#### COMPUTER SOFTWARE FOR THE AUTOMATED SPECIFICATION OF SOLID/LIQUID SEPARATION EQUIPMENT

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This paper details Filter Design Software<sup>®</sup> (FDS), Windows<sup>®</sup> software for the selection and simulation of solid/liquid separation equipment as well as the analysis of test data. FDS was developed in collaboration with multi-national companies spanning a wide range of industrial sectors to provide a comprehensive calculation, education and training tool that maintains a balance between ease of use, level of knowledge conveyed and comprehensibility.

The selection module of the FDS compares up to 7 user-defined selection criteria with information contained in databases to produce a numerically ranked list of potentially suitable equipment. The FDS allows access to text and pictorial descriptions of more than 70 equipment types and hyperlinks provide more specific equipment manufacturer details via the internet. The data analysis module facilitates interactive analysis of leaf filtration, jar sedimentation and piston press test data. Calculations are performed in a hierarchical manner using the available information, if some data are not measured then the FDS performs the best possible analysis using approximations. The results of an analysis can be used to refine (shorten) a list of selected equipment or provide scale-up information for equipment simulation. The two equipment simulation modules provide calculations of cake formation, compression, gas deliquoring and washing. The user is able to input filter cycle data in their preferred units and guidance is given as to suitable numeric ranges for the type of filter being simulated. Results are presented on-screen in graphical and tabular forms and a mass balance is given for the solid, liquid and dissolved solute components present. The results are also made available in data sheet form which can subsequently be imported into a spreadsheet.

# **KEYWORDS**

Equipment selection; Software; Solid/liquid separation; Simulation; Analysis of filtration data; Process design.

# INTRODUCTION

The specification of filters is generally performed through rules-of-thumb (or heuristics) rather than by applying fundamental theoretical relationships. Equipment is rarely specified without recourse to extensive laboratory and pilot scale tests, and the data produced can lead to erroneous specification and scale-up of separators unless care and consistency are observed. The lack of a standard 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.

Progressive developments have facilitated a combined theoretical and experimental approach to the use of computer software in filter specification and simulation<sup>1-3</sup>. The philosophy considers that with the present state of knowledge of suspensions, and their behaviour in separators, it is most appropriate to have interactive computer software that forms an integral part of an experimental

program (Figure 1). Within this context, the Filter Design Software<sup>3</sup> (FDS), designed to run under Windows<sup>®</sup>, was developed.

The FDS is a sequence of interlinked modules that can be used independently from one another. The full set of FDS modules offers many capabilities, including:

- A catalogue and explanation of the main operational and design features of 70+ equipment types and 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
- The ability to import data files from other software (e.g. spreadsheets)
- Web access to equipment suppliers.

### EQUIPMENT SELECTION AND EQUIPMENT CATALOGUE MODULE

The general procedure developed by Purchas<sup>1</sup>, and the use of ranking indices, provide the basic functionality for the equipment selection module of the FDS. Figure 2 shows a typical screen display.

When the software starts only the 'Specifications' box in the top left hand corner of the screen is displayed. The available entries allow the user to select up to 7 items from drop down lists. These define the Duty, which must be specified, and the Settling and Filtration characteristics which are optional entries. In the example, an item in each drop down list has been chosen indicating that experimental data are available. If equipment selection is performed by specifying only the items for Duty then a longer list of equipment is likely to result. Choosing the 'Select' command button displays the 'Selected equipment list' box towards the top right of the screen where the user specifications have been compared against the FDS database of separation equipment. Choosing an equipment item subsequently displays the text and pictorial information toward the bottom of the display. Additional features of the module functionality include:

- 'Selected Equipment List': Ranked listing of solid-liquid separation equipment that matches the specifications. The five indices shown for each equipment type range between 0 and 9 where the latter indicates best possible performance. The listing can be prioritised according to solids dryness, liquid clarity etc. and equipment that is a marginal choice is noted.
- 'Equipment Descriptions': General and detailed technical and design information about the chosen equipment
- 'Equipment Schematic etc.': Schematic diagrams and photographs of the chosen equipment. Additional information which can help to eliminate equipment from the ranked list is available as is a customisable display of equipment suppliers. The ability to 'cut and paste' the web address of a supplier to an on-line browser is provided.

In addition to the Equipment Selection procedure an Equipment Catalogue can be accessed by the user. The display is broadly similar to that in Figure 2, but the FDS equipment database is categorised according to 11 classes (e.g. continuous vaccum filter, gravity sedimentation) and arranged in the form of a reference manual in order to provide education and training.

### DATA ANALYSIS MODULE

The Data Analysis module of the FDS allows for the interactive analysis of constant pressure and constant flow filtration, jar sedimentation and piston press (expression) tests. Data obtained at the laboratory, pilot and even full scale can be analysed in a consistent manner to either give additional information for equipment selection or (by repeated use) scale-up correlations for equipment simulation. An example of the screen display for filtration analysis is shown in Figure 3 whilst Figure 4 shows the screen display during the calculation of scale-up coefficients for cake formation and consolidation.

Referring to Figure 3, the user is initially required to type or select choices in the 'General Information' box toward the top left hand corner of the display. Descriptions for the test to be analysed can be typed and the Data and Unit Files selected. The Data file is specific to the type of analysis, in the case of constant pressure filtration the data is time vs. cumulative volume of filtrate, and can either be typed by the user or imported from a spreadsheet as required. The Unit File allows the user to enter information in their preferred units by selecting from a list. The 'Experimental Data' box is used to enter other relevant data from a test, including properties of the feed and operational parameters for the test apparatus. Even with well conducted tests, some of the necessary input data can missing yet the best possible analysis must be done with the available information. The FDS deals with this situation in two ways. Firstly, when the input data are entered they are checked as far as is possible and if the FDS suspects that the data may be incorrect it warns the user or does not accept the data. In many cases the FDS displays a range of acceptable values for the data as a guide to the user. Secondly, the calculation sequences within the FDS are hierarchical. Depending on which data are missing, a sequence of assumptions are made in order to carry out the calculation. After an assumption has been made, a warning may appear against item(s) of output data in the 'Tabulated Results' box towards the bottom of the display.

Results of an analysis can be displayed in either tabulated form, as shown in the example, or graphical forms. For each type of analysis a 'Characteristic Plot' is produced toward the bottom right hand corner of the display, for constant pressure filtration this is time/volume vs. cumulative volume. Vertical line cursors are used to identify the linear region of the plot and these are initially positioned by the FDS, however, the user has the facility to interact with the software and move them as appropriate. The results of an analysis can be saved to disc on the computer (in spreadsheet accessible form) and the results of several analysis can be recalled in order to compare and contrast results and/or evaluate scale-up correlations for equipment simulation as shown in Figure 4.

### SIIMULATION MODULES

Two simulation modules are available. 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. Figure 5 shows an example screen display for the simulation of a bottom fed rotary drum filter fitted with a knife discharge.

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 a vacuum filter this may comprise combinations of cake formation, washing and gas deliquoring; the FDS prevents impractical stages on particular filters, for instance, cake washing on a rotary disc filter. Similar to the Data Analysis module,

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.

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.

Some key features of the Vacuum and Pressure filter simulation modules include:

- Simulation of the different modes of cake formation as determined by the type/method of pumping used (constant pressure, constant flow and variable pressure/variable flow), compression filtration/consolidation and cake post-treatment processes (cake washing and cake deliquoring)
- Checking of input data for each required entry the FDS displays a range of numerical values to guide the user as to what is realistic for a particular filter
- Where possible the simulation calculation sequences within the FDS are hierarchical depending on which data are missing, a sequence of assumptions are made in order to carry out the calculation
- The FDS takes account of practical constraints, for example, the minimum cake thickness that can be discharged from a particular filter
- Graphical or tabulated output of results
- On screen display of a process mass balance, indicating the input/output amounts of solid, liquid, and dissolved solute components
- The ability to save results to disk for later recall and viewing in spreadsheets.

### **Example of Simulation**

A pressure driven Nutsche filter is to be used to separate batches of a crystalline pharmaceutical product (see Figure 6) from a propanol based suspension. Variations in upstream formulation mean that crystallisation of the  $\beta$ -form, which is more difficult to filter, can occur in place of the  $\alpha$ -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 characteristics for both the  $\alpha$  and  $\beta$  particle forms in suspension have been determined experimentally and these are shown in Table 1 along with other suggested operational parameters.

For the  $\alpha$ -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  $\beta$ -form crystallisation occurs.

Repeated use of the FDS facilitates a solution to the example problem; more details are provided elsewhere<sup>1,4</sup>.

With the  $\alpha$ -form of crystal, the required filter area is 2 m<sup>2</sup> 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 bottom row of Table 2, the total cycle time is 2775 s.

With reference to Figure 6 and Table 1, the  $\beta$ -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  $\beta$ -form with the 2 m<sup>2</sup> Nutsche then the results shown in Table 2 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  $\alpha$ -form. The approximate four fold increase in specific cake resistance with the  $\beta$ -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  $\alpha$ -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  $\beta$ -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

This paper has described the principal features of Filter Design Software. The four integrated modules comprising the software, which can also be used in isolation, have been developed to enable:

- 1. A selection procedure that facilitates ranked listing and access to on-line equipment and process information from a knowledge of the required duty and basic experimental data
- 2. The consistent analysis of filtration, expression and jar sedimentation tests to allow the accurate determination of the parameters required for process simulation and the basic information needed for equipment selection
- 3. The detailed simulation of process scale batch and continuous filters involving combinations of filtration, consolidation, washing and deliquoring.

By doing so a number of benefits arise, including:

- 1. The ability to investigate new plant and ask 'what-if' questions about filter installations to facilitate optimum equipment selection(s), filter sizing, cycle configuration(s) and filter operation
- 2. The ability to troubleshoot existing filter installations and identify potential solutions

- 3. Consistent experiment analysis to give characterisation and scale-up parameters
- 4. Unbiased information on solid/liquid separation equipment so appropriate manufacturers can be approached in the early stages of equipment selection
- 5. The ability to educate and train a user in solid/liquid separation technology.

## ACKNOWLEDGEMENTS

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### REFERENCES

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#### **FIGURES AND TABLES**

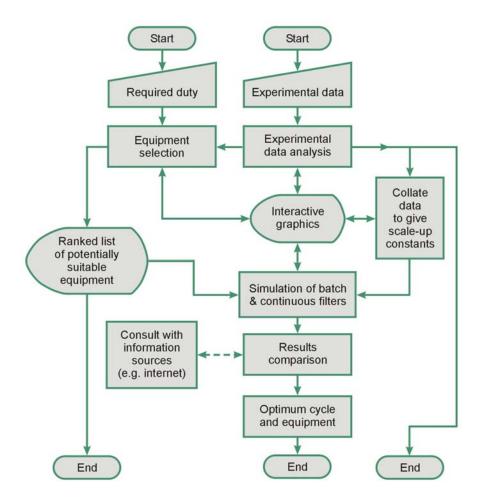


Figure 1: Flowsheet showing the integration of selection, analysis, scale-up and simulation.

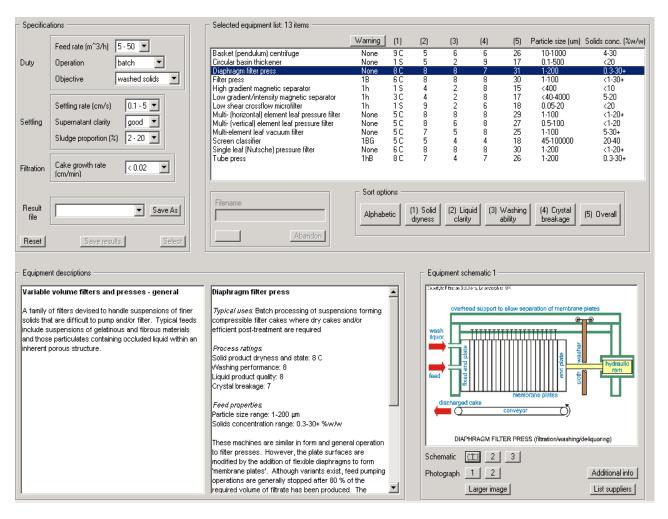


Figure 2: Example screen display from the Equipment Selection module of the FDS.

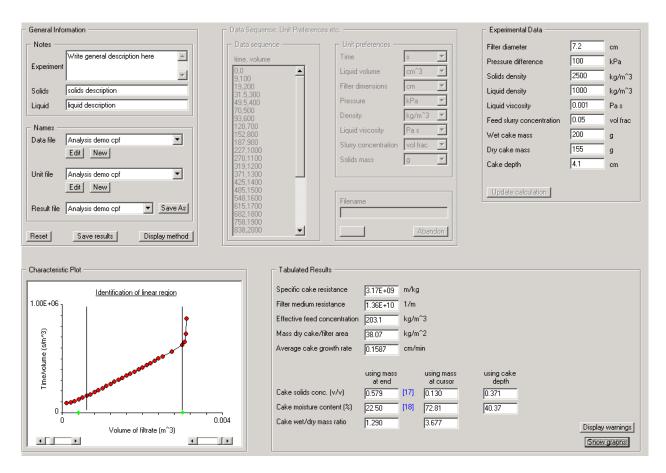


Figure 3: Example screen display of a constant pressure filtration analysis using the Data Analysis module of the FDS.

General Information	Comparison Selections			
Notes   Comparison file   Analysis demo pp   Edit	Datasheet name File 1 Experiment 1 pp File 2 Experiment 2 pp File 3 Experiment 3 pp File 4 Experiment 4 pp File 5 Experiment 5 pp File 6	Pressure (MPa)     Feed conc. [%v/v]       0.331     12.5       1.65     12.5       2.36     12.5       3.37     12.5       10.8     12.5	Cake resistance (m/kg)     Cake ( Filtration       6.29E+12     0.360       1.16E+13     0.417       1.33E+13     0.401       1.60E+13     0.438       3.44E+13     0.499	conc. (v/v)     Consolidation coeff. (m*2/s)       0.413     2.14E-08       0.472     7.28E-08       0.485     7.83E-08       0.499     1.04E-07       0.581     1.40E-07
Data Comparisons	Filename Abandon			
Specific cake resistance vs Prr 1.00E+14 g	Scale-up Coefficient Values	Coefficient 1 (c1)	Coefficient 2 (c2)	Correlation coeff. (R^2)
<u> </u>	Specific cake resistance (m/	(g) 6.82E+11 m/(kg	1 kPa^c2) 0.478	0.9850
Cate resistance (m/kg)	Cake concentration, filtration	(v/v) 0.209	0.091	0.9628
esistar	Cake concentration, consolid	ation (v/v) 0.233	0.096	0.9896
	Consolidation coefficient (m^2	2/s) 1.04E-09 m^2/	s kPa^c2) 0.550	0.9699
1.00E+12 100 Pressure (kPa)	100000 specific cake resistance vs pres	sure		

Figure 4: Example screen display of scale-up correlations obtained from a sequence of expression tests using the Data Analysis module of the FDS.

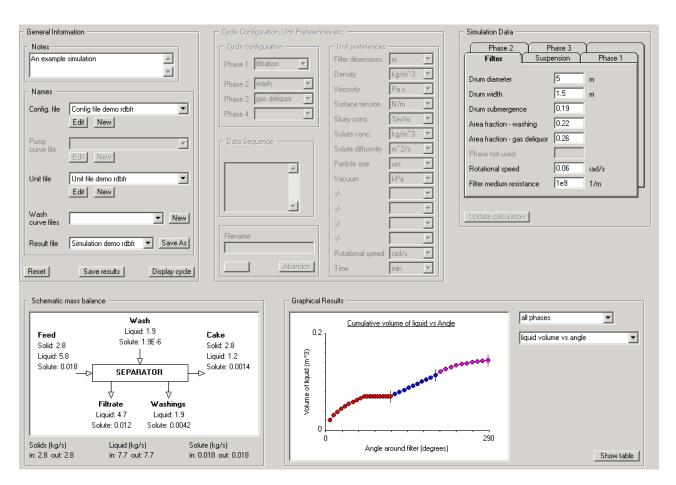


Figure 5: Example screen display for a rotary vacuum drum filter simulation using the FDS.

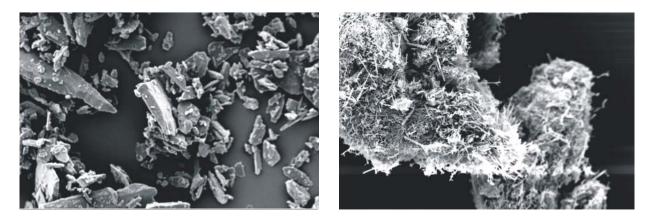


Figure 6: Scanning electron micrographs of two forms of crystalline pharmaceutical product; cubic,  $\alpha$ -form, (*left*) and needle,  $\beta$ -form, (*right*).

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

Table 1: Characteristic parameters for the Nutsche filter simulation.  $\Delta p_f \equiv$  filtration pressure;  $\alpha_{av} \equiv$  specific cake resistance;  $C_{av} \equiv$  cake solids concentration.

Parameter	α-form	β-form
Filtration phase		
Duration (s)	707	2363
Specific cake resistance (m kg <sup>-1</sup> )	1.62x10 <sup>10</sup>	6.36x10 <sup>10</sup>
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
Total cycle duration (s)	2775	5822
Total volume of liquids produced (m <sup>3</sup> )	0.773	0.736

Table 2: Comparison of filter cycle performance for two particle forms in a Nutsche filter.  $\Delta p_f = 200 \text{ kPa}; A_f = 2 \text{ m}^2.$