Cake filter scale-up, simulation and data acquisition - a new approach

CAKE FILTER SCALE-UP, SIMULATION AND DATA ACQUISITION – A NEW APPROACH

E.S. Tarleton (e.s.tarleton@lboro.ac.uk)
Advanced Separation Technologies Group, Department of Chemical Engineering, Loughborough University, Loughborough, Leics., LE11 3TU, UK.

ABSTRACT

This paper details the capability of a unique, automated filtration apparatus and the newly developed Filter Design Software (FDS) which facilitate equipment selection, scale-up and simulation through an integrated experimental and theoretical approach.

By way of example, experimental data were obtained with the apparatus over constant, variable and stepped pressure regimes. Inherent suspension properties were maintained throughout by utilising a computer controlled pressure controller and cake formation was monitored by micro-pressure transducers capable of providing up to seven independent measures of liquid pressure within 3.3 mm of the filter medium surface. For constant pressure and moderately compressible talc cakes the liquid pressure increased with cake height in a non-linear manner and generally exhibited a concave profile. When a pressure step was applied following a period of constant pressure filtration, the cake structure typically required up to 30 s to reach a new pseudo-equilibrium state. During this time the reciprocal filtrate flow rate vs. filtrate volume plot was non-linear and the liquid pressures in the cake increased rapidly before remaining nearly constant. When the cake was thicker or the pressure step larger, the liquid pressure measured closer to the filter medium remained either constant following the increase in pressure or increased slowly over the 360 s duration of the pressure step which indicates potential difficulties with the stepped pressure test.

The filtration data were analysed using FDS to obtain scale-up coefficients and the impact of using incorrect scale-up coefficients on likely filter performance at the process scale is shown. The simulation capabilities of FDS are also highlighted through a case study in which, by way of example, the influence of crystal formation and other operating parameters on the filter cycle for a pharmaceutical product are shown. Simulations quantify how crystal form can detrimentally influence all phases of a cycle and lead to, for instance, slower filtration and wetter filter cakes.

KEYWORDS

Filtration, transducers, scale-up, simulation, stepped pressure, mechatronics, Filter Design Software.

INTRODUCTION

Basic filtration data can be obtained in laboratories with relatively elementary equipment, an example being the single leaf filter operating at either constant under- or over- pressure. In some cases it is possible to collect data for sequential operations such as those constituting a filter cycle, though performing these experiments manually gives limited information and can introduce significant errors unless great care is taken. The need to manually perform tests and adjust operational parameters to maintain chosen experimental conditions are variables that lead to the use of ‘rules-of-thumb’ by filter manufacturers.

The development of more sophisticated filtration apparatus has facilitated both improved test methodologies and more detailed measurements [Fathi-Najafi, 1994; Green *et al.*, 1998; Jämsä-Jounela and Oja, 2000; Teoh *et al.*, 2001; Usher *et al.*, 2001; Johansson and Theliander, 2002;

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

Townsend, 2002; Anderson *et al.*, 2004]. In the case of the author, manual operation has been replaced by a mechatronics philosophy that combines electronics, computer technology and automatic control to facilitate experiments over any chosen pressure/flow regime without altering inherent suspension properties [Tarleton and Hancock, 1997; Tarleton, 1998; Tarleton, 1999a; Tarleton and Wakeman, 2006]. Raised levels of sophistication introduce new research opportunities, allow more consistent and accurate data to be obtained and provide an impetus for further developments.

Filtration equipment is rarely specified without recourse to extensive laboratory and pilot scale tests. However, the lack of a consistent approach can lead to the poor specification and sizing of filters with the result that required production rates may not always be achieved and unforeseen difficulties arise in filter cycle operations. Against such a background the author has developed a combined theoretical and experimental approach to the use of computer software in filter specification (as outlined in Figure 1) and produced the Windows® software, Filter Design Software (FDS, 2005) which comprises a series of independent, but interlinked modules whose capabilities include:

- A catalogue and explanation of the main operational and design features of 70+ equipment types coupled with a procedure for ranked equipment selection
- Full analysis capabilities for leaf filter, jar sedimentation and expression test results to give the parameters required for scale-up and simulation of solid/liquid separation equipment
- Comparison of data sets from a range of tests or trials
- Simulation of 20+ types of vacuum and pressure filters to give filter size and performance parameters for filter cycles involving cake formation at both fixed and variable pressure/flow conditions, compression and gas deliquoring as well as displacement washing. The vacuum filter module allows for the simulation of Nutsche, multi-element leaf, belt, drum, disc, table and tilting pan filters. The pressure filter module is able to simulate single and multi element leaf filters, diaphragm and filter presses as well as the tube press.
- The ability to educate the user in filtration technology
- Internet access to equipment suppliers.

This paper presents some examples of the capability of the automated filtration apparatus and the Filter Design Software and highlights some of the benefits of combining the two.

EXPERIMENTAL

Figure 2 shows the principal components of the laboratory scale pressure filtration apparatus which comprised a stainless steel (s/s) leaf filter fitted with a planar medium of 120 cm^2 area. A s/s suspension storage vessel was connected by s/s piping and computer controlled electro-pneumatic valving. Transducers attached to the apparatus allowed the filtration pressure(s) and other parameters to be recorded and/or controlled by the interfaced computer and dedicated software.

To monitor the transient properties of a forming cake, 15 diametrically opposed pairs of s/s electrodes were fitted internally within the filter cell. These were positioned at 1 mm vertical intervals above the filter medium and protruded *circa* 2 mm from the cell walls; previous work has shown that such intrusions cause negligible effects on cake formation [Tarleton, 1999b]. Signals to

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

each electrode pair were switched by the computer via electronic circuitry to give electrical resistance measurements of changing cake thickness and solids concentration.

Also positioned within the filter cell were ten titanium micro-pressure transducers attached to bespoke s/s holders and micro-bore tubes (see Figure 3). Each tube protruded *circa* 2 mm into the cell with the centre of the closest tube being 0.5 mm above the filter medium. The first seven transducers were positioned within 3.3 mm of the filter medium. A liquid bridge was created between the cake and the tip of a transducer by injecting water from a reservoir into each holder/micro-bore tube unit. The transducers were capable of monitoring the transient liquid pressures generated in a forming cake/filtering suspension and were placed in a spiral arrangement around the cell periphery. The response time of the transducers was << 0.1 s.

The pressure required to progress a downward filtration was provided by compressed air and an electronic pressure regulator up to 600 kPa. The regulator was adjusted by an electrical control signal from the computer and had a manufacturer quoted response time of < 1 s. The filtrate flow rate was determined semi-continuously via successive timed readings of mass from an electronic balance. A fixed (constant) pressure filtration required a constant control signal to the regulator. By monitoring flow rate, use of a closed loop proportional or integral-proportional negative feedback control algorithm facilitated variable pressure filtration without changing suspension properties. Sequentially incremented control signals, and hence constant pressures, allowed stepped pressure experiments to be performed such that different pressure levels were applied for set time intervals during a test.

5 %v/v suspensions of talc in distilled water were used in most experiments and Table 1 shows some basic properties; calcite suspensions were also used in a few experiments. A Gelman Versapor 0.2 μm rated membrane provided the filter medium and a new membrane was used in each experiment. In all tests the filtrate was visually clear.

EXPERIMENT RESULTS AND DISCUSSION

The experimental data presented in this paper are typical for the filtration apparatus and Figures 4 and 5 show repeatability data specific to the current work. The measured volume of filtrate (V) vs. time (t) data were reproducible and for the four experiments shown the specific cake resistance, filter medium resistance and cake porosity were respectively determined as $1.46 \times 10^{11} \pm 2 \times 10^{9}$ m kg^{-1}, $3.7 \times 10^{11} \pm 1 \times 10^{10}$ m^{-1} and 0.67±0.005. There was slightly more, but still acceptable, variation in the measured liquid pressure profiles as evidenced by Figure 5.

Constant Pressure Experiments

Typical liquid pressure profiles and time histories measured with the micro-pressure transducers are shown in Figures 6 and 7. At the start of filtration (t = 0 s) the talc suspension was in its original homogenously mixed state and measured liquid pressures were equal to the applied filtration pressure. Here, the particles in suspension were sufficiently far apart to carry zero compressive pressure. For a 400 kPa filtration pressure, after 10 s a cake of just over 2 mm had formed. At this stage many of the particles comprising the cake were (at least) in point contact to raise the solids compressive pressure and induce a corresponding reduction in liquid pressure. Near to the filter medium the solids pressure was at its greatest, as was the local cake resistance and solidosity, and the particles were also closest together. Further away from the medium the solids pressure was lower and at the ultimate cake height equal to zero. As time progressed in the filtration, cake thickness increased and the moderately compressible talc cake became more compact due to the continual flow of liquid through the cake interstices and the increasing weight of particles above those already constituting the cake. Towards the end of cake formation (t = 125 s) the cake thickness approached 9.3 mm as evidenced by the falling liquid pressure at that height.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

Over the 100-600 kPa pressure range, the measured liquid pressure profiles for talc were similar in form and of concave appearance between the filter medium surface and the top of the cake. Figure 8 shows profile comparisons for talc were the applied filtration pressure has been used to normalise the values measured by the micro-pressure transducers. For the same filtration time, data at the higher pressure were generally displaced above and to the left of those obtained at the lower pressure indicating a thicker and more compact cake. Such behaviour is to be expected for talc/water filtrations as the higher pressure forms a cake more rapidly and closer packing arises from the increased fluid drag. If liquid pressure profiles at the same cake thickness, rather than time, were compared then the profiles from low and high pressure experiments were more similar. This is comparable with earlier research which suggests that a plot of normalised cake height vs. liquid pressure is independent of flow conditions and total cake thickness [Shirato, 1987].

Stepped Pressure Experiments

An example of stepped pressure data and comparisons with constant pressure data over the same pressure range are shown in Figure 9. At 100 kPa, the step and constant pressure data compare favourably as would be expected from repeat tests. After 360 s, the pressure in the step test was raised automatically to 200 kPa and filtration continued through the cake formed in the previous period at 100 kPa. Immediately following the pressure step there was (typically) a 30 s time delay before the measurements of filtrate mass indicated a return to the expected linear behaviour on the dt/dV vs. V plot. In some cases, such as Figure 9, the y-axis intercept (used in the calculation of filter medium resistance) was displaced following the pressure step. The available data suggest that this displacement is not important in the calculation of specific cake resistance when cakes are relatively thin and pressure steps are relatively small.

Figure 10 shows the liquid pressure profiles measured from the onset of the pressure step. At the end of the 100 kPa filtration period (i.e. t = 0 s), the liquid pressure 0.5 mm above the filter medium was 27 kPa whilst that at 15.3 mm was ~100 kPa. The complete dataset indicated a cake thickness between 9.3 and 15.3 mm. When the pressure was raised to 200 kPa, the liquid pressure measured at 0.5 mm increased to 46 kPa within the first 15 s sampling period and stayed at that level for a further 15 s before progressively reducing during the remainder of the filtration. Measurements further up the cake exhibited a different behaviour where liquid pressures rapidly increased following the step and then continually decreased. If the pressure increment was larger and/or the formed cake was thicker prior to the pressure step, the effects observed very close to the filter medium were more pronounced. The data suggest that compression of cake layers can be retarded closer to the medium and that a sudden increase in driving pressure is not always able to immediately and fully transmit through a previously formed cake.

Figure 11 shows measurements taken during a 100→300→500 kPa stepped pressure test where 200 kPa increments were invoked every 360 s. Following the first pressure increment, the liquid pressure recorded at 0.5 mm above the filter medium increased from 26 kPa to nearly 52 kPa within the initial 15 s period, increased further to ~57 kPa over the next 60 s and then remained constant for the remaining 285 s of the 300 kPa period. Following the final pressure increment from 300→500 kPa, the liquid pressure increased continuously at the 0.5 mm height until the end of the experiment. Similar behaviour near to the filter medium was observed for 100→400 and 100→600 kPa step pressure experiments with talc suspensions.

Implications

The stepped pressure test was originally conceived to reduce the time required to obtain data for filtration scale-up and enable the calculation of the scale-up constants that characterise cake resistance [Murase, 1989]. In Figure 9, for instance, it is evident that values of specific cake resistance calculated from the individual constant pressure tests and the corresponding portions of the stepped pressure test will give near identical values. However, if more pressure increments are applied within the same test to extend the range, the tendency toward lower and more difficult

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

to measure flow rates at longer times leads to more data scatter. It follows that the determination of accurate gradients and hence specific cake resistances becomes more difficult.

Under constant pressure conditions, the duration of a filtration tends to generate a relatively large amount of data over the linear region of a *dt/dV* vs. *V* or *t/V* vs. *V* plot. For the stepped pressure regime, the amount of data available within each constant pressure phase is more restricted and so (potentially) is the accuracy of the data manipulation. Experiments suggest that each constant pressure period must be sufficiently long to establish pseudo-equilibrium cake conditions - this is evident from Table 2 where the scale-up for the 50 kPa step experiment differs significantly from the other calculated values.

When compressibility is present in a forming cake the rate of change in pressure gradient across the cake affects the manner in which the constituent particles are initially arranged and subsequently rearranged, and this in turn influences both the cake resistance and porosity. Much of the recent, and indeed past, filtration work has found difficulty in quantifying these differences and several modelling approaches assume that layers of forming cake instantaneously achieve an equilibrium solids concentration rather than undergoing some form of dynamic consolidation. The assumption is undesirable and incorrect given the nature of the data presented in this paper for a cake undergoing pressure changes.

In Figure 12 the scale-up constants from Table 2 for individual constant pressure experiments have been used in combination with FDS to provide predictions of volume of filtrate vs. time obtained from a laboratory scale pressure Nutsche filter; values for cake solids concentration were interpreted through electrode pair measurements. The predictions are good in comparison with the experimental data for both suspension types tested which highlights a benefit of integrating the automated experimental apparatus with computer software for data analysis and simulation. It is clear that the use of incorrectly determined scale-up coefficients will lead to erroneous filter specification. For the talc data presented in Table 2, the stepped pressure tests lead to under-predictions of cake resistance (α_{av}) of 21% (200 kPa increment every 480 s) and 36% (50 kPa increment every 240 s) compared to the value obtained from a sequence of individual constant pressure experiments. The implication is that a process scale filter could be significantly undersized and/or cycle times underestimated, either of which will lead to a process ‘bottleneck’.

CASE STUDY

To further illustrate the simulation capabilities of FDS the following case study is presented. Whilst the study is based on a real situation some aspects have been amended for reasons of confidentiality. However, the essence of the study remains intact.

Simulation Using FDS - General

Figure 13 shows an example screen display for the simulation of a pressure Nutsche filter. The ‘General Information’ box toward the top left hand corner of the display is used to start a simulation procedure. The cycle configuration is defined here. For many filters this will comprise combinations of cake formation, washing and gas deliquoring whilst variable volume filters also incorporate a cake compression phase. The Unit file allows the user to specify their preferred units for data entry and the default washing model can be over-ridden by the specification of an experimentally measured wash curve; although not required for a Nutsche filter, a pump curve can also be defined to facilitate the simulation of variable pressure filtration.

The remainder of the information required for simulation is typed by the user in the ‘Simulation Data’ box toward the top right hand corner of the display. Each ‘tab’ corresponds to a phase in the filter cycle or provides facility to enter data specific to the filter or the feed solids, liquid and solute. The results of a simulation are shown towards the bottom of the display. More detailed

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

descriptions of FDS are provided in Tarleton and Wakeman (2006); see also Wakeman and Tarleton (2005a; 2005b).

Problem

A pressure driven Nutsche filter (selected via use of FDS) is to be used to separate batches of a crystalline pharmaceutical product from a propanol based suspension. Variations in upstream formulation mean that crystallisation of the *β*-form, which is more difficult to filter, can occur in place of the *α*-form. In each batch, 50 kg of solids are present at a concentration of 6 %v/v and it is envisaged that cake formation will occur to a maximum depth of 50 mm. In order to meet product specifications this new filter installation requires a sequential cycle comprising filtration, displacement washing and gas deliquoring. Preliminary tests in the laboratory suggest that the cake formed in each cycle needs to be treated with 3.5 wash ratios of pure propanol to remove unwanted solute residues after which deliquoring (with pressurised nitrogen) proceeds for 1500 s to dry the cake ready for discharge with the plough.

The cake characteristics for both the *α* and *β* particle forms in suspension have been determined experimentally and analysed using FDS. These are shown in Table 3 along with other suggested operational parameters.

For the *α*-form, determine the required filter area, the solid, liquid and solute throughput rates, the filter cycle time and other performance indicators. Assess the impact on the filter cycle if *β*-form crystallisation occurs.

Solution

Repeated use of the simulation module in FDS facilitates a solution to the problem. With the *α*-form of crystal, the required filter area is 2 m^2 for the specified 50 kg of solids per batch and 50 mm cake thickness. Each cake discharged from the Nutsche contains ~16.6 kg of propanol and (theoretically) no undesirable solutes. A total of 637 kg of propanol passes through the filter per batch, including 178 kg of wash liquid and 5.14 kg of solutes are removed with the filtrate (4.57 kg) and washings (0.57 kg). As shown in the penultimate row of Table 4, the total cycle time is 2775 s.

With reference to Figure 14 and Table 3, the *β*-form of crystal is more acicular (needle-like) and forms a cake of higher compressibility as evidenced by the constitutive equations for cake resistance and solids volume fraction. If a sequence of calculations are performed for the *β*-form with the 2 m^2 Nutsche then the results summarised in Table 4 are obtained. Due to different intrinsic properties, a cake containing 50 kg of solids exhibits a thickness of 47.4 mm rather than the 50 mm observed with the *α*-form. The approximate four fold increase in specific cake resistance with the *β*-form more than doubles the total cycle time and leads to a significantly wetter cake at the end of deliquoring (i.e. 28.6 % compared with 24.9 % for the *α*-form). To achieve a 24.9 % moisture content would require either a deliquoring time of ~4300 s at the original 200 kPa pressure or a raised deliquoring pressure of 480 kPa applied for the specified 1500 s. The implications of processing the *β*-form of particle are significant in terms of either longer cycle times and/or raised equipment specification.

It is evident that a reduced maximum cake thickness would lead to reduced filtration and deliquoring times, albeit at the expense of a larger filter area and the potential limitation of increased channelling (during washing) with excessively thin cakes. Conversely a thicker cake would lead to a smaller filter but longer processing times.

CONCLUSIONS

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

The information and methodologies presented in this paper highlight how automated and well instrumented experimental apparatus can be used to provide for better and more versatile filtrations as well as improved measurements. It has been shown how experiments can be performed in a consistent manner over a range of pressure/flow regimes through software control. Moreover, both individual, stepped and variable pressure filtrations can be performed without altering inherent suspension properties. The experimental data presented suggest that liquid pressure profiles can be successfully measured for the thin filter cakes of most interest to industry. The unexpected results for stepped pressure experiments may cast doubts over the applicability of the technique to filter scale-up.

The removal of many uncertainties in experimental filtration and data analysis forms the basis from which filtration research can progress and points to the development of a new filtration philosophy that integrates a package for filter selection, design, scale-up and simulation (i.e. Filter Design Software) with experimental data. Use of the philosophy could prevent the implementation of equipment whose actual performance falls below anticipated operating demands, and certainly enables the user engineer to perform independent checks on equipment manufacturers design and performance claims. There is potential widespread application of the underlying methodology within the process and related industries.

REFERENCES

Andersen, N.P.R., M.L. Christensen and K. Keiding, “New Approach to Determining Consolidation Coefficients Using Cake-Filtration Experiments”, *Powder Technology*, **142**, 98-102 (2004).

Fathi-Najafi, M., “Cake Filtration – A Study of Non-reversible Compressible Materials”, *PhD Thesis*, Chalmers University of Technology, Göteborg, Sweden (1994).

Filter Design Software, 2005. Filtration Solutions, UK. (www.filtrationsolutions.co.uk).

Green, M.D., K.A. Landman, R.G. de Kretser and D.V. Boger, “Pressure Filtration Technique for Complete Characterisation of Consolidating Suspensions”, *Industrial Engineering Chemistry Research*, **37**, 4152 (1998).

Jämsä-Jounela, S.L. and M. Oja, “Modelling Module of the Intelligent Control System for the Variable Volume Pressure Filter”, *Filtration and Separation*, **37**(2), 39 (2000).

Johansson, C. and H. Theliander, “Measuring Concentration and Pressure Profiles in Dead-end Filtration”, *Proc. European Conference on Filtration and Separation*, pp.156-164, Nordic Filtration Society, Gothenburg, Sweden (2002).

Murase, T., E. Iritani, J-H. Cho and M. Shirato, “Determination of Filtration Characteristics Based Upon Filtration Tests Under Step-up Pressure Conditions”, *Journal Chemical Engineering of Japan*, **22**(4), 373 (1989).

Shirato, M., T. Murase, E. Iritani, F.M. Tiller and A.F. Alciatore, in *Filtration: Principles and Practices*, Eds. M.J. Matterson and C. Orr, pp.348-351, Marcel Dekker, New York (1987).
Tarleton, E.S., “A New Approach to Variable Pressure Cake Filtration”, *Minerals Engineering*, **11**(1), 53 (1998).

Tarleton, E.S., “Using Mechatronics Technology to Assess Pressure Filtration”, *Powder Technology*, **104**, 121 (1999a).

Tarleton, E.S., “The Use of Electrode Probes in Determinations of Filter Cake Formation and Batch Filter Scale-up”, *Minerals Engineering*, **12**(10), 1263 (1999b).

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

Tarleton, E.S. and D.L. Hancock, “Using Mechatronics for the Interpretation and Modelling of the Pressure Filter Cycle”, *Transactions Institution of Chemical Engineers*, **75**(A), 298 (1997).

Tarleton, E.S. and R.J. Wakeman, *Solid/Liquid Separation: Equipment Selection and Process Design*, Elsevier, Oxford (2006).

Teoh, S.K., R.B.H. Tan, D. He and C. Tien, “A Multifunction Test Cell for Cake Filtration Studies”, *Transactions Filtration Society*, **1**(3), 61 (2001).

Townsend, I., “Pressure Filtration”, Paper presented at conference “*Solid/Liquid Separation Plant Design*”, 13 pages, The Filtration Society, Birmingham (2002).

Usher, S.P., R.G. de Kretser and P.J. Scales, “Validation of a New Filtration Technique for Dewaterability Characterisation”, *American Institute Chemical Engineers Journal*, **47**(7), 1561 (2001).

Wakeman, R.J. and E.S. Tarleton, *Solid/Liquid Separation: Principles of Industrial Filtration*, Elsevier, Oxford (2005a).

Wakeman R.J. and Tarleton E.S., *Solid/Liquid Separation: Scale-up of Industrial Equipment*, Elsevier, Oxford (2005b).

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

FIGURES AND TABLES

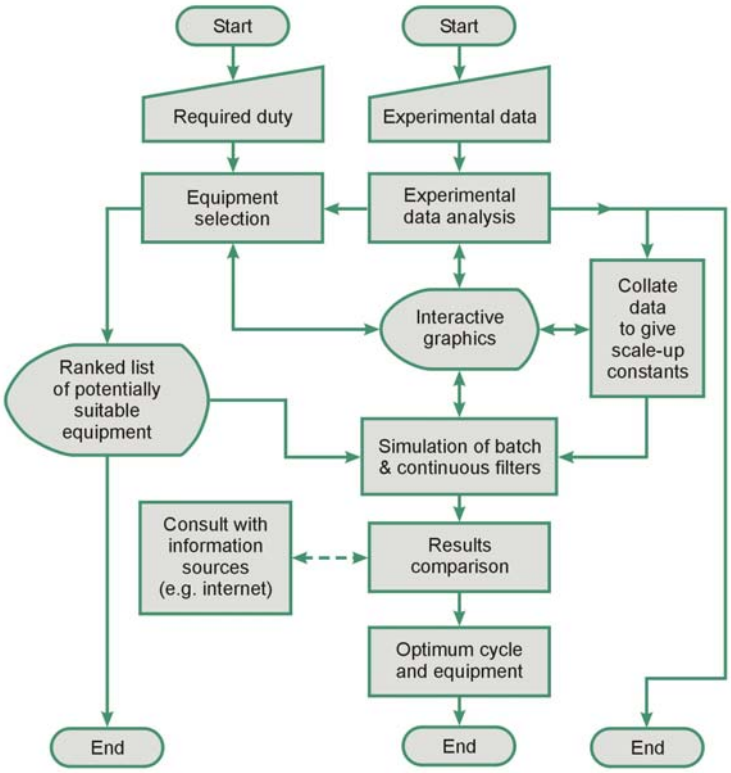

Figure 1: Flowsheet showing the integration of selection, analysis, scale-up and simulation in the Filter Design Software.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

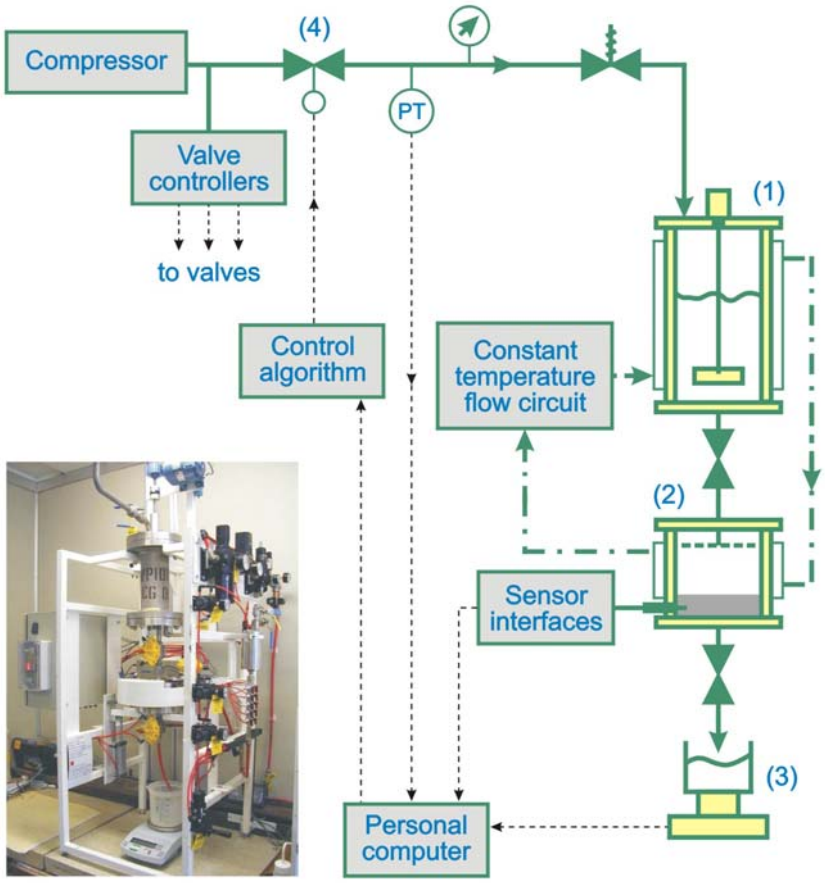

Figure 2: Schematic and inset photograph of the experimental apparatus. (1) suspension feed vessel; (2) filter cell; (3) electronic balance; (4) pressure regulator.

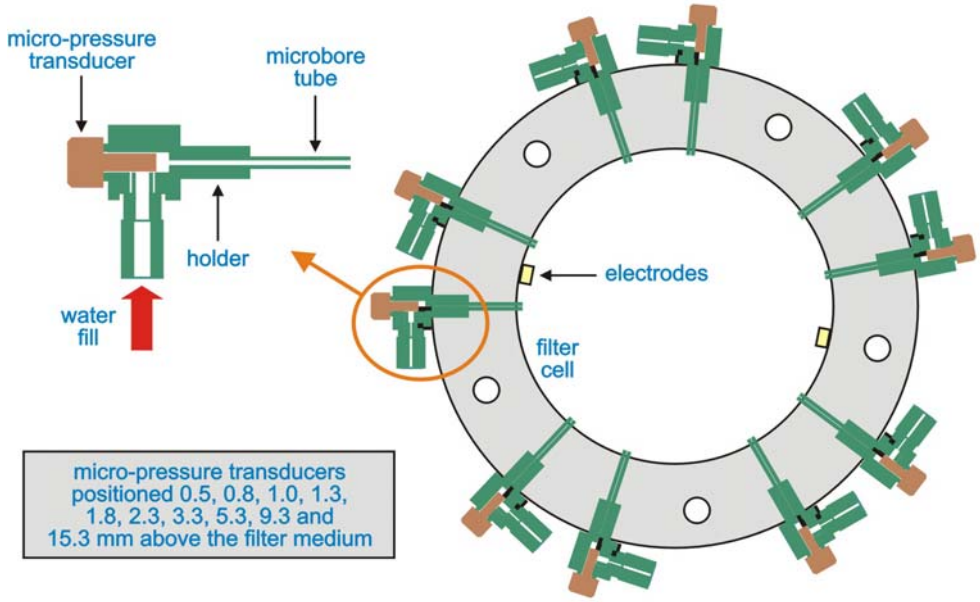

Figure 3: Schematic top view of the filter cell showing the micro-pressure transducer arrangement.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

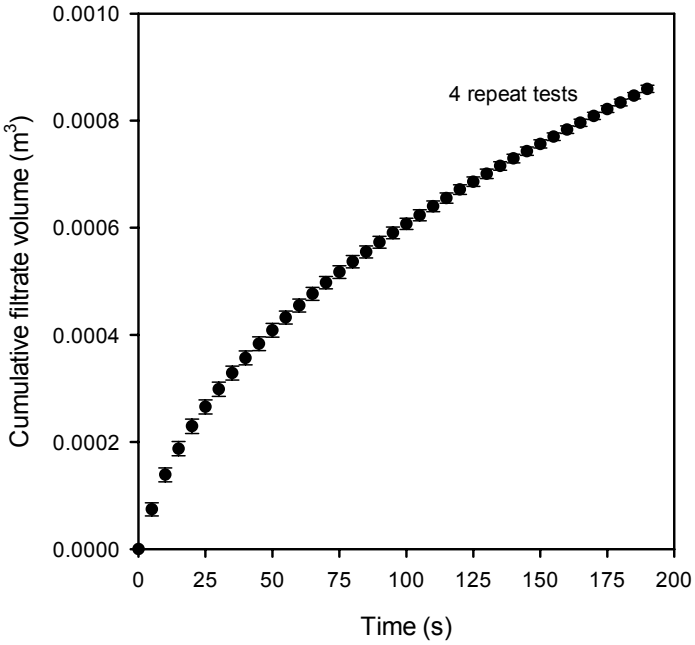

Figure 4: Repeat volume vs. time data for talc at constant filtration pressures of 400 kPa.

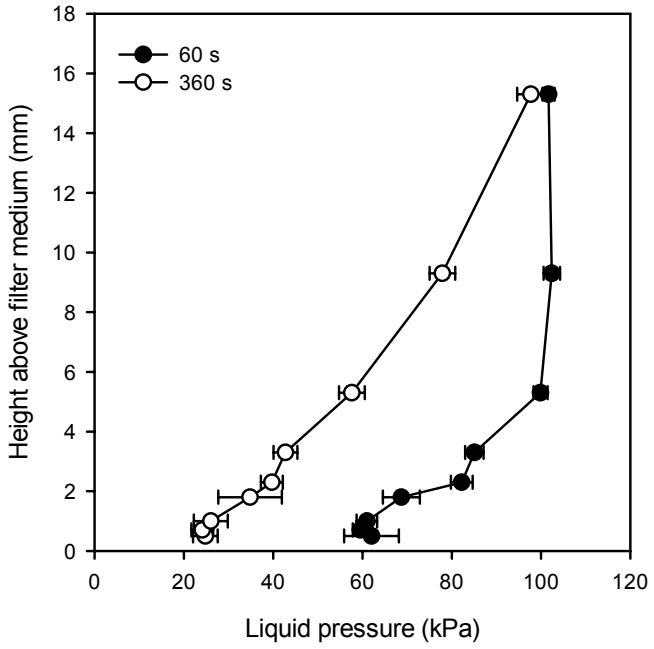

Figure 5: Repeat experiments showing liquid pressure profiles for talc at constant filtration pressures of 100 kPa.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

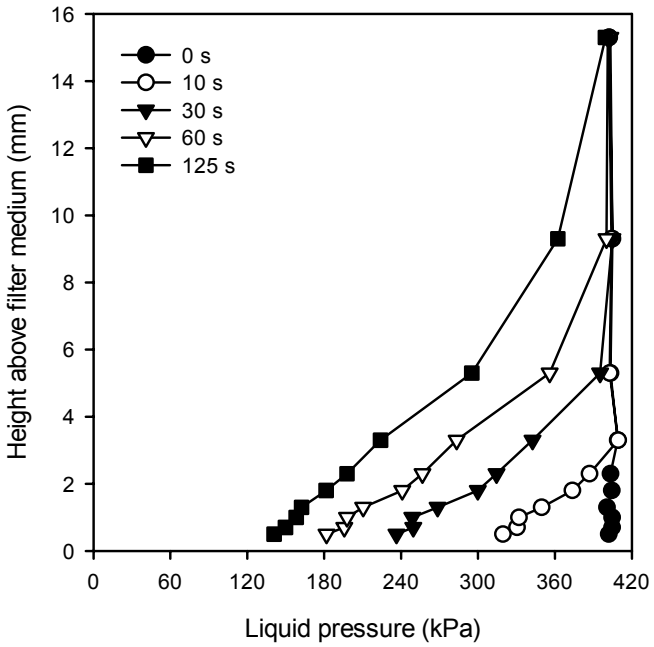

Figure 6: Liquid pressure profiles in a forming talc cake/suspension at a constant filtration pressure of 400 kPa.

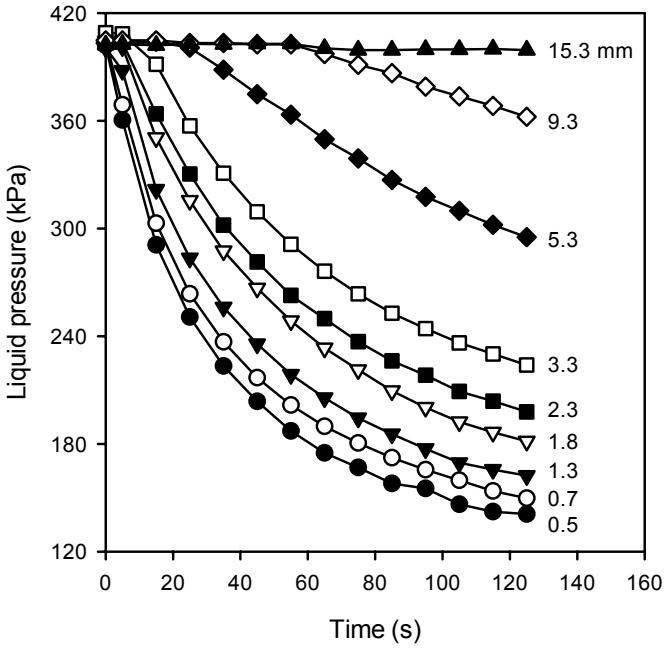

Figure 7: Liquid pressure history in a forming talc cake/suspension at a constant filtration pressure of 400 kPa.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

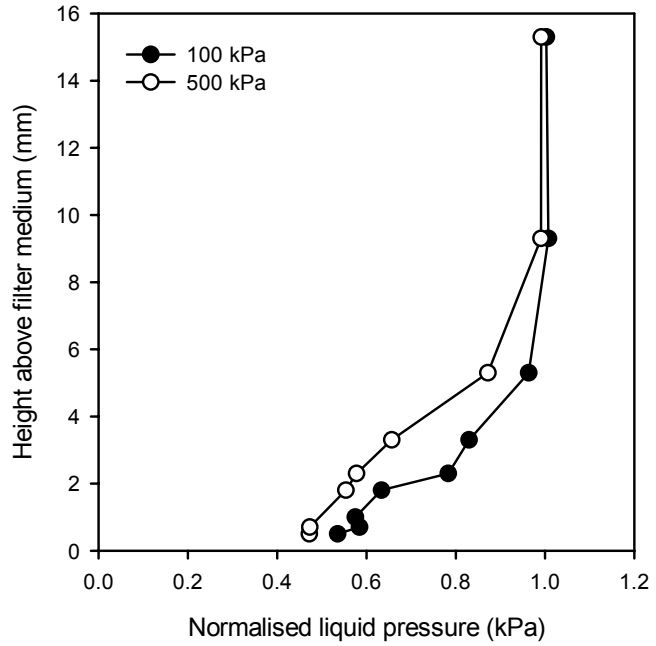

Figure 8: Normalised liquid pressure profiles for talc after 60 s (constant pressure filtrations).

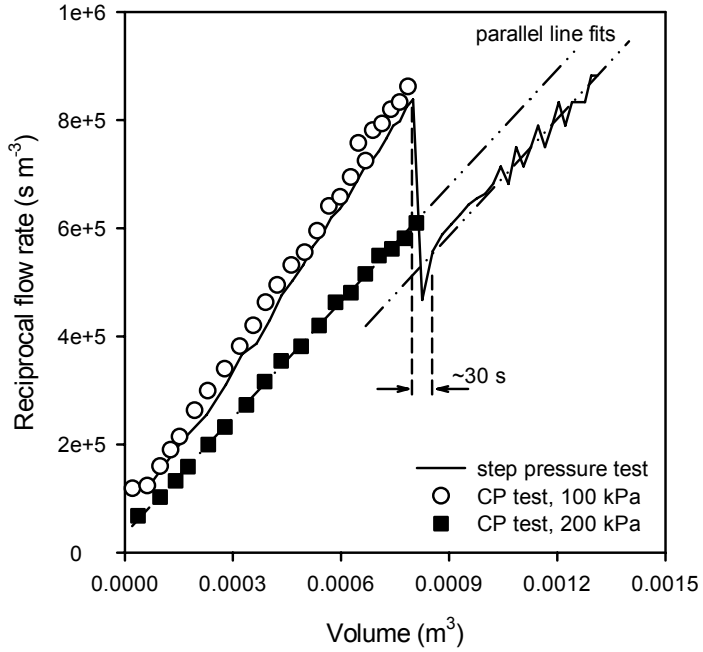

Figure 9: Comparison of individual constant pressure (CP) experiments and a stepped pressure experiment where a 100 kPa pressure increment is invoked after 360 s.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

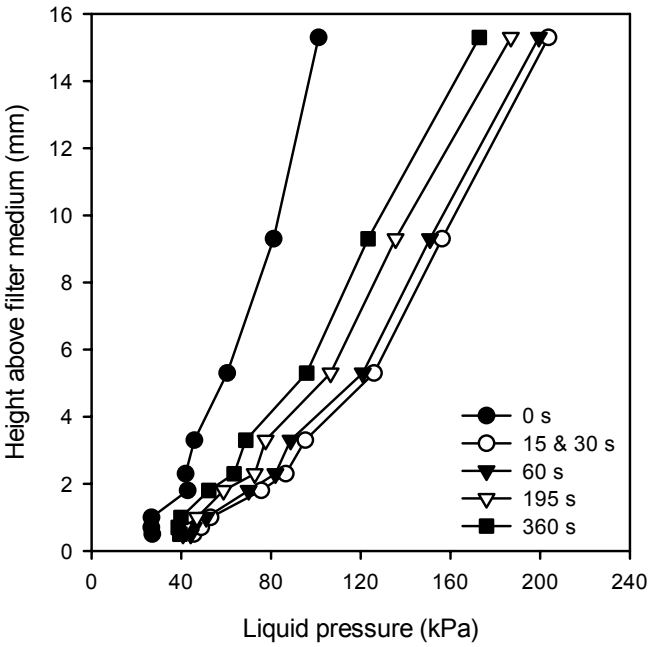

Figure 10: Liquid pressure profiles during the 200 kPa period of the 100→200 kPa step pressure experiment shown in Figure 8 (t = 0 s represent the end of the cake formation at 100 kPa).

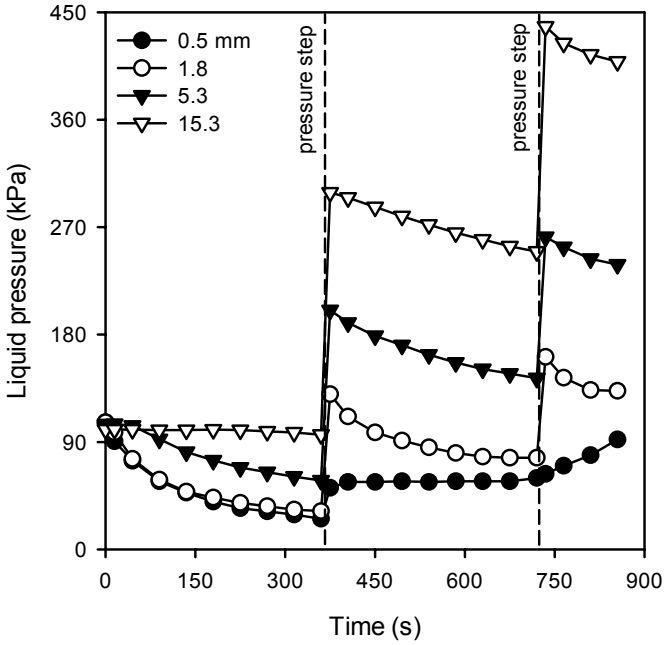

Figure 11: Liquid pressure histories for talc during a 100→300→500 kPa step pressure experiment (200 kPa pressure increment every 360 s).

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

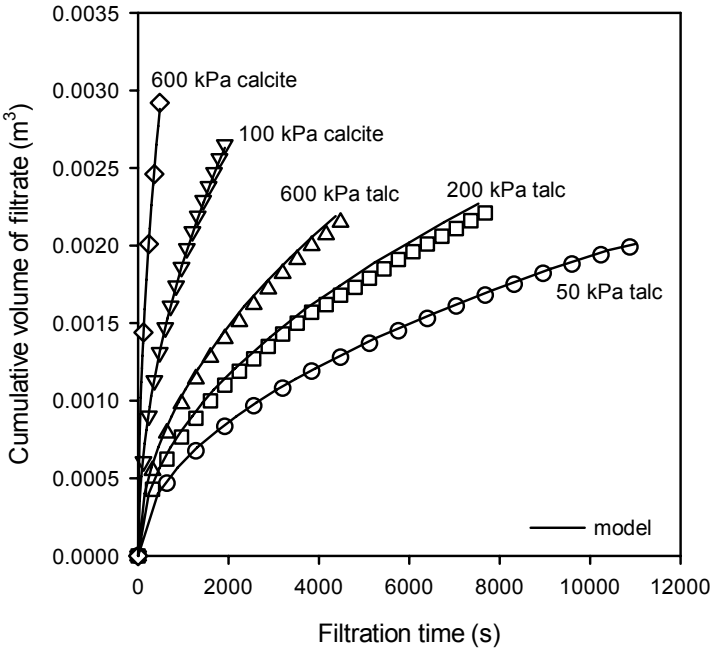

Figure 12: Comparison of individual constant pressure experiments obtained with a pressure Nutsche filter and simulations using Filter Design Software.

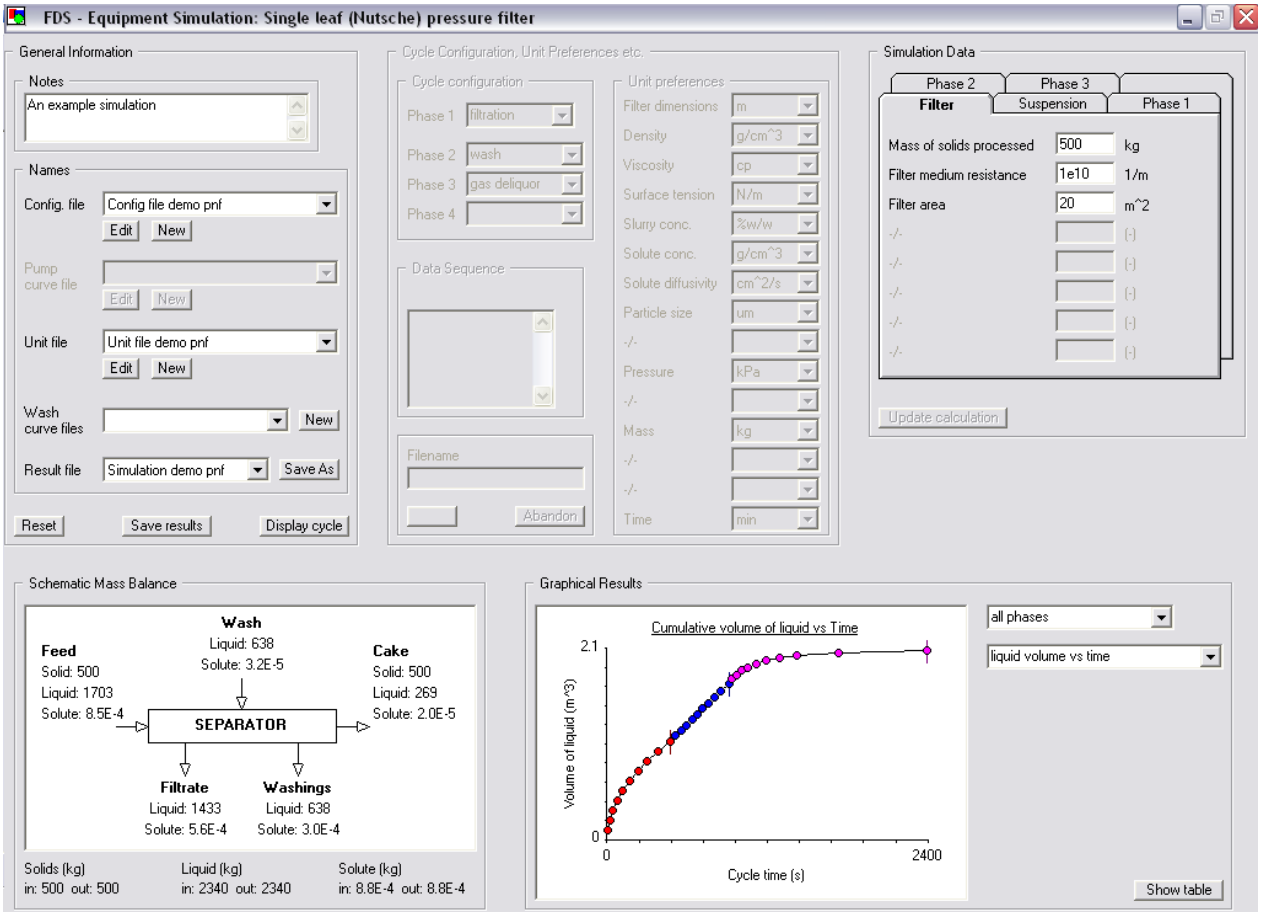

Figure 13: Filter Design Software screen display for a pressure Nutsche filter simulation.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

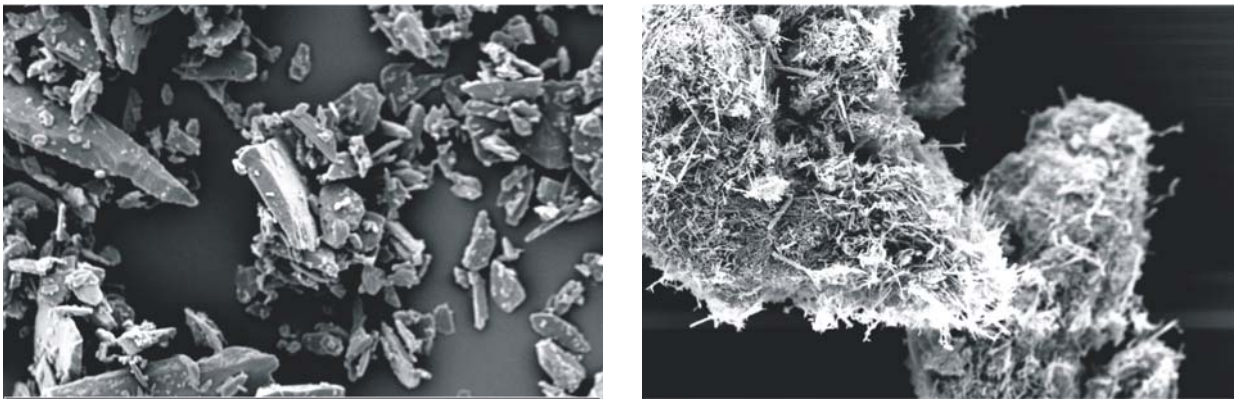

Figure 14: Scanning electron micrographs of two forms of crystalline pharmaceutical product; cubic, *α*-form, (*left*) and needle, *β*-form, (*right*).

Parameter	Talc	Calcite
50% particle size (μm)	8.5	12.8
Particle shape	Platelet	Rhomboid
Iso-electric pH	2[†]	9
Max. ζ-potential (mV)	-55 (@pH 11)[‡]	-20 (@pH 12)[‡]

Analar grade HCl[†] or NaOH[‡] used to alter pH

Table 1: Some properties of talc and calcite powders dispersed in distilled water.

Material	Type of test	α_0 (m kg^{-1} Pa^{-n})	n
Talc	sequence of individual constant pressure	1.23x10^9	0.42
Talc	stepped pressure (200 kPa increment every 480 s)	9.40x10^8	0.43
Talc	stepped pressure (50 kPa increment every 240 s)	5.45x10^8	0.53
Calcite	sequence of individual constant pressure	1.17x10^9	0.20

Table 2: Comparison of scale-up constants for specific cake resistance ($\alpha_{av} = \alpha_0(1-n)\Delta p^n$) and different types of experiment over the pressure range 50-600 kPa; correlation coefficients of the data fits for these calculations were in excess of 0.97. α_0 is specific cake resistance at unit applied pressure; n is compressibility index; Δp_f is filtration pressure.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

Parameter	Value
Septum characteristics	
Filter medium resistance (m^{-1})	$4x10^{10}$
Operating conditions	
Filtration, washing and deliquoring pressures (kPa)	200
Solute concentration in the feed (kg m^{-3})	9
Particle and fluid properties	
Density of filtrate and wash (kg m^{-3})	802
Viscosity of filtrate and wash (Pa s)	0.0023
Surface tension of filtrate and wash (N m^{-1})	0.025
Solute diffusivity (m^2 s^{-1})	$6x10^{-10}$
Particle and cake properties specific to α-form	
Density of solids (kg m^{-3})	1370
Constitutive equations for filtration (as determined with the aid of FDS). Δp_f in kPa	$\alpha_{av} = 5.6x10^9 \Delta p_f^{0.2}$ m kg^{-1} $C_{av} = 0.28 \Delta p_f^{0.05}$ v/v
Particle and cake properties specific to β-form	
Density of solids (kg m^{-3})	1420
Constitutive equations for filtration (as determined with the aid of FDS). Δp_f in kPa	$\alpha_{av} = 4.5x10^9 \Delta p_f^{0.5}$ m kg^{-1} $C_{av} = 0.27 \Delta p_f^{0.06}$ v/v

Table 3: Characteristic parameters for the Nutsche filter simulation. Δp_f is filtration pressure; α_{av} is specific cake resistance; C_{av} is cake solids concentration.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003

Parameter	*α*-form	*β*-form
Filtration phase		
Duration (s)	707	2363
Specific cake resistance (m kg^{-1})	$1.62x10^{10}$	$6.36x10^{10}$
Cake solids volume fraction (v/v)	0.365	0.371
Cake thickness (mm)	50	47.4
Cake moisture content (%)	50.5	48.9
Washing phase		
Duration (s)	568	1959
Fractional solute recovery	1	1
Deliquoring phase		
Duration (s)	1500	1500
Final cake saturation	0.33	0.42
Final cake moisture content	24.9	28.6
All phases		
Total cycle duration (s)	2775	5822
Total volume of liquids produced (m^3)	0.773	0.736

Table 4: Comparison of filter cycle performance for two particle forms in a Nutsche filter. Δp_f = 200 kPa; A_f = 2 m^2.

Cite paper as: Tarleton E.S., 2008, Cake filter scale-up, simulation and data acquisition, *J. Chinese Institute Chemical Engineers*, **39**, 151-160. DOI: 10.1016/j.jcice.2007.12.003