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THE PREDICTION OF PROCESS SCALE VACUUM FILTER CYCLES FROM LABORATORY SCALE TESTS

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ABSTRACT
This paper presents filtration data from a series of laboratory scale tests which quantify both the filtration and post-treatment characteristics of an industrial catalyst slurry. A computer automated and well controlled apparatus was used to acquire data for filter cycles comprising combinations of filtration, displacement washing and gas deliquoring. The influence of a matrix of parameters such as applied pressure gradient and cake thickness was examined to establish the scale-up parameters required for use in process simulations. Scale-up predictions from these simulations, developed as a part of another research programme, are described and compared with experimentally measured filtration data from an in-service process scale rotary vacuum filter (RVF). It was found that the simulations provided almost exact predictions of filtrate volume and mass of solids in the cake, along with estimates within 25% of the measured cake height for the experimental data obtained at both the laboratory and full scale.

INTRODUCTION
There currently exists relatively little research literature on the industrially important topic of scale-up in filtration processes. Whilst previous works provide some qualitative assessments and others provide a semi-mathematical basis to scale-up, it is evident that there is an over reliance on heuristics (or rules-of-thumb). Prediction of process scale filter performance from laboratory scale experiments has generally utilised relatively simple laboratory equipment. For example, a Buchner funnel can give an estimation of filter cake formation times and how well, or badly, a suspension will filter. Such techniques, however, will only provide qualitative notions of cake formation and not definitive analytical data. Due to the limited knowledge of scale-up from laboratory to process-scale, safety factors are generally included within the developed models, whereby a predicted filtration area is effectively increased by 25% or even up to 50%.

In order to facilitate a move away from scale-up heuristics a fully automated laboratory scale apparatus has been developed. This apparatus, which minimises operator interference, has been used to filter, wash, and deliquor an industrial catalyst suspension with SCAPA Primapor (vacuum grade) filter media. Comparison of the data obtained has been made with process scale filtration data.

EXPERIMENTAL APPARATUS AND PROCEDURES
The laboratory scale apparatus used in the investigation is shown in Figure 1. It is capable of performing the desired stages of the filter cycle – filtration, washing and deliquoring – without interruption, and in any desired order and number of stages. The apparatus has been previously described in detail and essentially comprised of a deadend, variable height stainless steel filter cell, with a filtration area of 0.013 m² and two feed vessels connected by automated valving and appropriate computer interface devices.

Characterisation of the aqueous catalyst feed suspension indicated a wide particle size distribution (0.1-250 μm), with noticeable peaks at 0.5, 6 and 40 μm as measured with a Malvern Mastersizer. Measurement of zeta (ζ-) potential indicated the iso-electric point (IEP) to be in the region of pH = 7 at ambient temperature and pH = 5 at 70°C, the experimental operating temperature. The filter
The medium used throughout laboratory testing was SCAPA Primapor (vacuum grade) with material thickness 1.25 mm, an estimated porosity of 4.7% and a permeability of 2.7x10^-13 m². Porosimetry tests indicated a mean flow pore size of ~4.5 μm.

Variables such as pressure gradient (25-300 kPa), feed concentration (15-25% w/w suspended solids), ζ-potential, temperature and filter cell orientation were assessed with the intention that this series of experiments reflected, as closely as possible, actual full-scale plant operation. Process-scale tests on a 10.8 m² RVF also yielded filter cycle data.

MATHEMATICAL MODELING

A process model has been developed and utilised for the laboratory scale experiments. In essence the simulation was based on three existing models; one for each phase of the filter cycle. Focusing on the filtration phase, the model used classical filtration theory in an attempt to take into effect cake compressibility, the principal equations being:

\[
t_f = \frac{\alpha_{av} \mu \rho_0 M_s}{2A^2 \Delta P \left(1 - M_s \left(1 + e_{av} \left(\rho / \rho_s\right)\right)\right)} V_f^2 + \frac{\mu R_m}{A \Delta P} V_f
\]

\[
L = \frac{V_f \left(1 + e_{av}\right)}{A \left(\frac{\rho_f}{\rho_0} \left(\frac{1}{M_s} - 1\right) - e_{av}\right)}
\]

\[
M_c = \frac{Ah \rho_s}{1 + e_{av}}
\]

where \(t_f\) is the filtration time, \(\alpha_{av}\) the average specific cake resistance, \(e_{av}\) the average voids ratio, \(\mu\) is the fluid viscosity, \(A\) the filtration area, \(\Delta P\) the pressure difference, \(V_f\) the volume of filtrate, \(R_m\) the medium resistance, \(\rho\) the filtrate density, \(\rho_s\) the solids density, \(M_s\) the solids mass fraction in the feed, \(L\) the cake thickness and \(M_c\) the mass of solids in the cake.

The model for process-scale filtration on an RVF utilises the expressions:

\[
V_f = \frac{A}{c} \left(\frac{\Delta P}{\alpha_{av}} + \sqrt{\frac{R_m}{\alpha_{av}}^2 + \frac{2c \varphi_f \Delta P}{\omega \mu \alpha_{av}}}\right)
\]

\[
L_f = \frac{1}{\rho_s (1 - \varepsilon_{av})} \left(\frac{R_m}{\alpha_{av}} + \sqrt{\frac{R_m}{\alpha_{av}}^2 + \frac{2c \varphi_f \Delta P}{\omega \mu \alpha_{av}}}\right)
\]

where \(c\) is the effective feed solid concentration, \(\varepsilon_{av}\) the average porosity, \(\varphi_f\) the fraction of filter surface devoted to the filtration phase and \(\omega\) the angular velocity.

RESULTS AND DISCUSSION

Laboratory scale experimental data was generated for nominal cake thickness' of 5, 10 and 20 mm. The results presented are for the 10 mm filter cakes, which are also representative of the other data. All experiments comprised filtration, displacement washing and air deliquoring stages.
(in that order) and were carried out over the pressure range 25-300 kPa, at pH = 7.8. Only results for the cake formation phase of the filter cycle are presented and compared with the theoretical predictions provided by equations (1)-(5). Figure 2 shows some typical results for the cake formation stage where experimental data are compared with the theoretical predictions obtained from equation (1).

An important parameter within the model is \( n \), the experimentally determined compressibility index of the catalyst material, which has an empirically determined value of 0.53. This suggests material moderate compressibility. Over the range of applied pressures (25-300 kPa), \( \alpha_{av} \) varied between \( 1.51 \times 10^{11} \) and \( 4.17 \times 10^{11} \) m kg\(^{-1} \) and \( \epsilon_{av} \) between 2.01-1.21 to give scale-up constants \( \alpha_{o} \), the specific cake resistance at unit applied pressure, \( \epsilon_{o} \), the voids ratio at unit applied pressure and \( b_{1} \), an empirical constant, as calculated from:

\[
\alpha_{av} = \alpha_{o} (1 - n) \Delta P^n
\]  
(6)

\[
\epsilon_{av} = \epsilon_{o} - b_{1} \log(\Delta P)
\]  
(7)

Predictions using equations (2), (3), (6) and (7) have been compared with laboratory data and these are shown in Table 1. The two sample experiments (at 100 and 300 kPa) which did not involve a deliquoring phase, allowed actual cake heights to be measured directly. Predictions of process scale RVF performance was carried out using equations (4) and (5) on the basis of filtrate volume and cake thickness and sample data are shown in Table 2.

CONCLUSION

The procedures outlined in this paper highlight the benefits of using well controlled laboratory scale apparatus to collect scale-up data. These facilitate the process modelling of larger scale filters and the accurate prediction of parameters such as cake mass and cumulative filtrate volume\(^6\). The philosophy also lays the foundations to provide more accurate scale-up models, not just for the cake formation phase of the filter cycle, but also, for subsequent combinations of cake washing and deliquoring phases.

REFERENCES


ACKNOWLEDGEMENTS

The authors gratefully acknowledge the financial support of the EPSRC for this work.

TABLES AND FIGURES

\[ \Delta P \text{ (kPa)} \quad \text{Cake thickness (mm)} \quad \text{Mass of solids in cake (g)} \]
<table>
<thead>
<tr>
<th></th>
<th>Measured</th>
<th>Predicted</th>
<th>Measured</th>
<th>Predicted</th>
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<td>11</td>
<td>11.6</td>
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Table 1: Comparisons of laboratory scale experimental data with theoretical predictions.

<table>
<thead>
<tr>
<th>RVF experiment</th>
<th>Submergence (m)</th>
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<th>Cake thickness (mm)</th>
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</tr>
<tr>
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<td>0.61</td>
<td>0.015</td>
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</tr>
</tbody>
</table>

Table 2: Comparison of process scale RVF data with theoretical predictions (\( \Delta P = -0.5 \text{ bar} \)).

Figure 1: Schematic diagram of experimental apparatus.
Figure 2: Theoretical predictions and experimental data for constant pressure laboratory scale filtrations of catalyst suspensions.