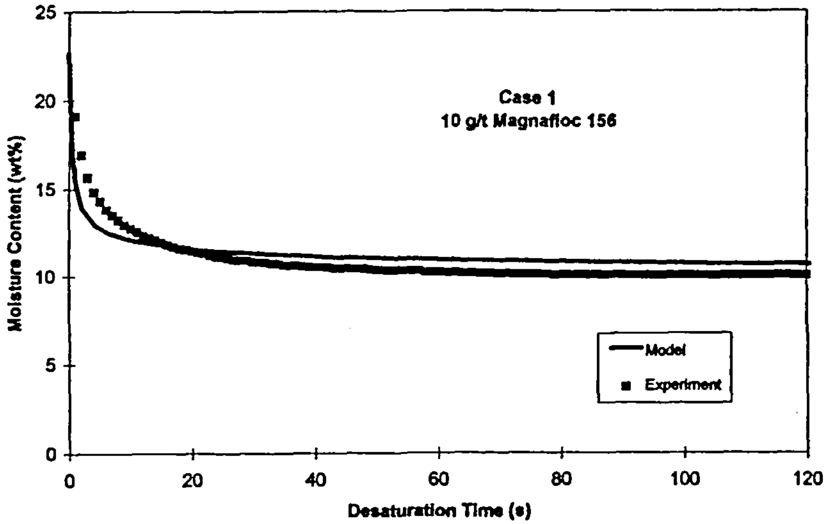

FIG. 8 Comparison of experimental data with desaturation kinetics predicted by Wakeman's model for Case 1.

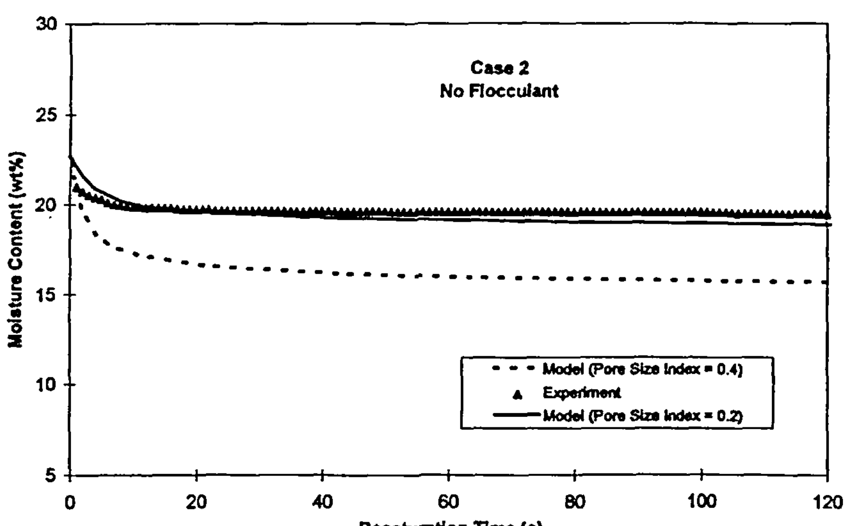

FIG. 9 Comparison of experimental data with desaturation kinetics predicted by Wakeman's model for Case 2.

data sets. This is perhaps not surprising, given that the same value of 0.4 for the pore size distribution index was used for both the flocculated and unflocculated systems. Addition of flocculant is known to exert a large influence on cake structure and hence on pore size distribution. The model prediction using a pore size distribution index of 0.2, which implies a wider spread in the size of the pores within the cake, is in excellent agreement with experiment as shown in Fig. 9.

Case 3

Case 3 involved modeling the addition of 5 g/tonne of Magnafloc 333, the desaturation data for which are given in Fig. 6. The model input data are given in Tables 5 and 6. A comparison of the model prediction and experimental data is given in Fig. 10.

In the first 40 seconds the model gives an underestimate of the product moisture by ca. 1.1–1.7 wt%, although above 60 seconds the agreement between model and experiment is closer. The problem may lie in the value of cake permeability that was used in the simulation because the model is sensitive to cake permeability. Changing permeability from 1.3 to 0.8 $\times$ 10^{-13} m^2 (a value obtained in one of the repeat experiments with this particular flocculant) resulted in a prediction line that is virtually coincident with experiment.

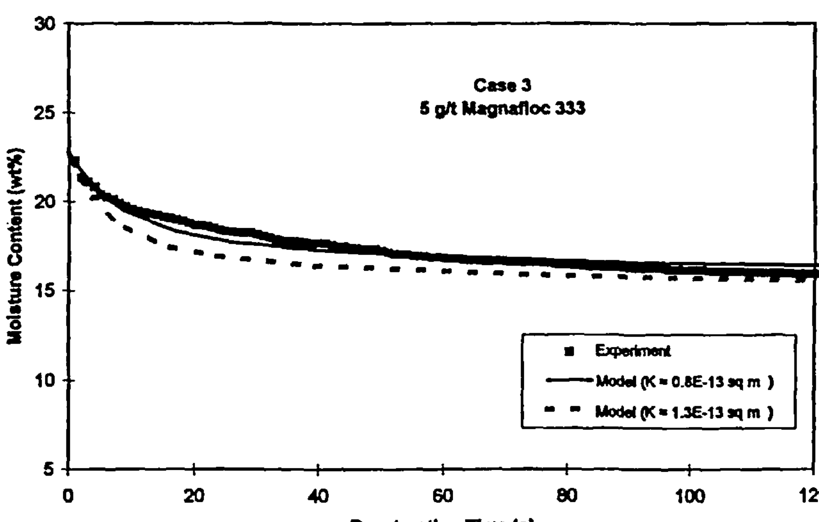

FIG. 10 Comparison of experimental data with desaturation kinetics predicted by Wakeman's model for Case 3.

CONCLUSIONS

1. The test iron ore sample was dewatered by vacuum filtration with no basic problems. Anionic polyacrylamides flocculated the material well at low dose (minimum 5 g/tonne) irrespective of polymer anionicity or molecular weight. The cationic and nonionic polymers tested were much less effective.

2. The anionic flocculant reduced cake formation time from 60 seconds without reagent) to below 10 seconds. In the bench-scale batch cell, product moisture of 10–11 wt% was obtained with a projected throughput, based on cake thickness and dewatering time, of 3200 $kg/m^2/h$.

3. Wakeman's model showed encouraging results in its ability to predict the kinetics of desaturation for the flocculated cakes, using values of breakthrough pressure and pore size distribution index derived from the vacuum filtration of coal fines, similar in size distribution to the iron ore. Further data, derived from a wider spread of cake properties, are required in order to confirm these results.

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