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SOFTWARE FOR DESIGN OF EXPERIMENTS AND RESPONSE MODELLING OF CAKE FILTRATION APPLICATIONS

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Abstract

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Filtration is a widely used unit operation in chemical engineering. The huge variation in the properties of materials to be filtered makes the study of filtration a challenging task. One of the objectives of this thesis was to show that conventional filtration theories are difficult to use when the system to be modelled contains all of the stages and features that are present in a complete solid/liquid separation process. Furthermore, most of the filtration theories require experimental work to be performed in order to obtain critical parameters required by the theoretical models.

Creating a good overall understanding of how the variables affect the final product in filtration is somewhat impossible on a purely theoretical basis. The complexity of solid/liquid separation processes require experimental work and when tests are needed, it is advisable to use experimental design techniques so that the goals can be achieved.

The statistical design of experiments provides the necessary tools for recognising the effects of variables. It also helps to perform experimental work more economically. Design of experiments is a prerequisite for creating empirical models that can describe how the measured response is related to the changes in the values of the variable.

A software package was developed that provides a filtration practitioner with experimental designs and calculates the parameters for linear regression models, along with the graphical representation of the responses. The developed software consists of two software modules. These modules are LTDoE and LTRead. The LTDoE module is used to create experimental designs for different filter types. The filter types considered in the software are automatic vertical pressure filter, double-sided vertical pressure filter,

horizontal membrane filter press, vacuum belt filter and ceramic capillary action disc filter. It is also possible to create experimental designs for those cases where the variables are totally user defined, say for a customized filtration cycle or different piece of equipment. The LTRead-module is used to read the experimental data gathered from the experiments, to analyse the data and to create models for each of the measured responses.

Introducing the structure of the software more in detail and showing some of the practical applications is the main part of this thesis. This approach to the study of cake filtration processes, as presented in this thesis, has been shown to have good practical value when making filtration tests.

Keywords: Filtration, experimental design, empirical modeling, cake filtration

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Mikko Huhtanen

Contents

Nomenclature

A	filtration area	m^2
c	effective solids concentration of slurry	$kg_{solids}\, m^{-3}_{filtrate}$
C_e	modified consolidation coefficient	$m^2 s^{-1}$
c_{R0}	solute concentration in the cake at t=0	$kg\, m^{-3}$
c_{Rt}	solute concentration in the cake at t=t	$kg\, m^{-3}$
D	molecular diffusivity	$m^2\, s^{-1}$
d	particle diameter	m
D_L	axial dispersion coefficient	$m^2\, s^{-1}$
D_n	dispersion parameter	$-$
g	gravitational constant	$m\, s^{-2}$
i	number of drainage surfaces	$-$
K	constant in Eq. (2.1)	$-$
k	empirical parameter in Eq. (2.21)	$-$
L	bed thickness	m
L_∞	the final cake thickness after consolidation	m
L_{tr}	the cake thickness at transition point	m
m_s	dry weight of solids	kg
m_{tr}	the ratio of the mass of the wet cake to the mass of dry cake	$-$

n	compressibility coefficient	$-$
N_{cap}	capillary number	$-$
p_b	threshold pressure	Pa
Q	filtrate flow rate	$m^3\,s^{-1}$
R	medium resistance	m^{-1}
Rc	filter cake resistance	m^{-1}
Re	Reynolds number	$-$
S	saturation, volume of liquid in a cake per volume of voids.	$-$
S_∞	irreducible saturation	$-$
Sc	Schmidt number	$-$
t	time	s
T_c	dimensionless consolidation time	$-$
t_c	consolidation time	s
t_d	deliquoring time	s
t_f	filtration time	s
t_p	compression time	s
t_t	total time	s
t_w	washing time	s
u	velocity of the fluid	$m\,s^{-1}$
V	filtrate volume	m^3
w	cake mass over the specific area	$kg\,m^{-2}$
W_R	wash ratio	$-$

x	mean particle size	m
y	measured response value	$-$
$\bar{y}$	mean of the observed data	$-$
$\hat{y}$	estimated response value	$-$
α_0	local specific cake resistance	$m\,kg^{-1}$
α_{av}	average specific cake resistance	$m\,kg^{-1}$
α	cake specific resistance	$m\,kg^{-1}$
Δp	pressure drop	Pa
Δp_0	scaling pressure for local specific cake resistance	Pa
μ	viscosity of the filtrate	$Pa\,s$
ω_0	solids volume per unit filtration area	$m^3\,m^{-2}$
ρ	filtrate density	$kg\,m^{-3}$
ρ_l	liquid density	$kg\,m^{-3}$
ρ_s	density of the solids	$kg\,m^{-3}$
σ	liquid surface tension	$N\,m^{-1}$
ε_{av}	average porosity of the cake	$-$

List of publications

I. Savolainen, M., Huhtanen, M.*, Häkkinen, A., Ekberg, B., Hindström, R., Kallas, J., Development of testing procedure for ceramic disc filters, Minerals Engineering, 2011, 24(8), 876 - 885.

II. Huhtanen, M.*, Häkkinen, A., Ekberg, B., Kallas, J., Experimental study of the influence of process variables on the performance of a horizontal belt filter, Filtration, 2011, 11(2): 120 – 125.

III. Huhtanen, M.*, Salmimies, R., Kinnarinen, T., Häkkinen, A., Ekberg, B., and Kallas J., Empirical modelling of cake washing in a pressure filter, Separation Science and Technology, accepted 22nd November 2011.

IV. Huhtanen, M.*, Häkkinen, A., Ekberg, B., and Kallas, J., Software for statistical design of experiments and empirical modelling of cake filtration, Filtration, accepted September 2011.

Authors contribution to publications

The author has designed and written the software for experimental design and modelling used in Papers I-IV. In all of the listed papers the author has been the corresponding author and has been in charge of preparation. In Paper I, the author provided the experimental design calculations and helped in modelling and preparing the paper for publication.

Other related publications

The results gathered during the project have been presented in several conferences. The following list details these presentations, which have not been attached into this thesis.

1. Häkkinen, A.*, Huhtanen, M., Ekberg, B., Kallas, J., Utilization of statistical design of experiments for improving the efficiency of test filtration tasks, 10th World Filtration Congress, Leipzig, Germany, April 14 – 18, 2008.

2. Häkkinen, A.*, Huhtanen, M., Ekberg, B., Kallas, J., Optimization of the performance of a filter press by statistical design of experiments and empirical modelling, Proceedings of the 21st Annual American Filtration & Separations Society Conference, Valley Forge, PA, USA, May 19 - 22, 2008.

3. Häkkinen, A.*, Experimental study on dewatering of quartz tailing in vertical automatic filter presses, 11th CST Workshop 2008, Separation and Waste Water Treatment Techniques in Chemical and Mining Industries, Lappeenranta, Finland, June 12 - 13, 2008.

4. Huhtanen, M.*, Häkkinen, A., Ekberg, B., Kallas, J., Numerical simulation of filtration and drying time distribution on ceramic disc filter plates, 11th Nordic Filtration Symposium, Copenhagen, Denmark, August 25 - 26, 2008.

5. Häkkinen, A.*, Huhtanen, M., Ekberg, B., Kallas, J., Experimental study on dewatering of copper concentrate by a ceramic disc filter, 11th Nordic Filtration Symposium, Copenhagen, Denmark, August 25 - 26, 2008.

6. Häkkinen, A.*, Huhtanen, M., Ekberg, B., Kallas, J., Software for improving the efficiency of test filtration tasks, Proceedings of the 22nd Annual American Filtration & Separations Society Conference, Bloomington, MN, USA, May 4 - 7, 2009.

7. Huhtanen, M.*, Häkkinen, A., Ekberg, B., Kallas, J., Experimental study on the influence of process variables on the performance of a horizontal belt filter, FILTECH 2009, Wiesbaden, October 13 – 15, 2009.

8. Sparks, T.*, Huhtanen, M., Kinnarinen, T., Salmimies, R., Häkkinen, A., The challenge of red-mud filtration, 13th Nordic Filtration Symposium, Lappeenranta, Finland, June 10 – 11, 2010.

9. Huhtanen, M.*, Häkkinen, A., Ekberg, B., Kallas, J., LabTop – software for experimental design, modelling and visualization, 13th Nordic Filtration Symposium, Lappeenranta, Finland, June 10 – 11, 2010.

1. Introduction

Solid/liquid separation processes are widely used throughout the chemical, pharmaceutical, metallurgical and mining industries. Practically, filtration is everywhere. Filtration has been regarded as a Cinderella technology (Purchas and Wakeman, 1986) since it has been neglected, in spite of its wide scope of application and importance. One of the reasons why filtration is overlooked is its complex nature, when considering the web of interactions between the process constituents, particulate matter, liquid phase of the slurry, wash liquid and gas phase.

Solid/liquid separation processes are of current interest due to the ever-growing demands for energy, material and water efficiency in all areas of the process industry(Chase and Mayer, 2005). Over the years, a lot of progress has been made in enhancing the theoretical understanding of the different aspects of solid/liquid separation processes. However, the resulting theories are limited to individual sub processes of the filtration cycle such as cake growth, cake washing, consolidation and dewatering. These basic filtration theories are readily available in filtration text books (Wakeman and Tarleton, 1999; Rushton et al., 2000; Svarovsky, 2000; Wakeman and Tarleton, 2005). Combining these filtration subprocess theories to describe the outcome of a complete filtration cycle is challenging and the results are often not reliable. Even the conventional scale-up constants are not always constant but may depend on the scale at which the filtration tests are performed (Tarleton and Willmer, 1997). The knowledge of filtration theory is vital not only for equipment manufacturers and developers, but end users also need to understand the basics of how the process variables affect the final filtration product.

The vast number of variables that can affect the outcome of filtration processes makes experimenting a necessity (Mayer, 2000). For example, the average specific cake resistance and the average porosity of the filter cake need to be determined by performing series of filtration tests. While there are theoretical models available that can be used to predict the average

specific cake resistances and average porosities, these models are either unreliable or they have been designed for specific applications (Shirato et al., 1987b; Wakeman and Tarleton, 2005). The conclusion is that there are no universally valid models available and that experimental work is always required in order to obtain the values of average specific cake resistances and average porosities. Experimental work is essential in all solid/liquid separation studies whether the goal is to understand the overall filtration process, to design new apparatus, to optimise existing filtration equipment, or to make scale-up calculations from laboratory to production.

Test filtration is an integral part of filter manufacturers' work for finding suitable filtration equipment for a client and for gathering information on how different types of filters perform with a given slurry. In test filtration, the aim is often to get an overall view of the capability of the selected filter type to achieve the required filter capacities, cake moistures, etc. This means that the complete filtration cycle needs to be carried out in order to obtain data of the filtration outcome. It is important to understand that a filtration cycle typically consists of different filtration sub-processes.

Let us consider a simple membrane filter press cycle that comprises the following three subprocesses; filtration (cake formation), compression dewatering and displacement dewatering with pressurised air, and suppose that we need to study the effects of four variables, say, slurry pumping pressure, pumping time, pressing pressure and drying time. Using filtration theories we first study the characteristics of the cake produced by applying the changes to the pumping pressure and pumping time. This needs experimental work, as the material characteristics are ususally unknown.

Clearly, using different combinations of these two variables will create cakes that have different properties in terms of porosity, thickness and average specific cake resistance. Assuming that we can create a model for how the pumping pressure and pumping time affect those properties, we still have two variables that need attention and the model does not provide information on how the produced cakes will behave in the next stage.

The next stage in this filtration cycle is compression dewatering, and the variable to be studied is pressing pressure. Knowing the cake properties in advance does not tell directly how the cake is changed in compression stage and we need experimental data in order to establish a model for the properties of the cake after the compression stage (with varying pressing pressures).

Compression stage studies need to be done with different cake thicknesses and porosities to develop a model that explains how this particular material behaves during the compression stage. These compression studies require a new set of experiments to provide cakes that have different properties.

Finally, we have displacement dewatering with compressed air. This stage has drying time as a variable and, again, the models from the previous stages do not provide direct information on how the cake will behave. Once again we need to perform a new set of experiments, because the cake properties are changed during the course of the filtration cycle. This time, we need to provide cakes that have gone through the compression stage, so that we can assess the effect of the drying time on the final moisture content of the cake.

After this form of experimental study, the models still need to be combined, which is no trivial task. Supposing that the combined model could have been created, it must be remembered that this model would be valid only for that particular set of variables, variable ranges, materials, filter type and filtration cycle. Any changes in the previously discussed constraints would mean that the model should be renewed and the test filtrations typically differ from each other case-by-case.

When this work was started, it was unclear whether or not it would be possible to model the test filtration tasks without extensive use of filtration theories at all. This is the reason why it was absolutely necessary that the case studies were done with a large number of experiments and with a wide range of different slurry types.

The scarcity of methods for combining different filtration sub-process theories to see the total effect of the process variables on the filtration outcome was the inspiration of this work. The process variables are those variables that are present when operating the filtration equipment. These include slurry variables, for example temperature, solid content and pH. Another set of process variables includes pressures and times used in the filtration cycle as well as some equipment specific variables, like the slurry level in the basin of a capillary action disc filter. The idea was to create a software package that guides the user to make experimental designs that allow statistical analysis and modelling of the experimental results as well as detection of the process variable effects on the measured responses. The models created with this software do not replace theoretical knowledge (Tiller, 2004) but work as a practical tool to gain information on how the overall process of

filtration works for particular cases.

The software was created, and continuously improved, alongside an extensive programme of test work. During the course of this work, ten different slurries were investigated and more than 500 individual test runs completed. The large number of experiments with different equipment and materials were required in order to ensure that the selections made during the course of this work, as well as the results obtained, are within the required accuracy range. Accuracy with this type of test work is determined by practicalities and finding the window of operation, i.e. what can be achieved with the selected variables and what is the performance level. In other words, maximising the accuracy of the models was not the main goal but rather it was to produce models that are easy to interpret in practical applications.

The models that are created from filtration tests are expendable models, and they are used to get an overview of how the tested filter type, material and variables behave together. The long term objective is to gather better quality data for future analyses.

The software created during this study and introduced in this thesis, LabTop, consists of two different software modules. These modules are called LTDoE and LTRead. The software has been written in the Matlab® scripting language accompanied with Statistical and Report Generator toolboxes.

The LTDoE-module can be used to create experimental designs for five different types of filter. The filter types that are included in the current version of the software are (i) automatic vertical pressure filter, (ii) double-sided vertical pressure filter, (iii) membrane filter press, (iv) vacuum belt filter and (v) ceramic capillary action disc filter. It is also possible to create experimental designs for other filter types, in which case the variables are totally user defined. The LTDoE-module creates the experimental designs so that the user only needs to select the filter type, the stages included in the overall filtration cycle, the variables of interest, variable levels (low and high values) and the number of experimental runs. Based on the user input, the experimental design is created and written to an Excel file. The software package that has been created during this work is easy to use and it requires no prior knowledge of statistical design of experiments or modeling. The experimental designs are created according to the principles of factorial designs as discussed in chapter 3.

The LTRead-module is used to read the experimental data gathered from

the experiments designed using LTDoE. The software analyses the data and creates regression models separately for each of the measured responses. The models and the statistical data are stored in an Excel file, alongside the original data. The user will also have the opportunity to create additional figures for reporting purposes and/or to create an auto-generated report containing the model data and figures. The LTRead-module can be used to find the optimum conditions for the measured response within the variable range that the user has selected, although this is not the aim of the software. Optimisation would require an additional software module which has not, to date, been implemented in the software.

This thesis consists of two parts. In Part I, the essential theories concerning filtration, statistical design of experiments and modeling strategy are presented. In Part II, the detailed structure of the software is introduced, together with some of the experimental results gathered during this work. The case examples are taken from conference presentations in order to show the results that have been obtained during the course of this study. Case examples also reveal the accuracy level that was acceptable for practical purposes.

There are also four journal publications attached to this thesis. In Publication I, the cake formation and dewatering time calculation methods used in the LTDoE - module for vacuum disc filters are presented. The cake formation and dewatering time calculation method improves the experimental accuracy of the leaf test filtrations and this method is written into the software. Improving the test filtration methods was one of the underlying topics during the course of this study. Publication I shows that a good agreement is achieved between the laboratory scale experiments and full scale filter results.

Publication II discusses different experimental design matrices and the applicability of these designs for the modeling of cake washing processes. Several different kinds of test designs were investigated, in order to define the minimum number of tests required in order to obtain satisfactory results for the investigated application. A comparison of different models showed that the amount of test work could be efficiently reduced by utilizing statistical design of experiments and empirical modeling tools.

In Publication III, the modeling of nonlinear response has been studied. Five different variables from a filtration, pressing, cake washing and air

drying stages were considered in the tests and the examined product characteristics were the overall capacity of the filter and the purity of the cake. The results obtained from the tests were used to create different kinds of regression models for explaining the influence of the studied variables on the success of the cake washing process. The goal of the modeling strategy for cake washing was to determine the simplest empirical models and compare these with theoretical equations complemented with linear terms. It was found that the empirical equation could model the results more accurately than the theory-based equations could.

In Publication IV, the outline of the software has been presented together with an experimental case study. Along with the results from the case study, LabTop software shows the strength of factorial experimental design and helps create measurement data that is structured for further analysis. It also works well in establishing the overall effects of selected variables on the response and provides tools for visualization of these effects.

The publications and case examples show the different aspects that have been taken into consideration while examining the test filtration tasks, experimental designs, empirical modeling and writing the software tool.

The software has been designed to assist, especially during the initial test filtration stage, when the behaviour of the slurry in filtration is unknown. Model functions created for test filtrations describe the local variable-response interaction with sufficient accuracy for decision-making, which is one of the main goals in test filtration. Test filtration is used as a stepping stone for sizing and design work and thus these tests provide information for designing new filtration processes for previously unknown slurries. The main questions that needs to be answered after completeting the test filtrations are: What is the operation window of the filter with the tested slurry and how do the variables affect the filtration outcome?

The novelty value of this work is in the application scope and not in the methodology. The experimental design and modelling methodologies used in this work are well established and tested, but the use of these methods in test filtrations for providing simple models, which are loaded with practical value, is new. The uncertainty over whether or not the filtration outcome can be modelled without applying the fundamental filtration theories is removed as the results show that an acceptable accuracy level for practical applications can be reached with simple linear models.

The comparison of the results obtained with factorial and fractional factorial experimental designs showed that models obtained with fractional designs give results that were almost as good as models that are based on factorial designs and that, therefore, a reduction of experimental runs can be made without losing too much valuable information.

Part I.

Theory

2. Filtration theory

This chapter presents the most relevant theories that describe filtration sub processes. These filtration theories are given here to show what type of mechanistic models are available in filtration studies, and to give an overview of how problematic it is to combine these theories into a single model that can be used to describe a complete filtration cycle. The theories have been limited to those that describe the most important responses, those that are almost always measured when performing filtration experiments. Filtration tests are quite often focused on the overall capacity of the filter, the residual moisture content of the cake and the purity of the filter cake, since these often define the success of a production process (Sparks, 2012). These responses are measured for practical reasons and the filtration theories deal with seemingly similar responses. However, the theories do not offer a direct route for calculating the aforementioned responses since they are affected by the conditions in other sub-processes of the complete filtration cycle. The overall capacity of a filter depends on average specific cake resistance, liquid permeability of the cake during washing and gas permeability of the cake during deliquoring. Cake residual moisture content is dependent on average porosity, compressibility, and parameters affecting air drying. Purity of the cake is basically the same as proposed in theoretical work but the differences between theoretical cake washing and practical filtration results are in the definition of wash ratio and on purity monitoring.

The filtration theory starts almost always with the Darcy's law (Darcy, 1856), which gives the flow velocity of a fluid through a porous bed:

$$u = K\frac{(-\Delta p)}{L} \tag{2.1}$$

where u is the velocity of the fluid, L is the thickness of the bed, Δp the pressure drop across the bed and K is a constant which is dependent on the particle and fluid properties and is referred to as the bed permeability.

In filtration, Darcy's law is usually applied in a modified form where the fluid velocity is replaced with the filtrate flow rate. Resistance terms replace the L/K term and thus the Darcy's law is given in terms of filtrate flow rate Q (Svarovsky, 2000):

$$Q = \frac{A\Delta p}{\mu\left(R + R_c\right)} \tag{2.2}$$

where Q is the filtrate flow rate, A is the filtration area, μ is the filtrate viscosity, R is the medium resistance and R_c is the cake resistance.

The medium resistance, R, is typically assumed constant during individual filtration experiments and the cake resistance, R_c, increases with time as the filter cake builds up. For incompressible cakes, R_c is assumed to be directly proportional to deposited cake mass and specific cake resistance:

$$R_c = \alpha w \tag{2.3}$$

where α is the specific cake resistance and w is the cake mass per unit area.

Combining Equations 2.2 and 2.3 gives:

$$Q = \frac{A\Delta p}{\alpha\mu w + \mu R} \tag{2.4}$$

Equation 2.4 shows the parameters α and R that need to be determined experimentally because there are no reliable methods to evaluate them from theory (there are some look-up tables, but there is quite a high level of uncertainty with these).

The Equation 2.4 forms the basis for the general filtration equation which is usually given in the following form:

$$\frac{dt}{dV} = \frac{\mu\alpha_{av}c}{A^2\Delta p}V + \frac{\mu R}{A\Delta p} \tag{2.5}$$

where c is the effective solids concentration in the feed slurry (or the mass

of cake solids deposited per unit volume of filtrate), t is time, V is the filtrate volume and α_{av} is the average specific cake resistance. The general filtration Equation 2.5 is rearranged and integrated for solving the constant pressure or constant rate filtration cases (Ruth, 1935). When the filtrate accumulation data with time are available, Equation 2.5 can be used to determine α_{av} and R.

2.1. Overall capacity of the filter

The overall capacity of the filter is an essential parameter when the filtration tests are used to collect data for sizing the selected filter type. The capacity of a filter depends not only on the filtration stage duration but on all of the filter stages that are present within the filtration cycle. A typical filtration cycle may contain the following stages: filtration, consolidation, washing and deliquoring. The total time t_t of the filtration cycle can be expressed as follows:

$$t_t = t_f + t_p + t_w + t_d \tag{2.6}$$

where t_f is the filtration time, t_p the consolidation time, t_w the washing time and t_d is the deliquoring time. In addition to these, a technical time (to account for cake discharge, cake release, etc.) must be taken into account when calculating capacity. Technical time is not discussed here because it is purely dependent on the filtration equipment.

When the total time is known then the filter overall capacity is calculated with:

$$Capacity = \frac{m_s}{At_t} \tag{2.7}$$

where m_s is the mass of solids.

The filter overall capacity is basically a measure of how much solid matter is fed into the filter and how long it takes to process it to a acceptable quality level. The amount of solids, in the form of slurry, that can be fed and how much time it takes, need to be determined experimentally.

Material properties, for example the particle size distribution, particle shape, solids concentration and liquid viscosity are the key elements that define the characteristics of the aforementioned subprocesses. The complex nature of capacity arises from the fact that the times used in different stages of the filtration cycle are dependent upon each other. For example if the filtration time is short, and the cake formed is thin, then the cake consolidation time needed is short, the time required for washing the cake to an acceptable level of purity might be long due to channeling or wash liquid maldistribution (Wakeman, 1998) and, finally, the deliquoring time is short if there is no cake shrinking or cracking.

The time required to complete the total filtration cycle is the sum of the durations of each of the different sub-processes, all of which have separate factors that either require separate test work or the utilisation of theoretical tools to obtain some estimated values for time consumption.

The filter medium is the basis on which the cake is build up. According to Mayer (2000), in cake filtration the filter medium resistance can be neglected because, usually, the cake resistance is much bigger than the cloth resistance.

The pressure difference used in filtration has an impact on the filtrate flow rate. Pressure level particularly affects compressible cakes and all filter cakes show some compressibility. Compressible cake shows increasing resistance with increasing pressure difference. A compressibility coefficient, n, is used as a measure for classifying cake compressibility characteristics. The classification is as follows (Sørensen et al., 1996); incompressible (n=0), slightly compressible ($0<n<0.5$), moderately compressible ($0.5<n<1$), highly compressible ($n>1$) and extremely compressible ($n>>1$). The compressibility constant can be established from a series of filtration tests at different pressures and the value for n is obtained from the following equation (Svarovsky, 2000):

$$\alpha_{av} = \alpha_0(1-n)\,(\Delta p)^n \tag{2.8}$$

where α_{av} is the average specific cake resistance, α_0 is the specific cake resistance at unit applied pressure, n is the compressibility coefficient, and Δp the pressure drop across the cake.

As stated by Rushton *et al.*(2000), increasing the filtration pressure results in an increase in cake solid concentration and thus leads to a decrease in cake

permeability which in turn affects cake washing, dewatering and finally the overall capacity of the filter.

2.2. Cake moisture content

The cake moisture content is an important factor for estimating how the filter can handle the final product specifications. The remnant moisture in filter cake is usually disadvantageous for the downstream processes, for example drying. (Removing the liquid from the solid matter by filtration is energy efficient when comparing to thermal drying.) The process steps in the filtration cycle that affect directly the final cake moisture content are compression and desaturation by gas displacement. Alongside these obvious factors, the filtration stage also plays a role in determining the final cake moisture content through the average specific cake resistance and average cake porosity. The average specific cake resistance indicates the ease of gas displacement and the average cake porosity shows the relative amount of liquid to be removed from the cake. The methods for reducing the cake moisture content are dependent on the filter type. Variable chamber pressure filters are usually able to perform dewatering by applying both compression and hydrodynamic displacement. The vacuum operated filters, for example drum- and disc filters, usually do not have a compression stage.

2.2.1. Compression deliquoring

Cake compression deliquoring is composed of three distinct stages. First, compression filtration where the mechanically applied pressure is used to filter the remaining slurry or semisolid material into a cake. This is also sometimes referred to as expression. The second stage is when all of the particles are having a point contact to other particles so that the cake fills the filtration chamber completely. This process is called primary consolidation. The third stage is the secondary consolidation during which the hydraulic pressure throughout the cake is almost zero and particles start to move into closer packing formation; some particles may even start to fragment.

The essential concepts regarding compression dewatering are the transition point and consolidation ratio U_c (Wakeman, 1975; Shirato et al., 1986a,b; Wakeman and Tarleton, 2005). The transition point is when filtration ends

and consolidation starts. The moment when transition from filtration to consolidation takes place has a corresponding time and cake thickness denoted as t_{tr} and L_{tr}. The transition cake thickness can be calculated from this equation (Shirato et al., 1986a):

$$L_{tr} = \left(\frac{m_{tr} - 1}{\rho} + \frac{1}{\rho_s} \right) \rho_s \omega_0 \tag{2.9}$$

where m_{tr} is the ratio of the mass of wet cake to the mass of dry cake at time t_{tr}, ρ_s is the density of the solids and ω_0 is the total volume of solids per unit filtration area. The term m_{tr} needs to be determined experimentally and it is often skipped since the term L_{tr} can be determined from experimental data (Wakeman and Tarleton, 2005).

The consolidation ratio shows the extent of the consolidation and is defined as

$$U_c = \frac{L_{tr} - L}{L_{tr} - L_\infty} \tag{2.10}$$

where L_∞ is the cake final cake thickness when consolidation time approaches infinity.

Theoretical models of consolidation often describe the consolidation ratio behaviour. The Terzaghi model for consolidation is one of the most used and it offers a basis upon which the later consolidation theories build. The Terzaghi model describes the primary consolidation phase and its equation form (Shirato et al., 1987a) is as follows:

$$U_c = 1 - \exp\left(-\frac{\pi^2}{4} T_c \right) \tag{2.11}$$

where T_c is a dimensionless consolidation time, which in turn is defined as:

$$T_c = \frac{i^2 C_e t_c}{\omega_0^2} \tag{2.12}$$

where C_e is the modified consolidation coefficient, i is the number of drainage surfaces and t_c is the consolidation time.

The modified consolidation constant, C_e, is empirical by nature and thus the value for C_e must be determined experimentally.

Secondary consolidation includes particle creep effects and the modification of the Terzaghi model to take these creep effects into account is known as the Terzaghi-Voigt model (Shirato et al., 1986a; Wakeman and Tarleton, 2005).

Shirato et al. (1986a) proposed a semi-empirical model which differs from Terzaghi and Terzaghi-Voigt models in the sense that it has fewer empirical constants (Salmela and Oja, 2005).

Consolidation takes place not only in pressure filtration, but it has also effect on vacuum filtration. The capillary forces at the surface of the cake can cause consolidation and this is important for fine materials where capillary forces are large (Stickland et al., 2010, 2011).

2.2.2. Displacement deliquoring

In displacement deliquoring, gas flow is used to displace the filtrate from the pore structure of the cake. Usually the gas used to displace the filtrate is air.

Cake saturation, S, is the volume of liquid in the cake divided by the volume of voids in the cake. The cake is fully saturated when all of the pores in the cake are filled with liquid. The cake saturation is calculated with the following equation:

$$S = \frac{Volume\,of\,liquid\,in\,the\,cake}{\varepsilon_{av} A L} \tag{2.13}$$

where ε_{av} is the average porosity of the cake.

The irreducible saturation, S_∞, is the saturation level after which further dewatering requires evaporative or thermal processes. The value for the irreducible saturation can be obtained by measuring the capillary curve for the material or it can be calculated from known cake properties with the capillary number N_{cap}. The capillary pressure curve is presented in Figure 2.1.

The average porosity is calculated with:

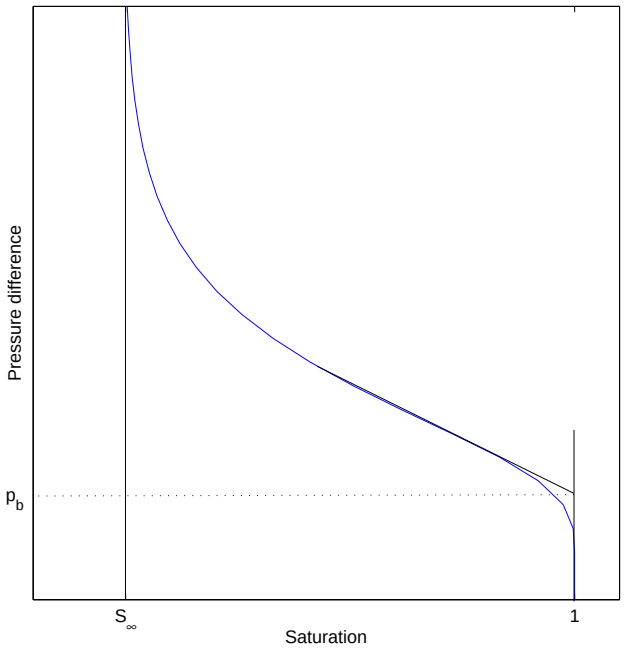

Figure 2.1.: Capillary pressure curve showing irreducible saturation (S_∞) and modified threshold pressure (p_b). Adapted from (Rushton et al., 2000).

$$\varepsilon_{av} = 1 - \frac{m_s}{\rho_s A L} \tag{2.14}$$

The threshold pressure p_b is the minimum pressure needed to initiate the deliquoring. This can be determined from the capillary pressure curve as the point where the capillary pressure curve starts to deviate from the line $S = 1$. The accurate determination of the threshold pressure might be difficult and therefore, instead of the actual threshold pressure, a modified threshold pressure is used. Figure 2.1 shows the graphical method for evaluation the modified threshold pressure. If there are no capillary curve

data available, the threshold pressure can be predicted from the following equation (Wakeman and Tarleton, 2005):

$$p_b = \frac{4.6(1-\varepsilon_{av})\sigma}{\varepsilon_{av} x} \tag{2.15}$$

The irreducible cake saturation for vacuum or pressure deliquored cake is calculated, according to Wakeman and Tarleton (2005), with equation:

$$S_\infty = 0.155(1 + 0.031 N_{cap}^{-0.49}) \tag{2.16}$$

and

$$N_{cap} = \frac{\varepsilon_{av}^3 x^2 (\rho_l g L + \Delta p)}{(1-\varepsilon_{av})^2 L \sigma} \tag{2.17}$$

where x is the mean particle size, ρ_l is liquid density, g is the gravitational constant, and σ is the liquid surface tension.

The key concepts in cake deliquoring are saturation, irreducible saturation, threshold pressure and average porosity. These values are used for calculating estimates of the final cake moisture or, alternatively, the time needed to obtain the desired moisture level. The equations from 2.15 to 2.17 are used to calculate dimensionless parameters used in design charts (Wakeman and Tarleton, 2005). Wakeman and Tarleton explain the use of design charts for cake deliquoring instead of mechanistic model equations with the fact that solving those equations is complex and requires numerical integrations of partial differential equations.

As an example, if a filtration process is completed in a variable chamber filter press, where the cake is first compressed and deliquoring continues with hydrodynamic displacement with pressurised air, it should be noted that the average porosity value is impossible to calculate because reliable cake thickness data cannot be obtained before the air drying is started.

Particle size, cake thickness and the applied pressure difference all affect both the the time to deliquor a cake to a specified moisture content and the average gas flow through the cake (Wakeman and Tarleton, 2005). In

practise this shows that the previous filtration process stages have an effect on the outcome of the cake drying stage.

2.3. Purity of the cake

The final product in cake filtration can be either solid matter, filtrate or both. In all of the cases the purity of the cake can be used for measuring how well the mother liquor and the solutes are withdrawn from the cavities of the cake. The purity of the cake refers to end results obtained by cake washing which in turn refers to the replacement of mother liquor with a fresh liquid. In cake filtration processes, the final product can be either solids retained in the filter cake, the liquid filtrate phase or in some special cases, it can be both the solids and liquids. If the final product is the solid phase then washing is used to remove any soluble impurities away from the final solid product. In the case of liquid being the product, the filter cake washing is applied as a method to remove the valuable product that is retained in the cavities of the filter cake.

The four most common ways to perform cake washing are:

- Co-current washing.
- Counter-current washing.
- "Stop-start" washing
- Re-pulping the filter cake with fresh liquid and filter the newly formed slurry again.

The first three operation methods can be regarded as displacement washing and the fourth is a dilution process. These operational methods describe the mechanisms of the washing process. Categorising the wash methods is also possible by the wash medium used. The wash medium can be the main fluid component of the mother liquid or a fluid that is not identical with the mother liquid and it can be either miscible or non miscible (Hoffner et al., 2004). In the article by Peuker and Stahl (2000), steam has also been used as a wash medium in cake filtration. Choosing the appropriate washing method is not necessarily straightforward because it depends heavily on the equipment available, product quality requirements, effluent and

solids material post-processing, wash liquid supply and so on (Hoffner et al., 2004). Sometimes it is reasonable to combine two different washing methods according to Tarleton and Wakeman (1999).

Gathering appropriate measurement data on the cake washing is a prerequisite for successful modeling. Typically, the washing measurement consists of solute concentration measurement as a function of wash liquid consumption. This might give an oversimplified picture of the procedure, especially when there are conditions and variables that should be kept constant. Those conditions and variables that are known to affect the washing curve are according to Svarovsky (Svarovsky, 2000):

1. Flow rate of wash liquid through the cake.
2. Mother liquor and wash liquid properties.
3. Solute to solvent diffusivity.
4. Cake properties like porosity, structure, initial saturation, homogeneity and thickness.
5. Washing inefficiencies such as cake cracking and channelling of the wash liquid.

The measurement data are usually in the form of averaged values of concentrations in the filter cake. Determination of the actual local concentration and dispersion coefficient values requires extraordinary measurement techniques such as those presented in the article by Lindau et al. (2007).

Regardless of the selected washing method or filtration type, the cake washing results are usually described by a wash curve. The wash curve usually has the dimensionless solute concentration of the wash filtrate plotted against the wash ratio as presented in Figure 2.2. There are of course other ways in which to represent the data, but most of these are tied to the solute concentration in washings or solute concentration in solids either retained or removed. The basic wash curves can be sometimes misleading since, in many industrial processes, the cake is the product and this is why industrialist often prefer wash data as presented in Figure 2.2 b) (Mayer et al., 2000; Mayer, 2001). When inspecting the figures showing washing data, one should also take time to check on the definition of the wash ratio used in the

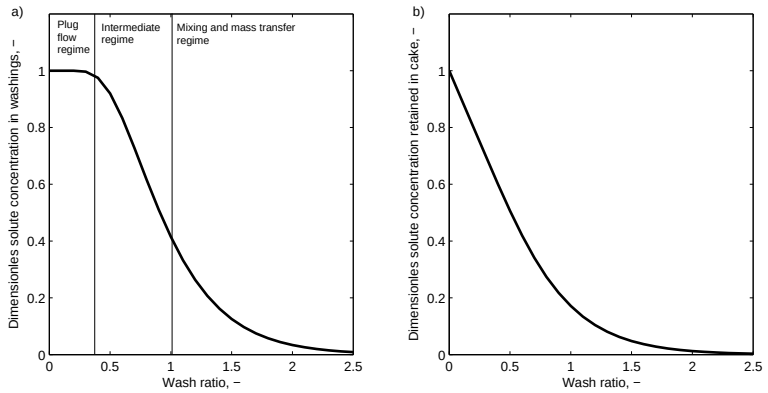

Figure 2.2.: a) A typical wash curve obtained when the solute concentration of the filtrate has been measured. b) The wash curve for the retained solute concentration in the cake.

figures. This is essential because sometimes the wash ratio can be expressed in different ways.

The wash ratio, W_R, is the volume of wash liquid used divided by the volume of filtrate retained in the cake at the start of washing. Sometimes the wash ratio is interpreted to be the volume of wash liquid divided by the void volume of the cake (Ruslim et al., 2007). The latter interpretation is by definition correct if the cake is fully saturated before the start of the washing. It should be noted that the curve in 2.2a) represents an initially fully saturated cake, and the curve in 2.2b) represents the retained solute concentration in the same cake. If the filter cake has been partially dewatered prior to the washing, the wash curve changes in such a way that the plug-flow plateau diminishes. In some industrial reports, the wash ratio has been replaced by the wash liquid volume divided by the mass of dry solids (Kruger, 1984). This type of wash ratio is used for practical reasons when the interest is in process economics and in the effect of changes of process conditions. Also, the ratio of wash liquid volume to cake volume has been used for visualising the wash curve (Ripperger et al., 2000). The cake washing models and theories in the literature (Wakeman, 1981; Eriksson et al., 1996; Hsu et al., 1999; Kilchherr et al., 2004; Arora et al., 2006;

Tervola, 2006; Arora and Potůček, 2009) focus on the solute concentration in the wash filtrate. Possibly the most used washing model is the dispersion model and its modifications for different washing regimes. The dispersion model for the case where the cake is fully saturated and sorption of the solute onto the solid matter is negligible can be described as follows:

$$\frac{c - c_w}{c_0 - c_w} = 1 - \frac{1}{2}\left\{\operatorname{erfc}\left[\frac{1 - W_R}{2\sqrt{W_R}}\sqrt{D_n}\right] + \exp\left(D_n\right)\operatorname{erfc}\left[\frac{1 + W_R}{2\sqrt{W_R}}\sqrt{D_n}\right]\right\} \tag{2.18}$$

where c is the concentration of the solute in the filtrate, c_w is the concentration of the solute in the wash liquid, c_0 is the concentration of the solute in the liquid in cake voids prior to washing, D_n is the dispersion number and W_R is the wash ratio. The definition of the dispersion number D_n is:

$$D_n = \frac{uL}{D_L} = Re\, Sc\, \frac{L}{d}\frac{D}{D_L} = \frac{\rho u d}{\mu}\frac{\mu}{\rho D} \tag{2.19}$$

where D is the molecular diffusivity of the solute, D_L is the axial dispersion coefficient, d is the particle diameter, L is the cake thickness, u is the superficial fluid velocity, μ is the viscosity of the filtrate and ρ is the density of the filtrate. The wash ratio W_R is defined as:

$$W_R = \frac{V_w}{V_{fo}} = \frac{ut}{S\varepsilon_{av}L} \tag{2.20}$$

The above equations are valid when washing a fully saturated cake with no sorption taking place in the washing process. According to Wakeman and Tarleton (2005), this model can be used in the predictive sense if the properties of the cake and liquid are known. The dispersion model has been further developed for cases in which the diffusion of solute takes place in micro-porous particles (Eriksson et al., 1996). The dispersion model, and its derivatives, are somewhat problematic for use outside of the laboratory. This is mainly due to problems in estimating the axial dispersion parameter and in obtaining the correct value for the molecular diffusivity of the solute. The exponential decay model by Rhodes (1934) is a simple, elegant, model

which describes the solute concentration in the cake (as opposed to the dispersion model, which describes the solute concentration in the filtrate). The exponential decay model is of the form:

$$c_{Rt} = c_{R0} \exp\left(-\frac{kut}{L}\right) \tag{2.21}$$

where c_{Rt} and c_{R0} are the solute concentrations in the cake at time t and at the beginning of the cake washing process, respectively, and k is an experimentally obtained parameter. This model, with a slight modification, has been utilised by Marecek and Novotny (1980) and Salmela and Oja (1999, 2006). They replaced the exponential term with the wash ratio and thus incorporated saturation and porosity into the model, unlike in the Equation 2.21. This modified exponential decay function is as follows:

$$c_{Rt} = c_{R0} \exp\left(-kW_R\right) \tag{2.22}$$

The exponential decay equation has been used successfully to model the removal of ferrous sulphate from hydrated titanium dioxide (Marecek and Novotny, 1980) and the removal of sodium chloride from starches (Salmela and Oja, 2006). The exponential decay model is based on the assumption that the solute concentration of the filtrate is in equilibrium with the solute concentration in the filter cake so that the solute concentration in the wash filtrate is directly proportional to the solute concentration in the cake at that instant.

2.4. Filtration theory and practice

The preceding filtration theories and practical test filtrations tend to be disconnected. This is true especially for cases where the test work is done by filter manufacturers and the primary goal of the test work is to gather data for sales and sizing purposes. In contrast, fundamental filtration research focuses on understanding the filtration sub processes like cake formation, washing and deliquoring phenomena separately and thus the variables used in filtration experiments may be selected and controlled in such a way that

the basic principles governing the phenomena can be revealed. The article by Tiller (2004) discusses this gap between practicalities and theories.

Filter manufacturers use test filtrations as a tool for providing information to their customers and for serving their own sizing and sales purposes, which is why the number of available variables used by manufacturers is typically larger than the number of available variables considered in fundamental filtration research. The larger number of possible variables in practical test filtrations done by filter manufacturers arise from the fact that these test filtrations have the complete filtration cycle under consideration as opposed to the one sub process typically studied in fundamental filtration research. Test filtrations can be divided into preliminary-, sizing- and pilot-scale tests. In some cases, the preliminary tests already provide enough information for sizing purposes, whereas pilot-scale tests are used for finding the optimum operational parameters for the current application. Sales and sizing test work is often done with pilot scale filters which mimic the production size filters in their operation.

There are other software packages that combine filtration theories and experimental data, for example Filos(Nicolaou, 2003) and FDS(Tarleton and Wakeman, 2007). The experimental data used as an input is basic filtrate accumulation data. The Filos and FDS packages are used mainly for analysing the filtration data from view point of the filtration theories. FDS also incudes an automated method for selecting filter types (for example, filter-press, belt-filter etc.) and this relies on the filtration theories, but in doing so the testing of variables that affect cake properties during the filtration sequence, say compression pressure or time, cannot be taken into account in predicting the filtration outcome. Another software package that has been used in analysing filtration tasks is DynoChem (Sparks, 2010). The DynoChem software requires that the user is familiar with the filtration theories so that he/she is able to input the appropriate filtration equations into the system so that it can perform parameter fitting for the entered equations.

Filtration theories can be used successfully when inspecting the subprocesses of a filtration cycle but they are not easily applicable when the complete filtration cycle needs to be modelled, as is often the case with industrial filtration problems. To overcome the problems in combining the filtration subprocess theories, the statistical design of experiments and empirical modelling are needed.

Fitting together filtration theories and practical filtration problems is not an easy task. The essential problem is that there is no direct link between the theories, so, for example, the information that the cake growth theories give cannot be connected into the theory of cake compression. Using filtration theories always requires experimental work. Strict scientific experimental work is regarded as too laborious and time-consuming from the practical point of view and the straightforward experimental work carried out by the filtration practitioners, from the viewpoint of theoreticians, lacks in finesse and overrides many important aspects of filtration phenomena. Nevertheless, practical filtration problems need to be studied and empirical modeling accompanied with statistical design of experiments offers a plausible approach method.

3. Design of experiments

The statistical design of experiments is a logical construction which enables one to gather maximum benefit from experimental activities. Here, the experimental activities are for recognising the important factors in solid/liquid separation processes.

The statistical design of experiments is used in a wide variety of experimental research, including filtration. However, within the filtration studies the scope of the statistical design of experiments has been mostly on the optimisation of existing filtration processes (Herath et al., 1989, 1992; Stickland et al., 2006), studies of filtration subprocesses (Tosun and Şahinoğlu, 1987) and in searching for the effects of upstream process variables (Togkalidou et al., 2001). One study, with a similar approach to this work, is described in Sung and Parekh (1996), though the reliability of this study is somewhat problematic since it includes variables that are not independent, like filtration time, solids concentration and cake thickness.

The process studied always involves inputs, controllable variables, uncontrollable variables and responses. In a solid/liquid separation process the variables can be material related or filter type related. Typical material related variables are, for example, slurry density, temperature, pH and particle size. Filter type related variables include: the selection of the stages to be included into filter cycle, pressure differences used in various stages of the complete process cycle, times used for separate subprocesses and wash liquid amounts used in washing.

It is essential that the variables are uncorrelated and independent in respect to each other and thus the experimental design matrix is orthogonal.

The experimental procedure involves changing the variables in order to see what effect these changes have on the response value and thereby gathering information on how the process behaves. If the process is robust and contains just a few controllable variables and the ad hoc information suffices then a best guess or 'one variable at a time' (OVAT) approach might provide enough information. However the best guess and OVAT strategy do not

provide enough data for modeling purposes, since they fail to consider the possible interaction between the variables (Box et al., 2005). The OVAT strategy is also dependent on the starting point of the experiments and may lead to different end results if the starting point, or the order of the variables, is changed. The starting point for the experiments is the initial combination of the variable values. This type of testing is sometimes used to assess the importance of variables in influencing the response, but even this can be impossible if the variables jointly influence the response (Mason et al., 2003). Statistical design of experiments methods that are able to provide enough data for modeling purposes include factorial-, fractional factorial-, Placket-Burman- and Taguchi designs (Croarkin et al., 2010). These designs are basically two level designs where variables are given only two values (namely low and high values). Two level designs, such as these, can only be used to fit linear models.

When it is suspected that the response function is nonlinear, it is advisable to use experimental design methods that have more than two levels for variables. Examples of experimental designs containing more than two levels are the Central composite design and Box-Behnken design (Box and Draper, 1987). These designs are called response surface methods. There are other experimental design methods, such as optimal design, Doehlert and supersaturated designs.

Optimal designs are used if there are constraints for experimenting, such as a limited number of runs, impossible factor combinations, too many levels or a complicated underlying model. The advantage of optimal designs is that they do provide a reasonable design-generating methodology when no other mechanism exists. The disadvantage of optimal designs is that they require a model from the user (Croarkin et al., 2010).

Doehlert designs are for treating problems where specific information about the system indicates that some variables deserve more attention than others. Compared to central composite or Box-Behnken designs, Doehlert designs are more economical, especially as the number of factors increase (Bruns et al., 2006).

Supersaturated design is a form of fractional factorial design in which the number of variables is greater than the number of experimental runs. This type of design would be useful when costs of experiments are expensive, the number of factors is large and there is a limitation on the number of runs

(Yamada et al., 1999). Though the supersaturated designs are appealing as the number of experimental runs is small, one should be very cautious in using these designs routinely (Mason et al., 2003).

There are plenty of different methods that can be used to create experimental designs. When starting this work, it was unclear what level of experimental design should be used and how the filtrations could be modeled. In this work the factorial and fractional factorial experimental designs were selected because these experimental design methods are robust, well documented, the basic structure of these designs is easy to understand and fractional factorial designs are relatively simple to augment, if needed.

The general guidlines for designing an experiment are, according to Montgomery (1997), as follows:

1. Recognition and statement of the problem
2. Choice of factors, levels, and ranges
3. Selection of the response
4. Choice of experimental design
5. Performing the experiment
6. Statistical analysis of the data
7. Conclusions and recommendations

The recognition and statement of the problem is restricted in this work to solid/liquid separation processes. The choice of variables and ranges is left to the experimenter, as is the selection of the responses. The selection of variable ranges is something that requires that the experimenter has knowledge of the filtration equipment type and one or two preliminary tests. As a result, the selected variable levels cover the practical working range of the filter type in use. Level selection and the type of the experimental design is built in the LabTop software. Statistical analysis is also carried out by the software. Finally the conclusions and recommendatios are left to the experimenter.

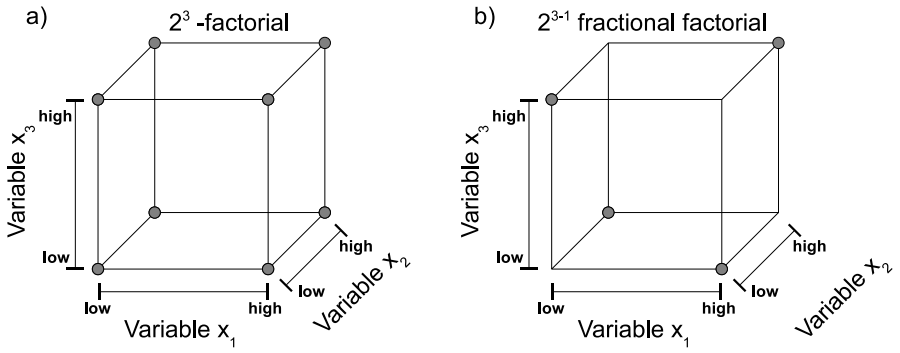

Figure 3.1.: a) Factorial and b) fractional factorial designs for three variables.

3.1. Factorial designs

Factorial designs can be regarded as the foundation of the experimental designs. The 2^k factorial designs are two level designs, meaning that the variables are given low and high values. For example, a two level factorial design with three variables is termed a 2^3 design. The geometrical interpretation of a three variable 2^3 design, as shown in Figure 3.1 a), is a cube where each corner represents a combination of the variable values to be used while experimenting. The 2^3 factorial design is composed of eight experiments (shown at the corners of the cube). Factorial designs can be very demanding, in terms of the number of experiments needed if the number of variables becomes high, for example the four variable full factorial requires $2^4 = 16$ experiments, five variable factorial $2^5 = 32$ experiments and so on. Factorial designs reveal the interactions between selected variables and the main effects of each individual variable. It is only possible to create linear models from two level factorial designs. General linear models represent planes in space.

3.2. Fractional factorial designs

Fractional factorial designs are subsets of the full factorial design as can be seen in Figure 3.1 b). These fractional designs are used when the number of experiments in full factorial designs become too large and some of the variable interaction terms can be neglegted. For example, with four variables the full factorial design requires 16 experiments, but if this is regarded as too high and reducing the number of variables is out of the question, then it is possible to construct a experimental design for four variables having only eight experiments. The experimental design table of a 2^{4-1} fractional design is shown in Table 3.1 and the graphical interpretation of the same design is shown in Figure 3.2.

Table 3.1.: 2^{4-1} fractional factorial design

Variable	x_1	x_2	x_3	x_4
Run				$x_1x_2x_3$
1	-	-	-	-
2	+	-	-	+
3	-	+	-	+
4	+	+	-	-
5	-	-	+	+
6	+	-	+	-
7	-	+	+	-
8	+	+	+	+

The fractional factorial design, Table 3.1, shows how the 2^{4-1} fractional factorial design is basically created from a 2^3 full factorial design. The levels for the fourth variable, x_4, are created by the simple multiplication of variables x_1, x_2, and x_3. This multiplication operation implies that the combined interaction of these three variables cannot be revealed anymore and the resolution of the experimental design becomes reduced.

Creating fractional factorial designs always means that some interactions are sacrificed in order to minimise the number of experimental runs. Table 3.2 shows a 2^{7-4} fractional factorial design where the variables from x_4 to x_7 are confounded with interactions. Let us look at the variable x_4, which is confouded with x_1x_2, now x_4 is aliased with x_1x_2 and this interaction cannot

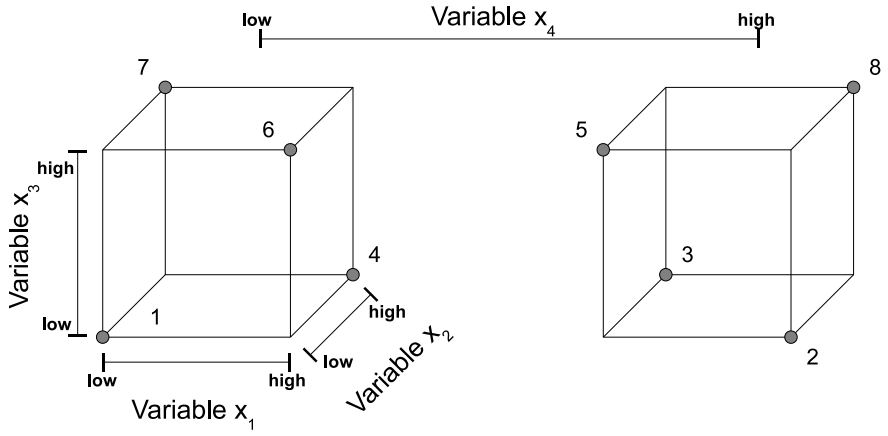

Figure 3.2.: Graphical interpretation of 2^{4-1} fractional factorial design

be estimated separately. x_1x_2 is said to be generator word. The danger in using heavily confounded fractional designs, like in Table 3.2, is that, in the worst case, what looks to be an effect of the variable x_4 is actually the effect of the combination of the main effect of x_4 and the two-factor interaction involving x_1 and x_2.

Resolution indicates the ability of the design to separate the main effects and interactions. The meaning of the most prevalent resolution levels is as follows: (i) Resolution III Designs where main effects are confounded (aliased) with two-factor interactions. (ii) Resolution IV Designs where no main effects are aliased with two-factor interactions, but two-factor interactions are aliased with each other. (iii) Resolution V Designs where no main effect or two-factor interaction is aliased with any other main effect or two-factor interaction, but two-factor interactions are aliased with three-factor interactions (Croarkin et al., 2010). From this it follows that the experimental design in Table 3.1 is a resolution IV design and in Table 3.2 is a resolution III design.

There are different levels upon which to create these subsets. The levels are half- quarter- 1/8- and so on, depending on the number of the experimental runs compared to a full factorial of the selected variables. In Table 3.2, the 2^{7-4} fractional factorial design is a 1/16 fraction design, because there are eight experimental runs whereas a full factorial design for seven

variables requires $2^7 = 128$ experimental runs and $8/128 = 1/16$.

Table 3.2.: 2^{7-4} fractional factorial design

Variable	x_1	x_2	x_3	x_4	x_5	x_6	x_7
Run				x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$
1	-	-	-	+	+	+	-
2	+	-	-	-	-	+	+
3	-	+	-	-	+	-	+
4	+	+	-	+	-	-	-
5	-	-	+	+	-	-	+
6	+	-	+	-	+	-	-
7	-	+	+	-	-	+	-
8	+	+	+	+	+	+	+

Factorial designs allow for the estimation of many higher order effects (Box et al., 2005). As can be seen from Table 3.2, even the factorial design originally for three variables can be developed for estimating the main effects for seven variables; however, it should be stressed that it is done at the cost of losing interaction effects. However, if the assumption is that the three variable interactions can be ignored then it should be remembered that 16 run design provides 3 three-factor interactions and one four-factor interaction.

Creating fractional factorial designs, as stated earlier, always requires compromises between separating the main effects and interactions. In order to create a fractional design, one should know what interactions are aliased. There are three design criteria, namely the maximum resolution design criterion, the maximum unconfounding design criterion and the minimum aberration design criterion. In this work the minimum aberration criterion was used and it is defined as the design of maximum resolution "which minimizes the number of words in the defining relation that are of minimum length" (Fries and Hunter, 1980). Rephrasing this means that the longest generator words are used first and the two variable interaction words are used only if absolutely necessary.

When applying two level factorial and fractional designs into completely unknown processes it must be remembered that these designs come with the assumption of linearity. It is possible to include some curvature into

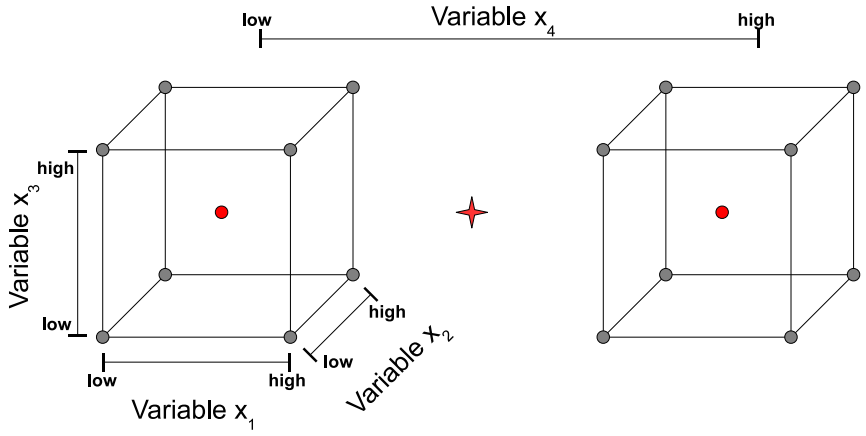

Figure 3.3.: 2^4 factorial design augmented with three points, one of being a center point.

response model by applying interaction terms into model function (Montgomery, 1997). By adding center points into the factorial designs it is possible to verify whether or not the linearity assumption is valid. Figure 3.3 shows an example of a 2^4 factorial design augmented with three points and one of them is a center point.

3.3. Response surface methods

Response surface methods are used when more detailed information about the response behaviour is needed. The Response surface method can be used to create experimental designs when there is a need to model higher degree polynomials and other nonlinear functions as well as for linear functions with interactions. These designs are most effective when there are fewer than 5 factors. Quadratic models are used for response surface designs and at least three levels of every factor are needed in the design (Croarkin et al., 2010). Figure 3.4 shows Central Composite and Box-Behnken designs for three variables. Both of these designs explore the same variable range but it is noteworthy that the Box-Behnken design does not utilise the extreme variable combinations. This is convenient, especially if the examined process

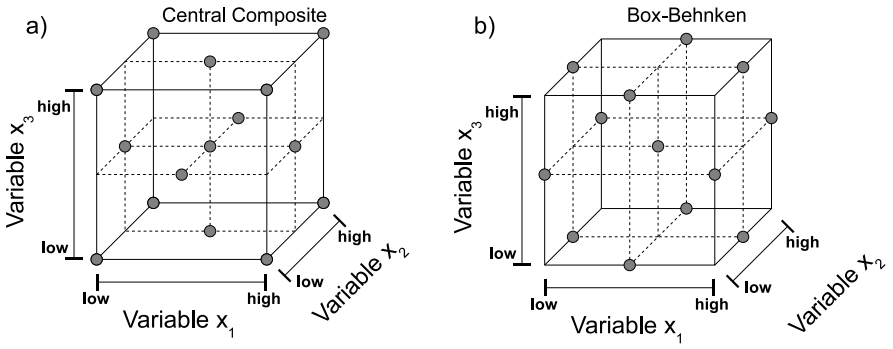

Figure 3.4.: a) Central composite and b) Box-Behnken designs for three variables

is, for example, an industrial process where setting extreme values for the variables simultaneously is difficult, if not impossible, to realize. To be more precise, the Central Composite shown on Figure 3.4 a) is one of the three variants of the Central Composite designs, namely the Face Centered Central Composite (CCF) the other two variants are Circumscribed Central Composite (CCC) and Inscribed Central Composite (CCI) (Croarkin et al., 2010). The diffenece between the different CC designs is shown on Figure 3.5. CC designs contain factorial or fractional factorial design, which is augmented with points that allow more precise estimation of the response behaviour when compared to pure factorial designs.

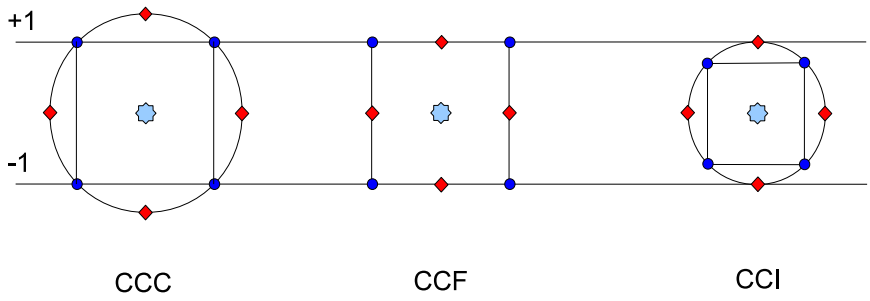

Figure 3.5.: Difference between the CCC, CCF and CCI designs. Blue dots are factorial design points and the red dots are the augmented design points. Adapted from (Croarkin et al., 2010).

Response surface methods are often used in optimisation problems. When there is a need to find optimum solutions experimentally, the Box-Wilson strategy is one of the most famous methods (Box and Wilson, 1951). This method is an iterative procedure where factorial or fractional designs are used to search for the steepest ascent or descent to find the region close to the optimum. New experimental design is created using response surface methods to study whether or not the optimum is within the tested area.

4. Modeling of the experimental results

After the experimental designs have been created and the experiments have been performed, the experimenter is faced with a large amount of data that needs to be analysed. Modeling is therefore essential in extracting information out of the experimental data, since the data in itself does not provide any means for interpretation of the measured response.

A mathematical model gives us a description of the response behaviour with changing variable values. When the mathematical model has been created, further investigations can be done by simulation. Mathematical models can be divided into two groups, namely empirical models and mechanistic models. The level of knowledge within the studied and modelled system is decisive in categorising the models. If the theories behind the studied phenomena are well established and known, but the parameters are unknown, then the model is said to be mechanistic or a first principles model, otherwise the model is empirical.

Process models, whether empirical or mechanistic, are used for estimation, prediction, calibration and optimisation (Croarkin et al., 2010). Estimation of regression function values gives value of a response variable for a particular combination of predictor variables. Prediction also gives a value of a response variable for a particular combination of predictor variables, but the prediction includes the noise that is inherent in the parameters, the uncertainty of new measurement and other error sources. Calibration is to quantitatively relate measurements made by different measurement systems. Optimisation is for the determination of process inputs to obtain a desired process output. In addition to these, visualisation is also an important application of process models.

Before the mathematical model is created with regression, it should be considered whether the variables should be coded or standardised. Variable coding means that the variable values are brought into numerically compar-

able ranges. This coding is often done by subtracting the mean and dividing by the standard deviation for each variable (Hair et al., 1998). The motivations for coding are for numerical or optimisation reasons. The numerical reason for coding is that, without coding, calculation of the regression coefficients may lead to a system of equations that are difficult to solve. The optimisation using the steepest ascent method with regression models created without coded variables is prone to errors. This is because the gradient is dependent on the variable scale. However, coding of the variable levels is not necessary if the model to be created is linear, without interaction terms, or if the model is used 'as is' and it is not used to design new experiments into the direction of steepest gradient.

4.1. Multiple linear least squares regression

Multiple linear regression (MLR) is the basic tool to be used whenever the variables are controllable and linearly independent i.e. no variable is a linear combination of some other variable. MLR is especially suited to data, that have been gathered from a controlled series of experiments and when there are at least as many experiments as there are variables. Linear regression is often understood to contain models that describe either straight lines or planes.

Linearity Let us suppose that we have two arbitrary variables x_1 and x_2 which relate to the response y with the following equation:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \epsilon \tag{4.1}$$

This equation has a plane as the response surface. Now if we add the interaction term of the two variables $x_1 x_2$ into the Equation 4.1, it becomes:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \epsilon \tag{4.2}$$

The response surface is no longer a plane but it has some curvature within it. Now we add quadratic terms into Equation 4.2 and get the following:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 + \epsilon \tag{4.3}$$

This Equation 4.3 has a response surface containing second-order curvature for both variables. If we let $x_3 = x_1^2$, $x_4 = x_2^2$, $x_5 = x_1 x_2$, $\beta_3 = \beta_{11}$, $\beta_4 = \beta_{22}$, and $\beta_5 = \beta_{12}$, then Equation 4.3 becomes:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_5 + \epsilon \tag{4.4}$$

which is a linear regression model. Similarly, Equation 4.2 can be turned into a linear model.

The key to understanding the linearity of the Equations 4.2 and 4.3 is to look at the parameters β. In general, any regression model that is linear in the parameters is a linear model regardless of the shape of the response surface that it generates (Montgomery, 1997).

Least squares Multiple linear regression uses the least square method in estimating the coefficients for the variables. In least squares estimation, the unknown values of the parameters β, in regression functions like equations 4.1 to 4.4, are estimated by finding numerical values for the parameters that minimise the sum of the squared deviations between the observed responses and the functional portion of the model. More detailed desriptions of least squares estimation can be found in (Montgomery, 1997; Box et al., 2005; Croarkin et al., 2010).

4.2. Non-linear least squares regression

The nonlinear least squares regression, as the name suggests, is a method that is used in modeling nonlinear response functions. The parameter estimation is done using the least squares method, but the model is non-linear with respect to the parameters β. An example of non-linear model is:

$$y = \frac{\beta_0 + \beta_1 x_1}{1 + \beta_2 x_2} + x_3^{\beta_3} \tag{4.5}$$

Note that the parameters β can't be brought into linear form.

4.3. Other modelling methods

In addition to MLR, there are other modelling methods like Principal Component Regression (PCR), Partial Least Squares (PLS) and Ridge Regression (RR). These methods form a small part of the techniques presented under the scope of chemometrics. The definition of chemometrics according to Massart et al. (1997) is "chemical discipline that uses mathematics, statistics and formal logic (a) to design or select optimal experimental procedures; (b) to provide maximum relevant chemical information by analyzing chemical data; and (c) to obtain knowledge about chemical systems". These methods are suitable especially if the data to be used contains either colinearities or some data is missing. The interested reader is recommended to familiarise his or herself with the following references Höskuldsson (1996), Massart et al. (1997) and Vandeginste et al. (1998).

4.4. Statistical parameters

There are some simple statistical parameters that can be used as measures of the quality of the model, to estimate the validity of the variables and to estimate the quality of the experimental data point. Perhaps the most used statistical parameter that is used for obtaing information on how well a model fits is the coefficient of correlation denoted as R^2. The coefficient of determination tells how much of the variation in the observed response is explained by the regression model and it is defined as:

$$R^2 = 1 - \frac{\sum_i (y_i - \hat{y}_i)^2}{\sum_i (y_i - \bar{y})^2} \tag{4.6}$$

where y_i are the measured response values, $\hat{y}_i$ are the estimated response values and $\bar{y}$ is the mean of the measured response values.

R^2 is given as either a percentage or numerical value in the range from 0 to 1, with larger values being more desirable. However it should be re-

membered that pursuing ever higher coefficient of determination values is somewhat risky because of overfitting. Overfitting is a situation in which the experimental error becomes a part of the model. The R^2 value alone does not tell whether the model is good or not. The coefficient of determination, Q^2, for the test set, is calculated with the same equation as R^2, but it is applied to those experimental points that have not been used in model creation.

NOTICE: In the attached Publications III and IV there is an unfortunate mistake! The term 'correlation coefficient' is used where 'coefficient of determination' should have been.

A residual or fitting error is the difference between the measured and estimated value. Residual values are used to visualise the model fit and thus they give valuable information on the goodness of the model.

The standard error of estimate (SEE) is a standard deviation of the residuals and it is of the form:

$$SEE = \sqrt{\sum_{i=1}^{N} \frac{(y_i - \hat{y}_i)^2}{N - p}} \tag{4.7}$$

where N is number of datapoints, p is number of parameters in the regression model.

The root mean squared error (RMSE) and root mean squared error of prediction (RMSEP) are used to get an overview of the model suitability.

$$RMSE = \sqrt{\sum_{i=1}^{N} \frac{(y_i - \hat{y}_i)^2}{N}} \tag{4.8}$$

and

$$RMSEP = \sqrt{\sum_{i=1}^{N} \frac{(y_i - f_i)^2}{N}} \tag{4.9}$$

where f_i is the predicted response value.

Decisions on whether the variable is valid or not are often done by applying hypothesis tests. The statistical hypothesis is formulated so that the hypotheses are mutually exclusive i.e. if one is true then the other is false. The null hypothesis can, for example, be that observations are purely from random chance and alternative hypothesis is that observations are due to non-random chance. In the case of MLR, the hypothesis tests can be applied to regression coefficients. The errors committed in hypothesis testing are either a Type I error when the null hypothesis is rejected when it is true, or Type II error where the null hypothesis is not rejected, even if it is false. The probability to commit a Type I error is often called the significance level of the test. Sometimes a power of the test is used. The power of the test is the probability of not committing a Type II error (Montgomery, 1997).

The significance, or P-value, is the probability that an effect at least as extreme as the current observation has occured by chance. P-values can be calculated for the whole regression model and for each of the parameters. Conventionally the significance level 0.05 is used; if the P-value for the whole model is greater than 0.05 then, statistically speaking, the model is no better than the model $y = constant$. This is, of course, an extreme example but the significance value is important when considering the goodness of fit. The P-values for model parameters show in a similar way whether the variable associated with the parameter is statistically significant. For example if the model equation 4.1 with three parameters is used for modeling and parameters β_0,β_1,and β_2 are given P-values of 0.007, 0.2, and 0.02 respectively, then parameters β_0 and β_2 are statistically significant and based on that the model could be written in the form:

$$y = \beta_0 + \beta_2 x_2 \tag{4.10}$$

If the linear regression for the equation 4.10 was based on orthogonal experimental design, then removing terms from the model does not change the parameter values, but the P-values should be recalculated because changing the model function causes a change in residuals.

P-values are provided to the user as additional information and the significance level 0.05 is used, because of convention.

A simple test for curvature, that can be applied, is calculating the mean value of the measured response values from the factorial design points and

then compare this mean value to the measured response value obtained from the center point (Box and Draper, 1987). This test is not yet applied into the software.

The starting point for this study was to create mathematical models that are easy to understand, and use, by the personnel who do not have prior experience of statistics and modelling. This works as a constraint to the statistical information that is to be given to the user. The empirical models are limited to linear functions because it was found that these models provide accurate enough information about the system under consideration. Model functions created for test filtrations describe the local variable-response interaction with an accuracy that is sufficient for decision making, which is one of the main goals in test filtration. Test filtrations are used as stepping stone for sizing and design work and so these tests provide information for designing new filtration processes for previously unknown slurries. The main questions that needs to be answered after completing the test filtrations are: What is the operational window of the filter with the tested slurry and how do the variables affect the filtration outcome?

Part II.

Software and results

5. LabTop software

The LabTop software package has been developed for test engineers, for example working for filtration equipment suppliers or researchers. The aim is to create properly structured experimental designs which can provide the most information about a filtration application with minimal work load. The software has been divided into two separate modules, LTDoE and LTRead.

The prerequisites for this software were: (i) To be easy to use and requiring no experience of statistical modelling or experimental design. (ii) To provide statistical information in a form that is easy to understand. (iii) To provide information in visual form. (iv) To make reporting easier.

One of the biggest decisions was to leave out the replicate points. This decision is compensated partly by augmenting the experimental designs with additional runs. The factorial and fractional factorial designs have been augmented with points that are within the variable range. An example of these augmentation points are shown in Figure 3.3.

5.1. Design of experiments

The LabTop design of experiments module, LTDoE, is applicable to a number of different filter models.

It is used to create experimental designs for the given filter type. As an output, LTDoE writes an Excel file which contains the design. Here the experimental design means the set of experiments that are to be carried out, in the same order as they are presented in the Excel file. After the experiments have been carried out, the results will be written into the same excel file as the original design. The user may write as many responses as are needed. Typically the measured responses are capacity of the filter and the moisture content of the cake.

The experimental design that is written in the Excel file is either full factorial or fractional factorial, depending on the user selection. The designs

have been augmented with a set of points of which one is a true center point. An example of augmentation points for a four variable design is shown in Figure 3.3. The design is augmented with one true center point and additional points. The number of augmentation points are done in the following manner: if number of variables is from one to three, only a center point is added, for four and higher, the number of augmentation points, including the center point, is calculated using $n_{ap} = 2(n_v - 3) + 1$, where n_{ap} is number of augmentation points and n_v is number of variables. The reason to use this kind of design augmentation is that it gives the center point and additional variation around the center without replication.

5.1.1. LTDoE basics.

When starting the design of experiments module, the software displays two windows. The first window to appear is the dos-box which shows some debugging and additional information. The actual user interface for the software starts with the filter type selection window, as shown in Figure 5.1.

Figure 5.1.: The filter type selection dialogue and the filtration cycle definition window used with PF, DS and MFP filters.

By clicking one of the push-buttons on the filter type selection dialogue, the user is prompted with a new window, as shown in Figure 5.1. The next window to be shown to the user is filter type dependent. As an example here, the user has selected the one sided pressure filter (known as PF). Once the user has selected PF, DS or MFP filter, the next stage is to define the filtration cycle.

The user can define freely the filtration cycle to be used during the experiments. The default cycle consists of pumping (i.e. slurry filtration), squeezing and drying stages (thus the coding ABD). As shown on the Figure 5.1 it is also possible to create cycles that contain multiple pumping, squeezing, washing and drying stages. The example cycle ABCBD represents: pumping, squeezing, washing, a second squeeze and drying.

Based on the filtration cycle, the program will create a table containing the set of possible variables to be used during the experiments as shown in Figure 5.2.

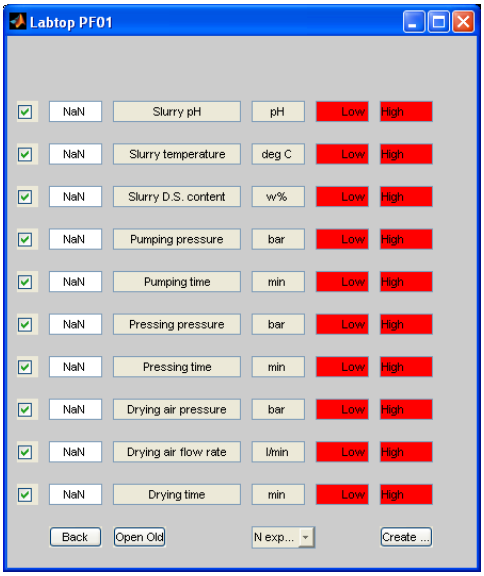

Figure 5.2.: The set of possible variables to be used during the experiments. This set of variables is based on an ABD filtration cycle i.e. pumping, squeezing and drying.

Despite the exhaustive list of possible variables, the user may now select only those variables which are judged to be important to the case in hand. The variable table structure, from left to right, is as follows:

- A checkbox indicating whether the variable is to be held constant during the experiments or not. If the checkbox is marked then the current field is to be held constant.
- Into the first edit box, the user may write the value for current constant (although this is not obligatory). If the background color is white then the current field is constant and the value is written into the experimental design.
- Constant or variable name field. The variable names are freely editable.
- Constant or variable unit edit box. The units can be edited according to the user's preferences.
- Two variable range edit boxes. If the background color is white the edit box on the left is reserved for the lower limit and the edit box on the right is for the upper limit value. A red background color indicates that the field is constant and that the values in these boxes are not used.

By default, all of the variables are marked initially as constants for the experimental design. Unmarking the cross from the checkbox makes them variable.

Now the user is ready to select the variables and the variable ranges according to the plan he/she has decided upon in advance. Figure 5.3 shows an example case of experimental design.

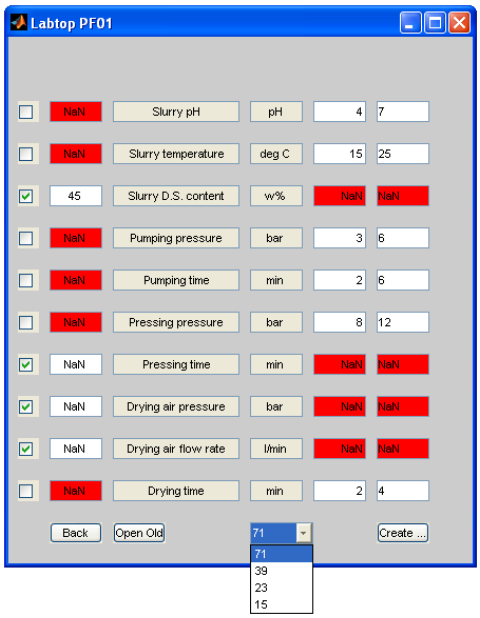

Figure 5.3.: An example case for experimental design.

Note that there are in total six variables selected and, therefore, the user may select the number of experiments from the drop down menu. In this example case (Figure 5.3), there are four different types of experimental design available (see Chapter 3) and the number of experiments that the user has to do is 71, 39, 23 or 15. Now it is for the experimenter to decide how many experiments he/she is ready to do. Even if it is very tempting to select the smallest number of experiments, one should be very careful when doing this. Selecting the the absolut minimum number of experiments is done with the cost of losing the interaction effects (see Chapter 3.2). The number of experiments in the menu comes from the two level factorial design schemes plus some center points. The first number of experiments that is presented is always the so called full factorial and the following numbers represent the fractional factorial designs starting with $\frac{1}{2}$-factorial and then smaller fractional designs, like $\frac{1}{4}$, and so on. The smallest value presented on the drop down menu is the absolute minimum number of experiments that are needed to evaluate the effect of the selected variables on the response. When

the user has selected the variables, filled the table and selected the number of experiments, the user can create the experimental design by clicking the 'Create Plan' button on the lower right.

There is one possible side step that is dependent on the variables the user has selected - slurry variable ranking. Looking at the variables selected in Figure 5.3, there are two slurry variables selected; slurry pH and slurry temperature. The user is prompted with the dialogue, shown in Figure 5.4, which prompts the user to select the order of these variables in decreasing order of difficulty. Based on the slurry variable ranking, the experimental

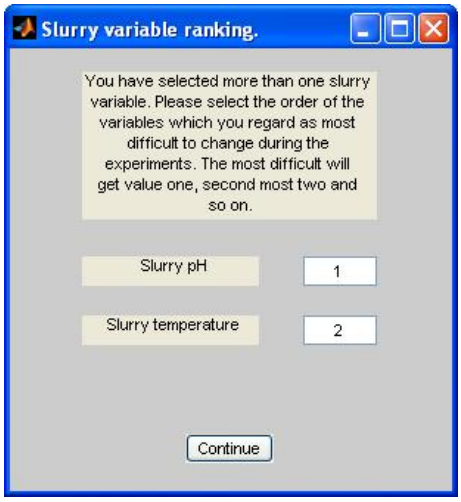

Figure 5.4.: Slurry variable ranking dialogue.

design table is divided into blocks that minimize the workload of the experimenter, while changing the slurry characteristics between the experiments.

The final dialogue the user will use when creating the experimental design is the standard file save dialogue. The user is allowed to select freely the file name and the directory where the file will be written. The design of experiments are written into an Excel '-.xls' file. The experimental design table contains the constant terms and an outline of the experiments that are meant to be carried out, in the order that they are written. Finally, the user will see the progress bar that indicates the file saving procedure status. When the progress bar is totally filled the software closes down and

the experimental design is written into the file and location the user has specified.

5.1.1.1. LTDoE and ceramic capillary action disc filter

The usage for the Ceramec filter differs slightly from that previously presented. The main difference is that there is no cycle definition available and there are two mandatory values that are needed. The absence of the cycle definition is a consequence of the action of the Ceramec filter. A precise description of testing procedure used with ceramic disc filters is presented in Paper I. The mandatory values are rotation time and slurry level. These values are needed even if they are not included as variables in the experimental design. This is because they govern the dipping and drying times used in leaf tests. Furthermore, the Ceramec type needs to be selected. There are three size ranges, namely 'Pilot CC-1', 'CC-6 to CC-60' and 'CC-96, CC-144'. The pilot CC-1 having 1m disc diameter, 'CC-6 to CC-60' types having a disc diameter of 1.9 m and 'CC-96, CC-144' a disc diameter of 3.8 m. It is mandatory to use seconds as the unit for rotation time. The slurry level is given as mm from the centre of the shaft. The available variable set for Ceramec filters is shown in Figure 5.5.

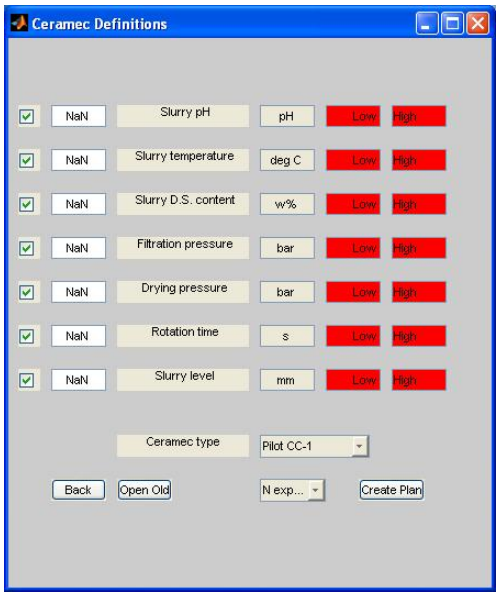

Figure 5.5.: Variable set for ceramic capillary action disc filters.

5.1.1.2. LTDoE and experimental filter

In the filter type selection dialogue (Figure 5.1) there is an "experimental" filter type available. This option creates an experimental design for cases where the variables of interest are not those that are used normally. When the user selects the experimental filter he/she is first shown a dialogue (Figure 5.6) where the user defines the number of variables he/she is going to use while experimenting. After the user has selected the variables, the variable definition table for experimental filter is shown (Figure 5.6). It is noteworthy that the variable names, as well as the units, can be edited according to user preferences. As shown in Figure 5.6, the slurry variables are available by default but it is not necessary to include them as variables into the experimental design.

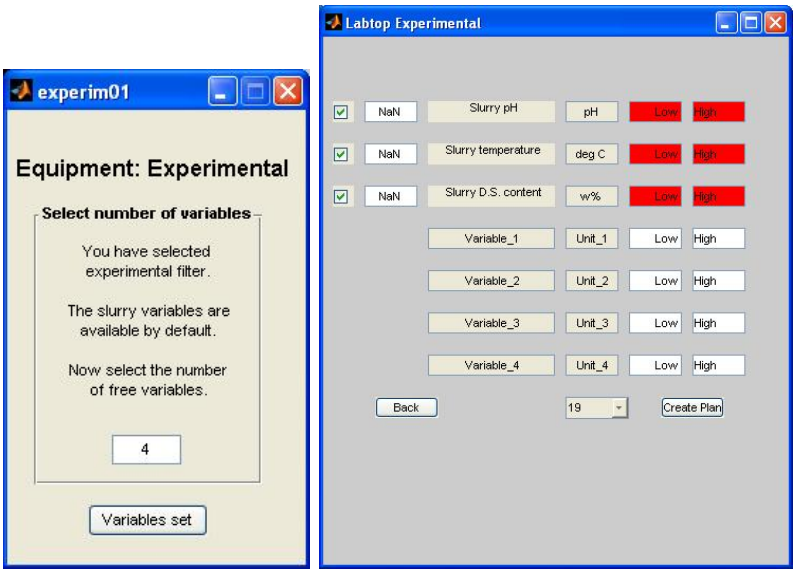

Figure 5.6.: Selection of number of variables and variable definition table for the experimental filter.

5.1.1.3. Opening previously created plans.

The software allows the user to open previously created experimental designs with a press of a 'Open Old' button. There is a field called 'Variable order:' in the experimental design Excel file (Figure 5.7). This 'Variable order:' field tells in which positions the variables are in the original design and this is the way that the variables are positioned correctly, even if the variable names have been changed from the original suggestions. 'Open Old' leads to a "select file to open" dialog in which the user can browse and eventually select the experimental design that has been created earlier.

5.1.1.4. Writing the results

The output from the LTDoE is an Excel file with the following structure: On 'Sheet1' (Figure 5.7) there is the complete experimental design and on 'Sheet2' there is a copy of the design table and space for writing the response variables, for example capacity and cake moisture content. The first cells on

'Sheet1' contain important values for the LTRead module. Using the Excel notation the values in the cells are; A1 number of variables, B1 number of constants, C1 number of experiments, and D1 the filter type (see Figure 5.7). Normally the user does not need to change these values at all. The only case where the user may modify these values is when the user has done more experiments than is advised on the original experimental design. In that case, the user adds those extra experimental points to 'Sheet2' and corrects the number of experiments value in cell C1. Otherwise it is not necessary to modify these previously mentioned cells.

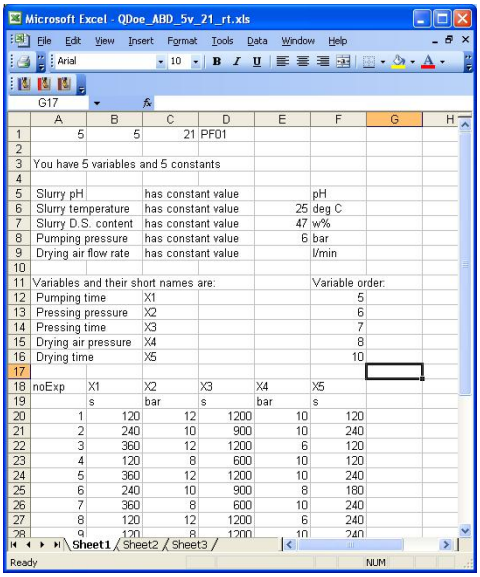

Figure 5.7.: Experimental design created with LTDoE.

Figure 5.8 is an example case, showing how the experimental data should be inserted on 'Sheet 2'. Notice that the experimental values look different compared to experimental design. This difference is due to the basic idea of design of experiments; that it should be regarded as a guideline to experimental work. Therefore it is vitally important that the real values are recorded as the experimental values. By recording the real values that were used in the experiments, the user helps to minimize the model error and, therefore, the mathematical model becomes more precise.

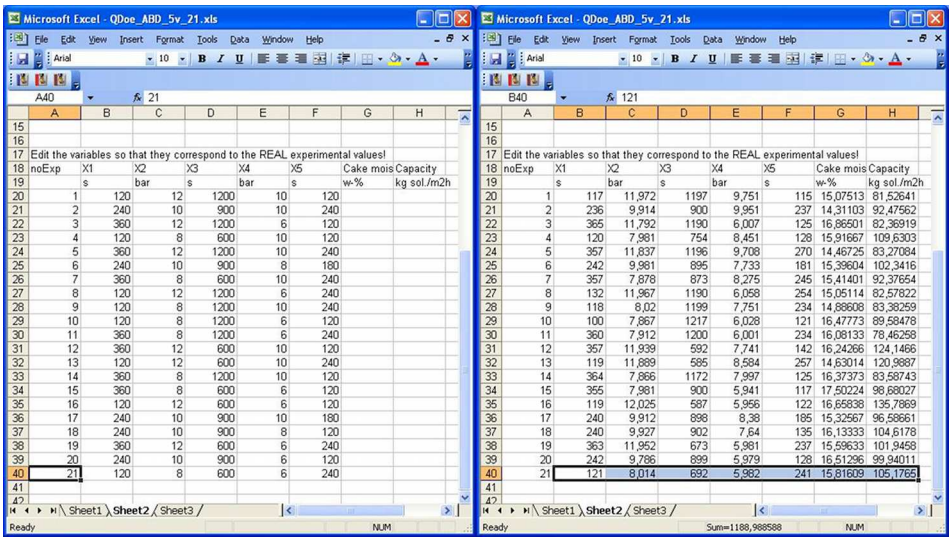

Figure 5.8.: The left-hand side shows an experimental design prior to the experiments and on the right the same table after the experiments. Note that the experimental design is taken as a guideline not as absolute truth.

On 'Sheet2' there are two responses available: 'Cake moisture' and 'Capacity'. These responses have been written on to the 'Sheet2' just to show where the responses should be written. If the user does not need these responses then he/she should just replace these with responses of choice. The important thing is that during the next phase 'LTRead' will start reading the names, units and values of responses starting from the same column where 'Cake moisture' currently is. Furthermore, the number of measured responses on 'Sheet2' is not constrained at all. There can be as many responses as the user chooses to have. For example, if the user has measured just one response, say 'Capacity', then he/she just deletes the cells containing 'Cake moisture' and 'w-%' and moves the text 'Capacity' and unit 'kg sol./m2h' into those cells.

5.2. LTRead modeling and visualisation

The LTRead module reads in the data from a completed experimental design set and creates models for each of the measured process responses. It also provides a graphical user interface for the creation of variable vs response plots. These plots can then be used for reporting purposes.

The LTRead module is used to calculate the model of the measured responses, using the Excel file where the results of the experiments have been written as an input. Based on the input file information, LTRead models the measured responses and produces graphs for the model and gives the user the opportunity to create additional graphs. The LTRead module can also create an auto generated report which contains all of the additional figures the user has created.

5.2.1. Basic usage

When the test engineer has completed the experiments, according to the plan presented in the experimental design file, and has written the results of the responses to the same file, on 'Sheet2', he/she is ready to use LTRead. As with the LTDoE module the user is first greeted with a dos-box and then after a while there will be the starting window (Figure 5.9).

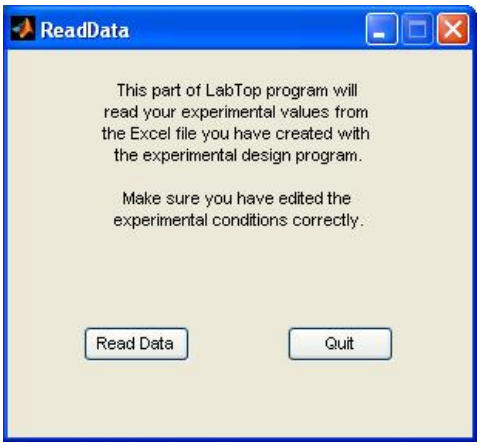

Figure 5.9.: The starting window for LTRead module.

The starting window provides a reminder that the user should always write the real experimental conditions on to the experimental design 'Sheet2' along with the measured responses. After the user clicks on the 'Read Data' push button the file open dialog opens. The user should browse to the location where the experimental plan is located and select the file he/she completed after the experimental work.

When the appropriate file has been selected the user will see the progress bar that indicates the status of reading. Immediately after the experimental data has been read it will be analysed and the results will be written into the same Excel file. The LTRead program creates a new worksheet for each of the responses that are present on Sheet2 of the experimental design file and the names of these sheets are the names of those responses. For example the response named Capacity will be written into new worksheet named Capacity. The writing of the data into Excel file is time consuming and therefore the user is shown a progress bar for the writing status.

There are two predefined figures for each modeled response – the residual case order plot and modeled vs. measured plot as shown in Figure 5.10. The residual case order plot and modelled vs. measured plot give the user graphical information of the goodness of the model.

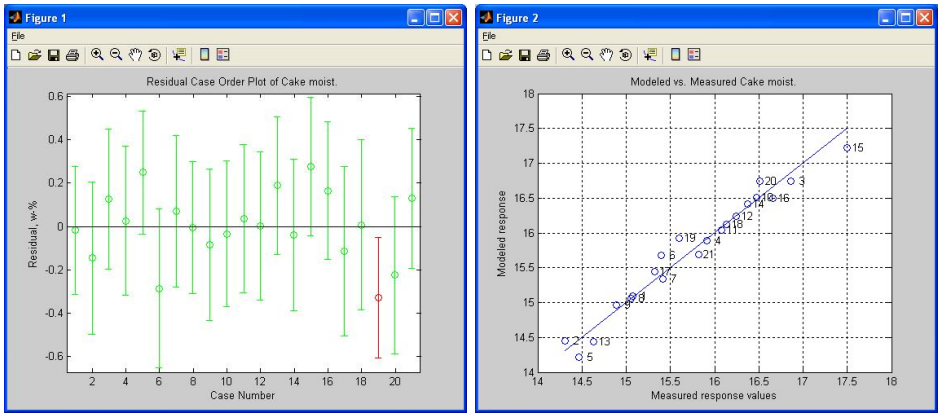

Figure 5.10.: The predefined figures of residual case order plot and modelled vs. measured plot of response named Cake moist.

After successful writing the user may start creating additional figures for

reporting purposes. This is done with the graphical user interface that is shown in Figure 5.11. The user interface has been divided into four different tables or tabs: '2D', '3D', 'Responses' and 'Reporting'. This user interface is used to preview the figures one can create. The user selects the variable and response and, when satisfied, clicks the 'Draw Now' button and gets a new figure. Later on the user may save these figures into separate (.jpg) image files.

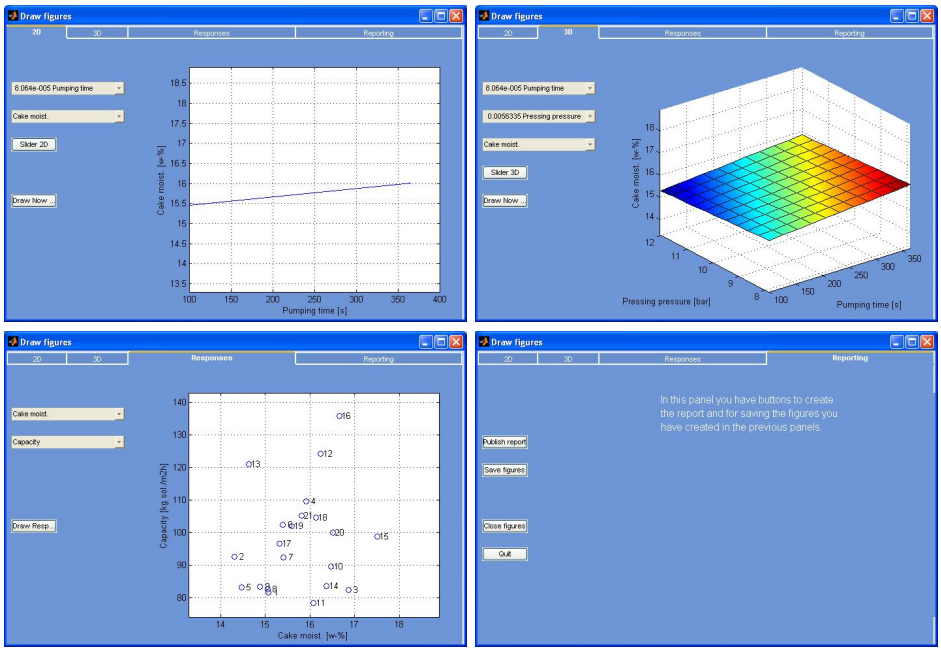

Figure 5.11.: User interface of LTRead showing tabs. From top left 2D, 3D, Responses, and Reporting tabs

Each of the tabs has their unique task. The 2D tab is for creating ordinary single variable against response figures. The 3D tab is for creating three dimensional figures where there are two axes for two variables and one axis for a response. The Responses tab is for creating response vs. response figures (this is of course possible only if there are two or more responses measured). The final Reporting tab is for saving the images and for creating the auto generated report file.

5.2.2. 2D and 3D tabs

There are two popup menus available on the 2D and 3D tab where the user may select the variables and the response he/she wants to use on the additional figure. The preview window shows the axes of choice before the user makes the decision whether he wants to use this figure. The user selects the variable and response and, when satisfied, clicks the 'Draw Now' button and gets a new figure window.

The additional figure created also contains the values of those variables that are present in the model but have a fixed value in the figure. If there is some specific value combination the user wants to use when drawing the additional figures, it is possible to select those values. The selection of the remaining variable values is done by clicking the 'Slider 2D(3D)' push-button. The 'Slider 2D(3D)' push-button creates the selection dialog for those variables that are not selected to the axis of the figure. The user can set the values using either the slider tool or by entering the desired value directly into the edit box. The impact of changing the values for those variables left out from the figure axis can be seen on the preview window as the line or plane is raised or lowered according to the changes in variable values. By default the software creates the figures using the mean value of the variable range used in the experiments. Now that the user has selected the new values to be used for the figure he/she can redraw new figure by pressing the 'Draw Now' push-button.

5.2.3. Responses and Reporting

The responses tab is available only if there are two or more measured responses. With this tab the user may create figures using measured responses on the axes of the figure. Plotting the measured response values on the same graph is an efficient way to visualise the ranges that are possible to achieve with the variable ranges used in the experimental runs.

The reporting tab is used to save the additional figures and to create an auto generated report containing the additional figures the user has created with the 2D, 3D and responses tabs.

The 'save figures' pushbutton opens a file save dialog where the user selects the filename to be used with figure files. The software will now save the figures as separate jpg files with running numbering for each figure that is

open at the time. For example, if there are seven additional figures open and if the user selects the file name to be, say, "customerpf" then the files will be named as customerpf1.jpg, customerpf2.jpg,... and customerpf7.jpg. The user should notice that the images that are saved do not contain the frames. Examples of saved images are shown in Figure 5.12.

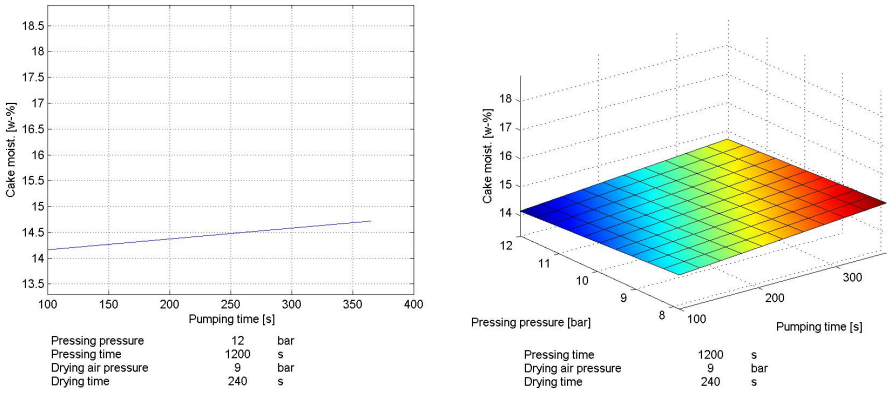

Figure 5.12.: Example figures of saved images.

By pressing the 'Publish Report' pushbutton, a Microsoft Word document is created with the same name as the Excel file that contains the numerical data. For example, if the original file containing the experimental design and the measured values is called 'CustomerPF.xls' then the resulting auto generated report containing the figures will be 'CustomerPF0.doc'. The trailing number is added automatically if the user later decides to create a new report with different set of figures. The auto generated report contain the same statistical information that has been written into the Excel file.

5.2.4. Regression modeling and statistical parameters

It is important to remember that the calculated models are experimental by nature. The models are solely based on the experiments that the test engineer has conducted. There are no filtration theories included and this is why these models are only applicable to the case that has been studied during the experimental work. And, furthermore, the models are valid only

within the variable space that has been used in the experiments.

The first thing that the test engineer should do is to examine the residual plot (on the left of Figure 5.10). This shows all of the experiments (cases) and the measured response values along with their error bars. The zero level on the residual plot represents the modeled level of the response. If the experiment fits completely the model then the dot is exactly on the zero-line. Each experimental value has calculated error bars showing 95% confidence intervals on the residuals. You can gain insight into the "goodness" of a fit by visually examining a plot of the residuals: residuals that are spread randomly and evenly on both sides of the zero line suggests that the model fits the data well, and residuals displaying a pattern indicate a poor fit. If an error bar does not contain zero, then the residual is larger than would be expected, at the 5% significance level. This is evidence that the observation may be an outlier.

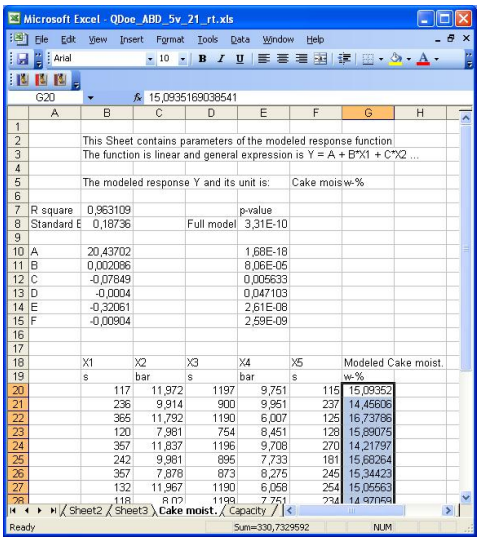

Figure 5.13.: An example sheet of the modeled response cake moisture added to Excel file.

LTRead creates a new sheet for each modeled response. This sheet has the same name as the response. An example of the model and the statistical data is shown in Figure 5.13. The statistical and model data on the sheet

are:

- The R-squared value indicating what percentage of the variation can be explained with the current model.
- Standard Error. In regression the standard error of the estimate is the standard deviation of the observed y-values about the predicted y-values.
- P-value. For a model, this should be smaller than significance level 0.05 so that the null hypothesis could be rejected (Montgomery, 1997).
- Coefficients B, C... are the coefficients that tell what effect each variable has on the response. If the coefficient has a positive sign then it increases the value of the current response and vice versa. It should be remembered, when evaluating the variable effects on the response, that the coefficient value and the unit should be considered together.
- P-values for coefficients B, C... indicates the significance of the corresponding variable.

A simple definition of P-value is: the probability (ranging from zero to one) that the results observed in a study could have occurred by chance. Sometimes it could be said that a coefficient having a P-value of 0.05 or smaller is statistically significant. But one must remember that a coefficient having a P-value greater than 0.05 does not necessarily mean that it is insignificant in practice (Box et al., 2005). This is because experimetal error can have an effect on the p-value and numerical values should be regarded as a guide line when making decisions about the statistical significance of the model.

When drawing additional figures one should notice that the numerical values in front of variables are the p-values of the corresponding variable coefficient in the model. P-values indicate the significance of that variable to the model. This software calculates the model with a significance level of 95% ,therefore the p-values should be smaller than 0.05 to be considered a significant variable to the model.

6. Results from the verification tests

Along with the software development, a number of different filter types were experimentally tested using various types of test materials and different combinations of operational variables. The tests were done according the full factorial design scheme, supplemented with additional data points. These tests were then used to verify whether the assumption of the linear model is valid and to which extent it is valid. All of the results showed that the linearity assumption gives an accuracy level that is acceptable for cake moisture and capacity. It should be kept in mind that the test filtrations do not aim for absolute accuracy, but recognition of important variables to a practical level of accuracy. Furthermore, the variable levels used in test filtrations are planned so that the variable range covers the practical working range of the filter. This means that there should not be any need to broaden the variable range any more than has been tested. The case examples presented here are for demostrating an accuracy level that is typical and acceptable in practical applications. The reason why linear models are used is that the interpretation of these models is straightforward and they are easy to understand. Not all possible filter types and material combinations were tested, but the combinations were limited to those pairs that are typical in filtration test cases. The number of experiments for verification tests was exhaustive, for example, the case of a Pressure filter with Quartz tailings having five variables meant that there were 49 experiments to be carried out, and as a result, it was concluded that one can obtain fairly accurate models and predictions with only 8 experiments. In total, more than 500 filtration experiments were conducted in the course of this study.

The filter types tested were (i) pressure filter, (ii) capillary action disc filter, (iii) double sided pressure filter, (iv) membrane filter press and (v) vacuum belt filter.

The materials used in these filtration tests were quartz tailings, copper

concentrate, native wheat starch, bauxite residue from Bayer process (Red Mud), titanium dioxide, ground calcium carbonate, precipitated calcium carbonate, magnetite, cobalt sulphide and zinc concentrate. Due to the extensive number of filtration experiments, only a fraction of the results from the verification tests have been published in conference proceedings. The following three chapters present typical filtration case studies completed with LabTop software. These case studies are to illustrate the test filtration cases and to show the validity of the selected approach.

6.1. Case I: Dewatering of quartz tailings by vertical automatic filter press

This case can be found in the conference paper 3. Häkkinen, A.*, Experimental study on dewatering of quartz tailing in vertical automatic filter presses, 11th CST Workshop 2008, Separation and Waste Water Treatment Techniques in Chemical and Mining Industries, Lappeenranta, Finland, June 12 - 13, 2008

This study was carried out to optimize the performance of a vertical automatic filter press for the tested mineral suspension. The target of these tests was to define the influence of the duration and pressure applied during the feeding, compression and air drying stages on the overall filter capacity and final cake moisture content. The tests were performed using a vertical pilot-scale filter press according to the test plan that was created by following the basic principles of factorial design. The results obtained were used for creating regression models for both of the studied responses. In order to determine the minimum number of tests required for the studied application, several different fractional factorial test designs were also created. By examining the statistical parameters of the different models, it was possible to compare the different test designs with each other. In this way, the minimum number of tests required for obtaining satisfactory results could be determined.

The examined filtration process consisted of 5 process variables and 1 constant. It was therefore decided that the base design for the tests should be a 2^5 – full factorial design. This means that each variable was given two levels (high and low) and every possible combination of these variable levels was included in the design. For five variables, this form of design resulted

in 32 tests. In addition to these tests, it was decided that a certain amount of center points were also needed in the design, in order to detect possible nonlinearities. The general structure of this kind of 2^5 – full factorial design with 9 center points –is illustrated in Figure 6.1 and the variable notations, together with the selected variable levels are summarized in Table 6.1.

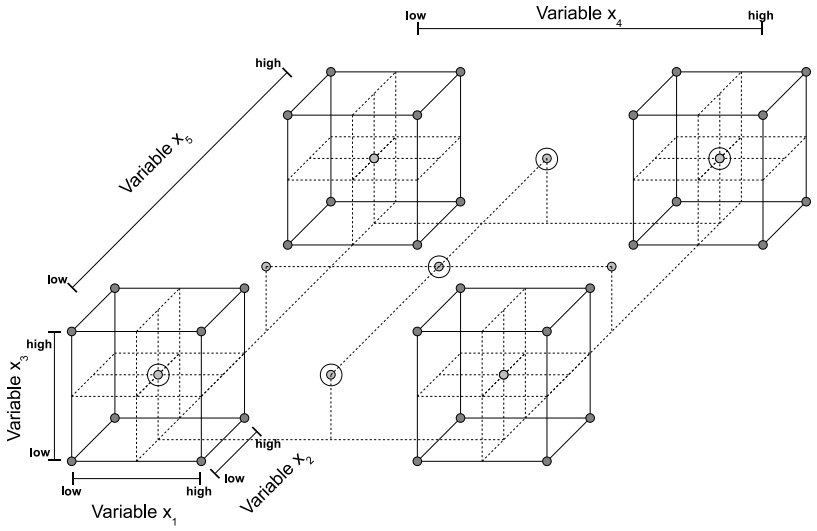

Figure 6.1.: 2^5 full factorial design showing nine center points. The five centerpoints that are circled are those used with Model 3.

In addition to the experimental design explained above, some tests were also made without the air drying stage. The purpose of these tests was to obtain reference values for estimating the overall influence of the air drying stage for the tested material. These 8 tests were carried out according to the 2^3 – full factorial design. The overall amount of tests was therefore 49. The experimental design used is shown in Table 6.2.

The measured data of the responses is shown in Figure 6.2. The experimentally determined cake moisture contents for the 49 tests ranged from 14.3 to 19.3 w-%. The average effect of the air drying stage on the cake

moisture contents was found to be approximately 3 percentage points (i.e. say from 19 % after pressing to 16 % after air-drying). The values for the experimentally determined capacities varied between 77.5 and 145.0 kg_{solids} / m^2h. The experiments were done in random order and the results show no time dependent trends.

Table 6.1.: Variables used with quartz tailings filtration case.

	Variables	low	high
x_1	Pumping time	120 s	240 s
x_2	Pressing pressure	8.0 bar	12.0 bar
x_3	Pressing time	600 s	1200 s
x_4	Drying air pressure	6.0 bar	10.0 bar
x_5	Drying time	120 s	240 s

Table 6.2.: Factorial experimental design used with quartz tailings filtration case.

	x_1	x_2	x_3	x_4	x_5		x_1	x_2	x_3	x_4
run	s	bar	s	bar	s	run	s	bar	s	bar
1	360	12	1200	10	240	26	240	10	900	6
2	240	10	900	6	240	27	360	8	600	10
3	360	12	1200	10	120	28	120	12	1200	10
4	360	12	600	6	120	29	120	12	600	10
5	240	10	900	8	120	30	360	12	1200	6
6	360	12	600	10	240	31	360	8	1200	6
7	360	12	600	10	120	32	120	8	1200	10
8	240	10	900	8	180	33	120	8	600	10
9	120	8	1200	10	240	34	120	12	1200	6
10	240	10	900	10	120	35	360	12	1200	6
11	120	12	600	10	240	36	360	8	1200	10
12	120	12	600	6	120	37	240	10	900	6
13	120	8	1200	6	120	38	120	8	600	6
14	120	12	1200	6	240	39	360	8	1200	6
15	360	8	600	6	240	40	360	12	600	6
16	240	10	900	10	180	41	240	10	900	8
17	120	8	600	6	120	42	360	12	1200	0
18	360	8	1200	10	120	43	120	8	600	0
19	120	12	600	6	240	44	360	8	600	0
20	360	8	600	6	120	45	360	12	600	0
21	120	12	1200	10	120	46	360	8	1200	0
22	240	10	900	10	240	47	120	12	1200	0
23	360	8	600	10	240	48	120	12	600	0
24	120	8	600	10	240	49	120	8	1200	0
25	120	8	1200	6	240					

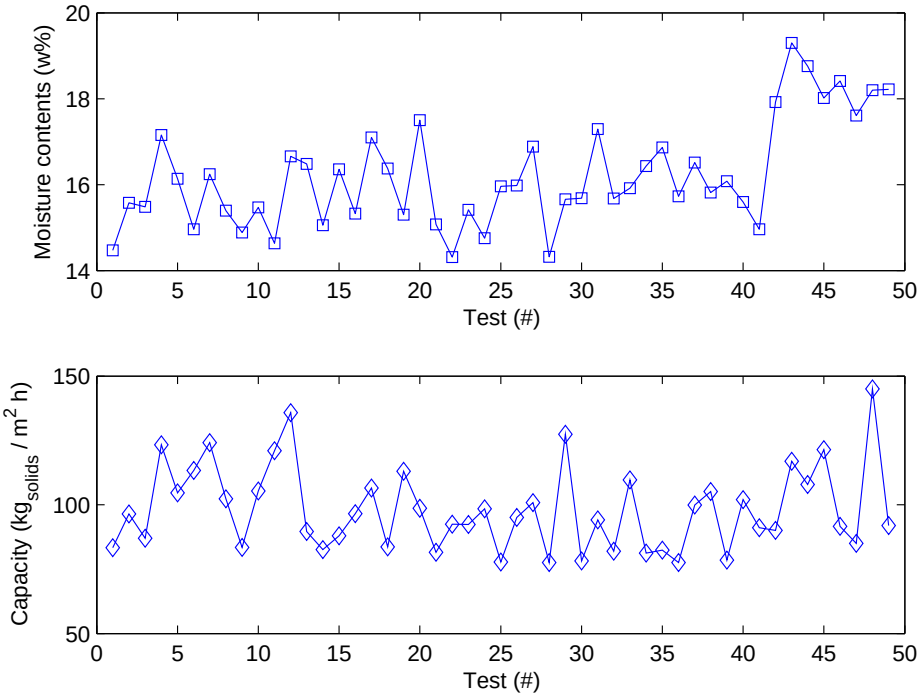

Figure 6.2.: Measured cake moisture contents and capacities of quartz tailings filtration case.

Figures 6.3 and 6.4 illustrate the predictions obtained with the linear models that were created using all of the data obtained from the 41 filtration tests. The eight filtration tests without air drying are not shown in these figures. These figures show the moisture contents and capacities that were predicted by applying the regression models against the values obtained experimentally. Therefore, if the predictions given by the models were perfect, all the data points would lie on the diagonal line. Figures 6.3 and 6.4 also show the coefficients of determination (R^2) which are reasonable for both response models. Furthermore, all the data points are distributed evenly on both sides of the diagonal which implies that the models are stable and significant outliers do not exist.

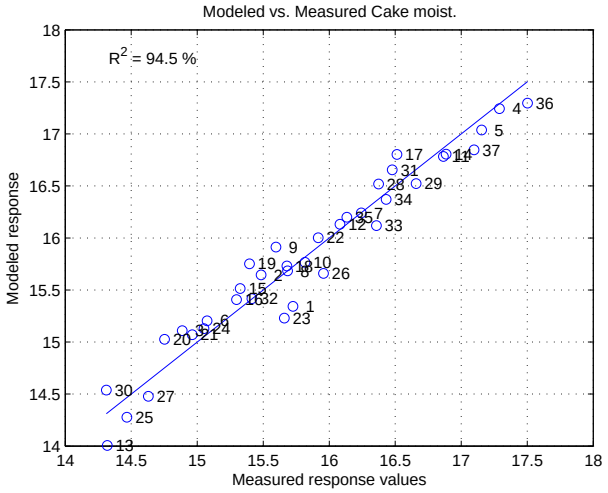

Figure 6.3.: Measured and modeled values of quartz tailings moisture for 2^5 design with 5 centerpoints.

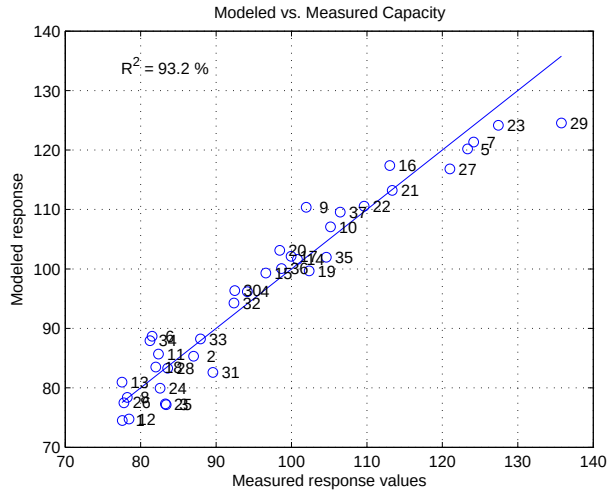

Figure 6.4.: Measured and modeled values of quartz tailings capacity for 2^5 design with 5 centerpoints.

Table 6.3 shows that the amount of experiments required for different designs varies significantly. All of the introduced designs are, however, based on the assumption of linear behavior and those should therefore result in similar models for a system where all relationships are entirely linear. Table 6.4 shows coefficient values for linear models and the standard error values of the cake moisture models. Table 6.5 summarises the p-values for each of the coefficients and the p-value for the full model when cake moisture contents was modeled. Table 6.6 shows the coefficient values for linear models and the standard error values of the capacity models. Table 6.7 summarises the p-values for each of the coefficients and the p-value for the full model when capacity was modeled. Table 6.8 summarizes the coefficients of determination, root mean squared errors, cross validated coefficients of determination and root mean squared errors of prediction for the different models.

Table 6.3.: Different kinds of experimental designs considered in quartz tailings Case I

Model 1	All tests included, also the ones without air drying (49 runs)
Model 2	Factorial design with 9 center points (41 runs)
Model 3	Factorial design with 5 center points (37 runs)
Model 4	Factorial design without the center points (32 runs)
Model 5	Fractional design (1/2) with 9 center points (25 runs)
Model 6	Fractional design (1/2) without the center points (16 runs)
Model 7	Fractional design (1/4) with 9 center points (17 runs)
Model 8	Fractional design (1/4) without the center points (8 runs)

The experimental design with the smallest number of experiments (8) appears to give the highest coefficient of determination both for the cake moistures as well as for the capacities. This implies that the amount of tests can be considerably reduced using design of experiments, but it also means that the possibility of nonlinear behavior should be checked. In this study, the validity of these models was verified using an external test set. Moisture model 8 (Table 6.3) and Capacity model 8 were used to predict the values for the tests that were left outside these models. Only the experiments with the air drying stage were considered which means that the test set consisted of $41 - 8 = 33$ tests. The coefficients of determination for the test sets Q^2 was 89.3 % and 84.0 % for moisture model 8 and capacity model 8 respectively.

Table 6.4.: Linear moisture content model coefficients and standard error values in quartz tailings case.

	Coefficient						
Model	β_0	β_1	β_2	β_3	β_4	β_5	Std Err
1	19.92	1.586E-3	-0.133	-0.546E-3	-0.184	-7.369E-3	0.342
2	20.50	1.919E-3	-0.091	-0.293E-3	-0.312	-9.167E-3	0.223
3	20.56	1.914E-3	-0.091	-0.312E-3	-0.314	-9.116E-3	0.217
4	20.58	1.937E-3	-0.093	-0.350E-3	-0.304	-9.120E-3	0.206
5	20.35	2.090E-3	-0.077	-0.378E-3	-0.317	-9.095E-3	0.189
6	20.48	2.111E-3	-0.081	-0.454E-3	-0.307	-9.172E-3	0.178
7	19.75	2.617E-3	-0.058	3.321E-05	-0.324	-9.461E-3	0.176
8	20.05	2.648E-3	-0.062	-8.808E-5	-0.316	-9.965E-3	0.149

Table 6.5.: Linear moisture content model coefficients p-values and model p-value in quartz tailings case.

	p-values						
Model	β_0	β_1	β_2	β_3	β_4	β_5	Mod. pval
1	1.165E-39	0.001	2.202E-05	0.017	9.501E-10	1.728E-11	0
2	1.361E-37	1.379E-06	0.0001	0.074	1.656E-14	3.939E-17	0
3	1.498E-34	1.429E-06	8.426E-05	0.053	1.464E-13	6.415E-16	0
4	1.753E-30	1.094E-06	5.048E-05	0.027	6.669E-12	9.612E-15	3.33E-16
5	3.750E-22	3.932E-05	0.0052	0.058	7.661E-10	4.473E-11	4.19E-12
6	1.203E-13	0.0002	0.0056	0.029	3.197E-06	1.727E-07	2.10E-07
7	2.773E-13	0.0004	0.107	0.895	7.156E-07	1.884E-07	8.39E-08
8	0.0005	0.027	0.158	0.722	0.016	0.008	0.016

Table 6.6.: Linear filter capacity model coefficients and standard error values in quartz tailings case.

	Coefficient						
Model	β_0	β_1	β_2	β_3	β_4	β_5	Std Err
1	160.5	-9.834E-03	0.869	-0.063	0.148	-0.064	4.737
2	155.6	-7.944E-03	0.971	-0.060	0.217	-0.062	4.397
3	155.6	-8.019E-03	0.974	-0.060	0.171	-0.060	4.498
4	154.9	-7.870E-03	0.946	-0.061	0.302	-0.059	4.739
5	160.3	-0.021	0.974	-0.059	-0.189	-0.060	5.019
6	160.5	-0.021	0.929	-0.059	-0.192	-0.052	6.079
7	140.7	0.021	1.121	-0.058	0.641	-0.061	3.242
8	135.4	0.021	1.052	-0.058	1.200	-0.048	2.391

Table 6.7.: Linear filter capacity model coefficient p-values and model p-value in quartz tailings case.

	p-values						
Model	β_0	β_1	β_2	β_3	β_4	β_5	Mod. pval
1	7.645E-30	0.122	0.030	4.899E-24	0.662	1.154E-06	0
2	2.031E-23	0.232	0.023	3.606E-20	0.662	6.527E-06	0
3	2.779E-21	0.239	0.027	1.805E-18	0.747	3.285E-05	0
4	3.841E-18	0.274	0.042	4.702E-16	0.615	0.000191	1.188E-14
5	2.177E-12	0.056	0.151	3.326E-10	0.803	0.004	5.567E-09
6	2.302E-07	0.123	0.267	2.065E-06	0.869	0.060	3.166E-05
7	7.830E-09	0.051	0.092	5.777E-08	0.304	0.002	1.059E-06
8	0.003	0.101	0.144	0.003	0.206	0.077	0.014

Table 6.8.: Coefficients of determination for the different models quartz tailings Case I

	Model for cake moisture				Model for capacity			
	R^2	$RMSE$	Q^2	$RMSEP$	R^2	$RMSE$	Q^2	$RMSEP$
Model 1	92.95	0.321			92.51	4.44		
Model 2	93.64	0.206			92.74	4.06		
Model 3	94.48	0.198	41.43	0.278	93.19	4.12	53.46	3.56
Model 4	95.19	0.186	76.53	0.298	93.57	4.27	53.94	3.29
Model 5	95.48	0.165	88.47	0.292	90.28	4.38	93.07	4.34
Model 6	97.16	0.140	90.67	0.246	92.15	4.81	89.79	4.34
Model 7	96.74	0.142	85.58	0.312	94.81	2.61	85.85	6.46
Model 8	99.34	0.075	89.29	0.258	99.42	1.20	83.95	5.96

Figure 6.5 shows measured and modeled plot of cake moisture and capacity obtained with Model 3. The test set consisted of four augmentation points that were not used in modeling. The low value of the coefficient of determination of the test set is due to the small variance in test set response which in turn increases the quotient term in Equation 4.6 and, as a result, the Q^2 value decreases.

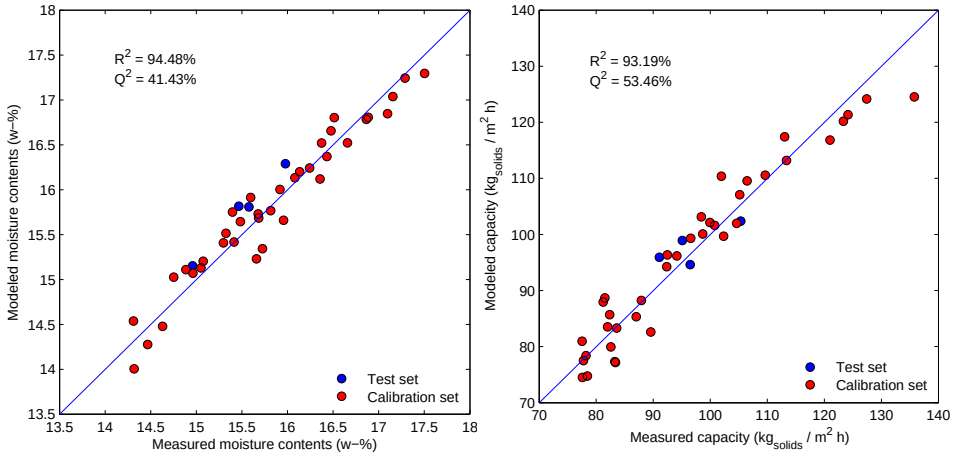

Figure 6.5.: Measured/modeled plot of cake moisture and capacity obtained with Model 3.

Figure 6.6 presents the measured and modeled plot of cake moisture and capacity obtained with Model 8 and the results achieved with the external test set.

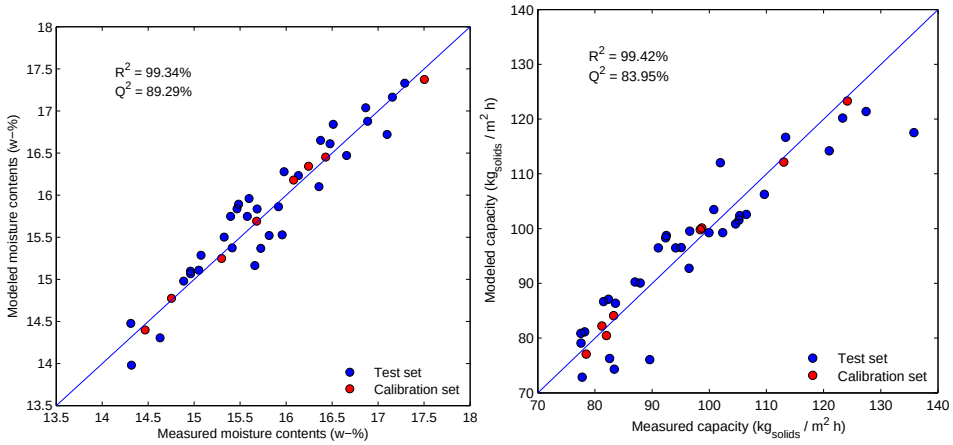

Figure 6.6.: Measured modeled plot of cake moisture and capacity obtained with Model 8.

The results presented for this case clearly and reliably showed that by using statistical design of experiments and regression models, the amount of filtration tests required for determining the main effects of five filtration process variables could be reduced from 49 to 8 and fairly accurate models and predictions could still be obtained.

6.2. Case II: Red mud filtration with horizontal membrane filter press

A more detailed description of this case can be found in the conference paper 8. Sparks, T.*, Huhtanen, M., Kinnarinen, T., Salmimies, R., Häkkinen, A., The challenge of red-mud filtration, 13th Nordic Filtration Symposium, Lappeenranta, Finland, June 10 – 11, 2010. Proceedings pp. 52-57.

Red mud is the digestion residue that is produced from bauxite as a side stream in the Bayer Alumina Process. Given the composition of bauxite, for every tonne of alumina produced there is approximately another tonne

of red-mud. Since the total production of alumina is around 80 million tonnes per year, there are, globally, approximately 5,000 tonnes of red-mud produced every hour. Although the filtration of red mud is just one of the many filtration steps that are used in the alumina process, it is important in environmental terms. In this case, a membrane filter press was used to dewater and wash a sample of red-mud. The target of this study was to find out how the operating conditions of a membrane filter press influence the dewatering. Five variables were selected, together with two responses. The variable notations together with the selected variable levels are summarized in Table 6.9. The experimental design was fractional factorial design, completed with five centre points. The dewatering and overall capacity responses were modeled using linear regression.

Table 6.9.: Variables and variable levels for the RedMud case

	Variables	low	high
x_1	Pumping time	60 s	180 s
x_2	Pressing(I) pressure	6.0 bar	16.0 bar
x_3	Pressing(I) time	60 s	180 s
x_4	Washing time	60 s	180 s
x_5	Drying time	60 s	180 s

The tests that were made were designed according to the principles of statistical Design of Experiments. The filtration process that was examined was described by 5 process variables and 5 constants. Due to the fairly large number of variables, the full factorial design was considered to be too laborious especially when the experimental design was completed with a set of centre points that are used to detect the possible nonlinearities. It was therefore decided that a fractional factorial design should be carried out. This means that only 21 out of the original 41 tests had to be performed. The fractional design was a resolution V design with generator $x_5 = x_1x_2x_3x_4$. In addition to this fractional factorial design, 4 tests were also made for determining the washing efficiency when larger wash ratios were applied. The experimental design used is shown on Figure 6.7. The measured response values are shown on Figure 6.8. The experimentally determined cake moisture contents for the 25 tests ranged from 23.0 to 25.7 w-%. The values for the experimentally determined capacities for the first 21 tests varied between

71.5 and 123.2 kg_{solids} / m^2h.

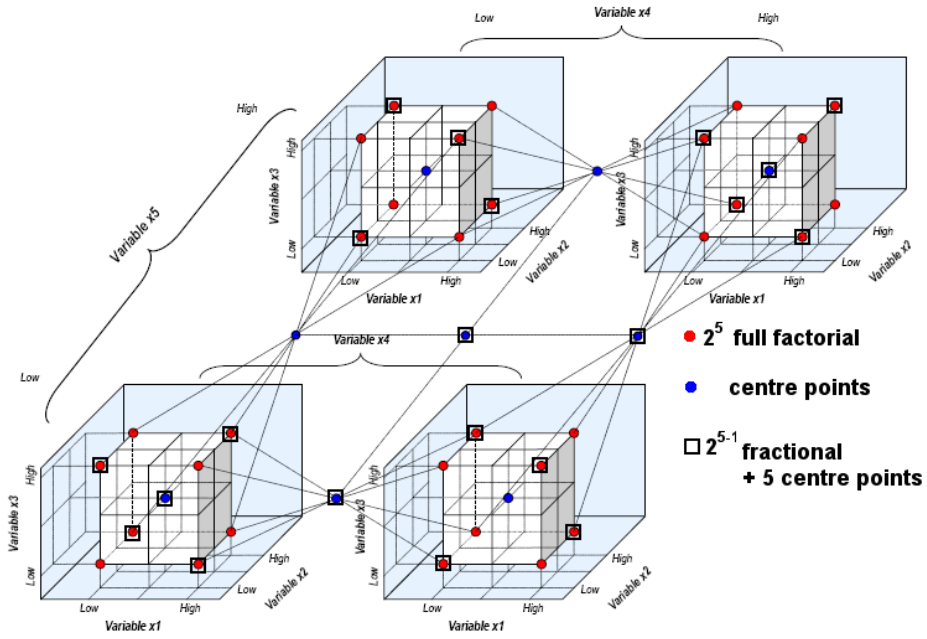

Figure 6.7.: Experimental design used in Red Mud filtration case.

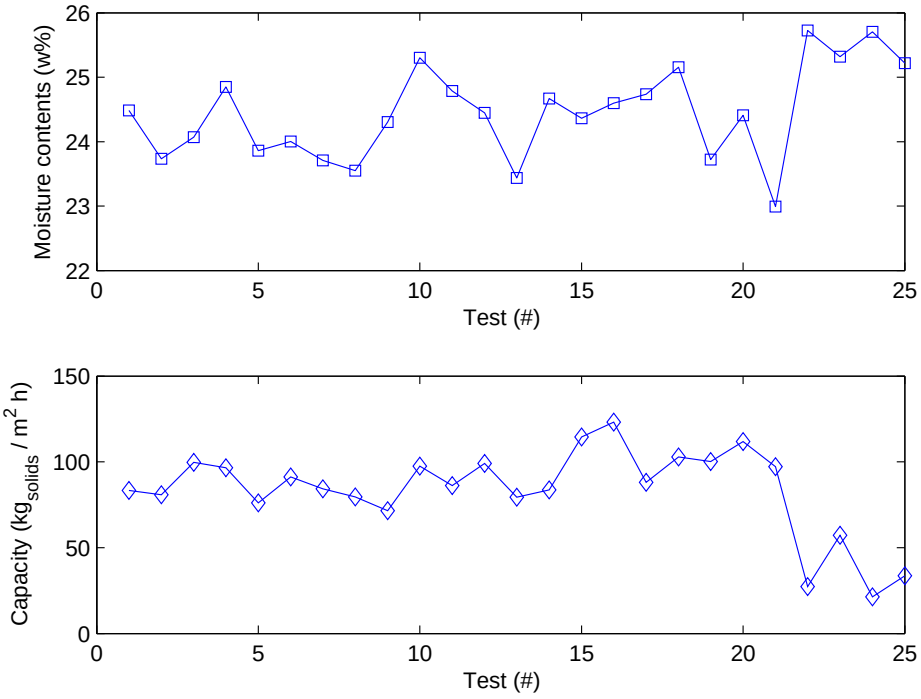

Figure 6.8.: The measured response values in RedMud filtration case.

The coefficient values and p-values are summarized in Table 6.10. The predictions that were obtained for the cake moisture contents (Figure 6.9) are relatively good, although the value of the coefficient of determination is only 73.7 %. It can, however, be seen from Figure 6.9 that the RMSE value is 0.36. The poor coefficient of determination is therefore probably caused by the fact that variation in the experimentally determined cake moisture contents was very small and therefore any experimental inaccuracy influences strongly the calculation of the coefficient of determination. Red mud is generally considered to be material that is difficult to filter and this is why it is often disposed of as a slurry and not dried into cake form and stacked.

Figure 6.9 shows the predictions obtained by the capacity model. The differences in the experimental values for the capacities were very large, especially in the case of the four tests performed with significantly higher

Table 6.10.: The coefficient values and corresponding p-values for the Red mud case.

	Moisture contents		Capacity	
	Coefficient	P-value	Coefficient	P-value
β_0	25.03	6.131E-23	123.8	4.506E-11
β_1	0.121E-03	0.945	0.0162	0.673
β_2	-0.035	0.107	0.115	0.802
β_3	0.003	0.096	-0.122	0.004
β_4	0.677E-03	3.576E-05	-0.033	3.134E-10
β_5	-0.007	0.0003	-0.141	0.644E-03
	std. err	Model p-val.	std. err	Model p-val.
	0.415	5.411E-05	9.061	7.381E-09

wash ratios. Since these four tests deviated so strongly from the other tests, they were not considered in the models. It can be seen that the pure linear model for the 25 tests of the initial fractional factorial design explains the variations in the capacity values very well. The coefficient of determination is 90.0 %, RMSE is 7.90 and, as can be seen from Figure 6.9, the deviation from the diagonal line is small for all data points.

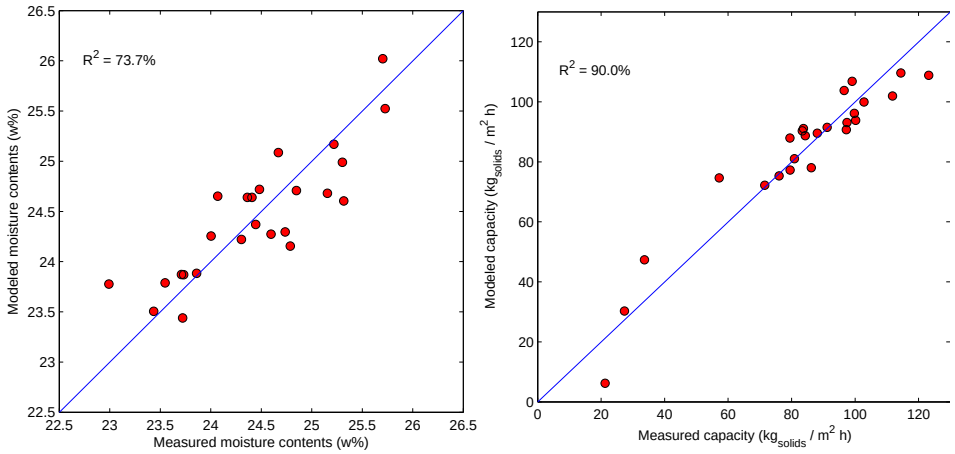

Figure 6.9.: The measured and modelled cake moisture content and capacity of Case II RedMud.

It can be seen that with rather simple models, responses like cake moisture content and filter overall capacity can be evaluated. The basic linear models perform well.

6.3. Case III: Dewatering of Cu-concentrate with ceramic capillary action disc filter

A more detailed description of this case can be found in the conference presentation 5. Häkkinen, A.*, Huhtanen, M., Ekberg, B., Kallas, J., Experimental study on dewatering of copper concentrate by a ceramic disc filter, 11th Nordic Filtration Symposium, Copenhagen, Denmark, August 25 - 26, 2008.

Ceramic vacuum disc filters are used widely for dewatering copper ore slurries in the mining industry. The scale of copper production processes is typically very large, which means that small changes in the operation of the filter can have a significant impact on the overall effectiveness of the process. Some of the variables that determine the performance of ceramic disc filters are the rotation speed of the disc, the level of slurry in the feed basin, the vacuum level applied during the filter cake formation and dewatering stages

as well as the solid concentration and the temperature of the feed slurry. This experimental study was carried out to determine the influence of several process variables on the performance of the ceramic vacuum disc filter. The variable notations together with the selected variable levels are summarized in Table 6.11. Experiments were performed using laboratory scale leaf test equipment and the obtained results where then scaled to describe the operation of an industrial scale filter. Overall capacity of the filter and residual moisture content of the copper ore cakes were used to characterise the performance of the filter. The results were utilized for the creation of regression models which were successfully used for discovering the most significant process variables and for estimating the effects of those variables on the operation of the filter.

Table 6.11.: Variables and variable levels considered in the copper concentrate experiments.

	Variables	low	high
x_1	Slurry level	200 mm	600 mm
x_2	Cycle time	30 s	60 s
x_3	Vacuum level in drying	0.50 bar	0.95 bar
x_4	Temperature of the slurry	10 °C	50 °C
x_5	Solids concentration	50 w-%	70 w-%

The filtration process that was examined in this study consisted of 5 process variables and 1 constant. It was decided that slurry temperature and solids concentration should be given 3 different levels whereas only 2 levels were given to all other variables. This design was created according to the basic principles of full factorial designs, which means that all possible combinations of the variable levels were included in the design. This kind of design resulted in 72 tests. In addition to these tests, it was decided that a certain amount of center points were also needed in the design in order to detect the possible nonlinearities during modeling. The overall number of tests in the final design was 81. The graphical interpretation of the design is shown in Figure 6.10.

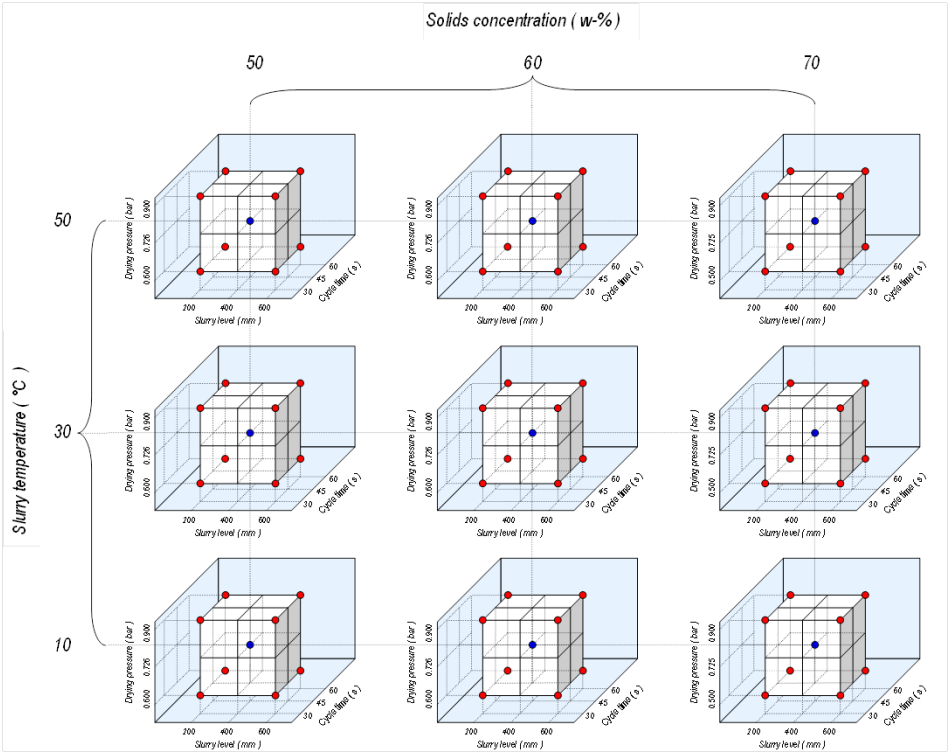

Figure 6.10.: Experimental design used in copper concentrate experiments

The measured response values are shown in Figure 6.11. The experimentally determined cake moisture contents for the 81 tests ranged from 10.2 to 17.8 w-%. The values for the experimentally determined capacities for the 81 tests varied between 166.9 and 2153.7 kg_{solids} / m^2h. The growing trend in the measured response values shown on Figure 6.11 is due to the order in which the experiments were done. Complete randomisation was not used because variable 5 (slurry solids concentration) and variable 4 (slurry temperature) were blocked so that the first 27 experiments were done with the low concentration, the next 27 experiments with the medium concentration and the last 27 with high concentration. Similarly, within these 27 experiments, the slurry temperature was divided into 9 experiment blocks with low, medium, and high temperature test sets. The remaining variables were done in random order within the blocks.

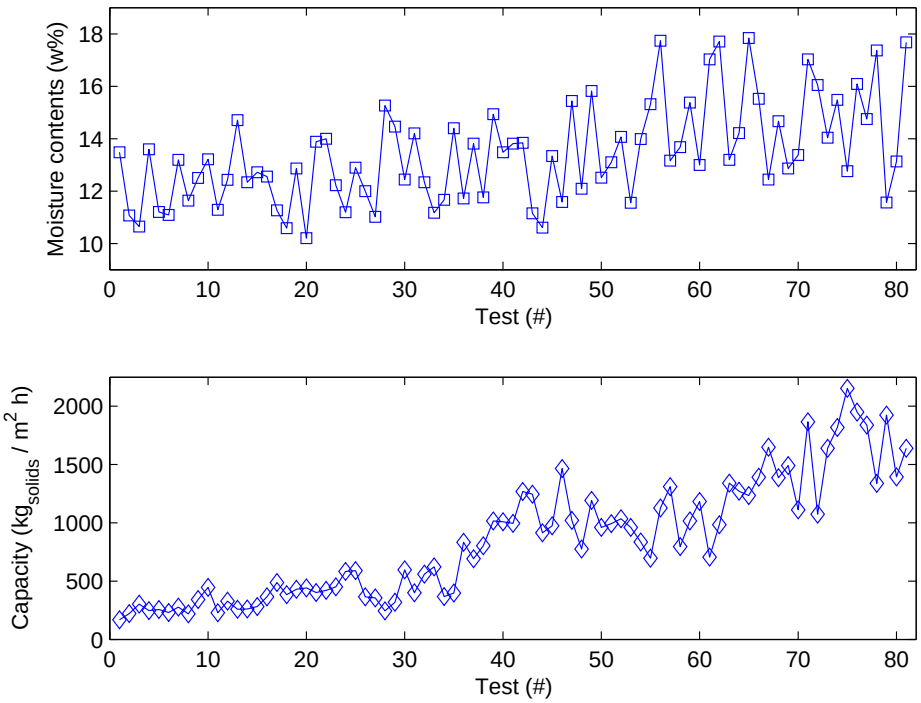

Figure 6.11.: Measured cake moisture contents and capacities for the copper concentrate case.

One target of this study was to find out if the amount of experiments could be reduced by using the statistical design of experiments. For this purpose, the full data sets were divided into smaller (fractional factorial) designs and new regression models were made for those designs. The smallest design consisted of only 18 tests and the results for this design are presented in Figure 6.12. This Figure show the typical measured vs. predicted diagrams and also those predictions that were obtained using the so-called external tests set. Test sets are used here for reliably defining the actual prediction ability of the models. This means that completely independent test results are predicted by the obtained models and compared with the actual results. Since the full data consisted of 81 experiments, the test set contained 81 – 18 = 63 independent tests.

At first the models were created with the full test set. The coefficients of

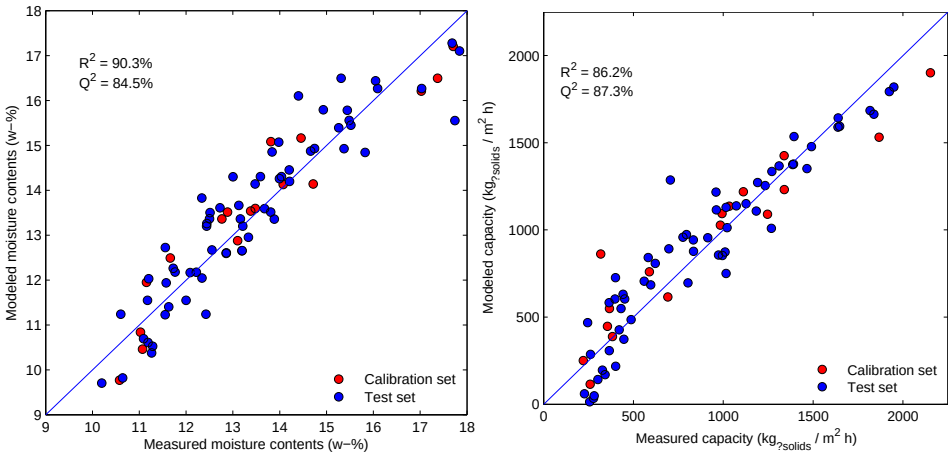

Figure 6.12.: Measured vs. modelled – diagrams of the cake moisture contents and filter capacity for the external test set. Predictions have been calculated using the model created for the fractional factorial design (18 tests).

determination for cake moisture and capacity models were 86.9 % and 87.8 % respectively. The statistical information for the model created with all of the experimental data and for the fractional design is shown on Table 6.12. Table 6.13 and Table 6.14 summarize the coefficients and corresponding p-values for the models calculated with the different experimental designs.

Main effects of the studied variables were calculated for the moisture content of the filter cakes and for the capacity of the filter. These are shown on Table 6.15. The main effect is the change in the response produced by the change in the level of the variable.

Table 6.12.: The Coefficients of determination, RMSE, coefficient of determination for the test set and RMSEP values for the test set.

Runs		Model for cake moisture				Model for capacity			
cal. s.	test s.	R^2	$RMSE$	Q^2	$RMSEP$	R^2	$RMSE$	Q^2	$RMSEP$
81	0	86.94	0.684			87.76	179.5		
18	63	90.26	0.652	84.46	0.722	86.18	210.9	87.29	177.0

Table 6.13.: Coefficients and P-values and standard error for cake moisture content models with copper concentrate case.

	Full factorial 81 runs		Fractional 18 runs	
	Coefficient	P-value	Coefficient	P-value
β_0	8.456	6.752E-17	7.757	0.001.216E-03
β_1	-0.004	0.406	-0.008	0.521
β_2	0.150	3.448E-22	0.164	4.379E-05
β_3	-6.221	5.394E-27	-6.451	6.588E-06
β_4	-01.618E-03	0.0002	-1.777E-03	0.104
β_5	0.025	2.470E-05	0.031	0.038
	std. err	Model p-val.	std. err	Model p-val.
	0.711	0	0.798	1.096E-05

Table 6.14.: Coefficients and P-values and standard error for capacity models with copper concentrate case.

	Full factorial 81 runs		Fractional 18 runs	
	Coefficient	P-value	Coefficient	P-value
β_0	-2562	6.396E-20	-2902	0.389E-03
β_1	10.316	7.547E-12	10.392	0.017
β_2	57.649	6.589E-32	63.257	8.149E-06
β_3	138.3	0.161	180.2	0.526
β_4	-0.376	0.001	-0.339	0.321
β_5	-7.542	2.073E-06	-8.616	0.072
	std. err	Model p-val.	std. err	Model p-val.
	186.6	0	258.3	8.470E-05

Table 6.15.: Main effects of the studied variables for the moisture content of the filter cakes and the capacity of the filter for copper concentrate. Case III

Variable	Range	Main effect on filter cake moisture	Main effect on the filter capacity
Slurry level	200 → 600 mm	-0.65 w-%	-150 kg/m^{2}h
Cycle time	30 → 60 s	+0.75 w-%	-226 kg/m^{2}h
Vacuum level in drying	0.5 → 0.95 bar	-2.80 w-%	+62 kg/m^{2}h
Temperature of the slurry	10 → 50 °C	-0.16 w-%	+412 kg/m^{2}h
Solids concentration	50 → 70 w-%	+3.00 w-%	+1153 kg/m^{2}h

Figure 6.12 shows good agreement between the models created with the fractional factorial design and the external test set that is used to validate the model. When comparing the coefficient of determination of the model (R^2) and the coefficient of determination of the external test set (Q^2) one can see that the correlation is basically on the same level both for the model and the validation test set.

The results show that linear models are valid in most cases. It is important to remember that the calculated models are experimental and are solely based on the experiments the test engineer has concluded. There are no filtration theories included and this is why one should never use the given models on anything other than the case that has been studied during the experimental work. And furthermore the models are valid only within the variable range that has been used for the experiments.

7. Summary and conclusions

Test filtration, with many variables involved, can be very labour intensive and time consuming if conventional methods such as "one variable at a time" are used. The use of experimental design can reduce the amount of experimental work. Changes in variables are done in a structured way and cross correlation between variables is avoided. The objective of this project was to create a user-friendly software package for reducing the amount of experimental work and costs in typical test filtration applications. There are a number of simplifications in the software that has been created during this work. These simplifications are in the modelling and in the statistical information that is provided to the user. These simplifications are done in order to keep the software as simple to use as possible, so that the user experience is at an acceptable level for users with no statistical background. Human nature in making experimental work is one of the obstacles.

When starting this project other experimental design methods were discussed but the decision favoured factorial designs as it was unknown how the test filtration tasks behave in different types of filter equipment. Robustness was a decisive factor in selecting the experimental design methods. The experimental design method selected is to be used routinely on different test filtration tasks by personnel who are not familiar with experimental design or statistics.

The linear models used in this work show good agreement with the experimental data. One criticism can be that these models are too simple, but on the other hand, even if the variables used in the experimental setup do have a nonlinear effect on the response it may be the case, that within the selected variable range, the effect can be modelled sufficiently well with a linear function. This may simply be because, when selecting the variable range it happened that there was no local minimum or maximum within the selected range. Furthermore, the variable ranges used in practical filtration tests are often narrower that the ranges used in filtration tests used to gather data for theories. The results with different test cases also showed that the data

gathered with fractional factorial designs is accurate for practical purposes and, thus, the number of experiments can be reduced without the fear of losing too much information.

The statistical design of experiments has proved to be a powerful tool for all kinds of experimental work. The software package produced during the course of this thesis combines the power of design of experiments and empirical modeling into one package. It should be remembered that the targeted users of this software are filtration practitioners and not researchers working in academia. The software not only guides the user to follow good testing practices but it also reveals the possible pitfalls faced in individual tests by showing residual case order plot and a modeled vs. measured plot. The visual information that is given to the users is one of the important features of the software. The practical value of this work comes from the fact that this software provides both numerical data in the form of model equations that are easy to use and visual representations showing how the selected variables affect the responses. Based on the data given it is easier to make well founded decisions and conclusions for the test filtration cases.

Filtration and empirical modelling has not been tested to this extent previously. In the aftermath, one criticism could be that the used experimental design method produces designs that are too large, but it must be remembered that when starting this work it was unknown whether the test filtrations could be modelled with linear models or not.

This work should be seen as a first step of introducing statistical methods into the field of test filtrations. The methods here may look crude in the eyes of chemometricians, but the author believes that these steps are needed in order to make filtration specialists (who have previously focussed on creating mechanistic models of filtration subprocesses) aware of statistical experimental designs and chemometrical methods.

When looking at the wide variety of different techniques and devices that are used for solid/liquid separation, it is understandable that there are no practical methods to combine filtration theories into a single model so that a complete filtration process could be simulated purely ab initio. Therefore, the author believes that the practical value of experimental design and the use of empirical modeling is significant.

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Article I

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Development of testing procedure for ceramic disc filters

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ABSTRACT

Small-scale laboratory tests for sizing and designing disc filters are typically performed by using leaf test equipment. Although the basic tests are fairly simple and quick, the accuracy and repeatability of the results may be poor due to various reasons. In addition to errors caused by variations in the structure of the equipment, also the skills and experience of the person performing the tests is of great importance. This paper introduces an experimental study carried out for defining the most important sources of errors in these kinds of laboratory tests and for estimating their influence on the final test results. The results obtained in this study were utilized for designing an improved version of the test equipment and also for creating a standard procedure for performing the tests. The results acquired with the small-scale laboratory tests were in good agreement with the performance results obtained from full-scale ceramic disc filters operating in industrial processes.

1. Introduction

Ceramic capillary action disc filters are continuously operating units that are mainly used in high-tonnage applications for dewatering relatively free-filtering suspensions, such as mineral and metal concentrate slurries. The performance of the filters is mostly based on the special properties of the ceramic filter medium and the capillary phenomenon, due to which the filters are energy saving and durable against severe conditions, thus providing an excellent alternative for conventional disc filters with low cake moistures and high capacities (Outotec, 2010). Preliminary testing for rotary vacuum disc filters has traditionally been carried out by performing small-scale leaf tests. In these tests, small test plates are used for collecting initial data about the filterability of the target suspensions and information regarding the suitability of disc filters for the tested application. Additional pilot-scale testing has typically been used for obtaining reliable and accurate final data for the sizing of the full-scale filters. Pilot-scale tests take a great deal of time due to the limited availability of test units, and they are usually very expensive to arrange. Reliable test data is therefore needed quickly and with a low cost for sizing and sales purposes.

However, the accuracy of the results obtained in typical leaf tests has been observed to be poor in many applications, and significant overestimations of the overall filter capacities have occurred due to various reasons (Osborne, 1981; Thompson, 1993; Wakeman and Tarleton, 2005). The most significant sources of inaccuracy are assumed to be the decrease of filter medium permeability in long-term operation in full-scale units, differences in the effective vacuum levels, non-comparable mixing conditions, and general human errors in the leaf testing procedure.

The aim of this study was to experimentally determine the significance of the different sources of errors regarding leaf testing for ceramic capillary action disc filters. General leaf testing procedures for conventional rotary vacuum disc filters and error sources suggested in the existing literature are firstly reviewed, and their applicability regarding ceramic disc filters is evaluated. Small-scale leaf tests with ceramic filter plates were performed in laboratory conditions, and improvements to the test procedure were made according to the test results. The results obtained by using the improved test procedure were finally compared to the performance of full-scale filters in three different industrial applications.

2. Rotary vacuum disc filters

Rotary disc filters are vacuum-driven continuous action units, where the slurry is introduced into a basin located around the lower part of discs with filter cakes forming on both faces of the discs that are rotating on a horizontal axis. The discs consist of individual segments connected to a common center shaft. The filter media used in the segments are traditionally conventional woven filter cloths or micro porous ceramic material. Disc filters are mainly used for dewatering fairly coarse and heavy mineral and metal concentrate slurries, making it important to ensure sufficient agitation in the filter basin to prevent the sedimentation and classification of the

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slurry. The most commonly applied agitation device in disc filters is an oscillating cradle-type agitator located in the bottom of the basin. The direction of the flow created by this kind of an agitator is mainly parallel with the filtration surfaces of the discs.

2.1. Operation of rotary vacuum disc filters

Disc filter operation includes at least three stages, which are cake formation, cake dewatering and cake discharge. Other possible stages are cake washing and backflow washing of the filter medium. Cake washing is applied in conventional disc filters only in rare special cases, as the washing efficiency is typically poor due to the vertical orientation of the cakes. On the other hand, cake washing has been proved to be efficient in ceramic disc filters due to the even structure of the cakes, the steady flow profile of the ceramic filter media and the gas free filtrate flow. An illustration of the main stages of the filtration cycle in disc filters is presented in Fig. 1 (Outotec, 2010).

Cake-formation (Fig. 1a) takes place on the disc segments as they move through the slurry basin, and the time available for this stage depends on the rotation speed of the disc and the height of the slurry level in the basin. As soon as the segment rises from the slurry, dewatering of the cake (Fig. 1b) begins. The available drying time is usually an operating constraint of the filter unit, and also this depends on the rotation speed and slurry level. Cake discharge (Fig. 1c) occurs after the dewatering stage due to blade or wire scrapers installed on both sides of each disc or to a fast pressurised air pulse (snap blow), as is the case in conventional disc filters. Just before the segments are immersed into the slurry again, there may also be a short back flow washing stage (not shown in Fig. 1) for refreshing the filter medium and thus ensuring good cake formation and dewatering results during the next cycle. Due to the rotating movement of the discs, the rotation speed and the height of the slurry level directly determine the cake formation and drying times, and they are therefore the most important operating parameters of disc filters. Other important operation variables are the applied (or achieved) pressure difference and the slurry properties, such as solid concentration, particle size distribution and temperature. The slurry properties can be partially dependent on the homogenization degree of the slurry in the filter basin, and therefore, the agitation intensity should also be considered as one of the operation parameters.

3. Typical leaf test procedure for disc filters

Small-scale filtration tests are typically carried out during the preliminary stages of selection and sizing of filtration equipment. The tests are quick and relatively cheap compared to pilot-scale test runs, which are usually avoided unless considered absolutely essential or cost-effective (Thompson, 1993). When considering the tests needed for designing and sizing continuous rotary vacuum disc filters, the main target is in most cases to predict the dry cake production capacity and the residual moisture content of the discharged cakes. These properties depend on a wide variety of different variables, such as the characteristics of the filter medium, the operating pressure difference, the rotation speed and submergence of the discs, as well as the specific properties of the slurry. It is often important also to consider the secondary characteristics of the separation process, such as cake cracking and the role of agitation, already during the initial testing phase. A great deal of work has been carried out for developing test methods for different kinds of filtration equipment, and guidelines for performing the tests have been presented, for example, by Osborne (1981), Purchas and Wakeman, (1986), Johnston (1995), Thompson (1993), Rushton et al. (2000) and Wakeman and Tarleton (2005). The main principle of the test procedure applied to conventional rotary vacuum disc filters is that a small filter element, a leaf, is used for simulating a section of the full-scale filter disc. This means that the first requirement for successful tests is that the properties and structure of the leaf are exactly identical to the full-scale discs. It has also been reported (Johnston, 1995; Rushton et al., 2000) that the orientation of the filter leaf during the different stages of the tests should correspond to the orientation of the filter medium in large-scale operation. Some of the main principles regarding the leaf test procedure introduced in the literature are the following:

- Probably the most important issue concerning the reliability of the tests is that the slurry sample used should be representative, as the results obtained from filtration tests can only be as good as the sample tested (Thompson, 1993). Also the amount of slurry should be large enough, preferably at least 5–15 dm^3 (Wakeman and Tarleton, 2005). The preferred method is to take the test slurry directly from the actual process stream and perform the tests immediately after collecting the sample. This is also the best way to ensure that the results will not be affected by the ageing of the slurry.
- A sub-sample should always be taken before the tests from the homogeneous suspension in the test vessel for determining or verifying its solids concentration. Also other slurry properties (such as temperature, pH, etc.) and details concerning the possible pre-treatment of the slurry should be recorded. The vacuum level should also be adjusted as accurately as possible before starting the test.
- The actual tests are performed by submerging the filter leaf into an agitated suspension, after which the timer is started and the vacuum is switched on. It is important that the agitation of the slurry is continued throughout the test to prevent the sedimentation of the solids. It has been reported that in most cases sufficient agitation can be achieved by using a wide-faced spatula or by a gentle up and down motion of the test leaf. An excessively high agitation intensity may hinder the cake formation or even change the particle size distribution of the product (Wakeman and Tarleton, 2005).

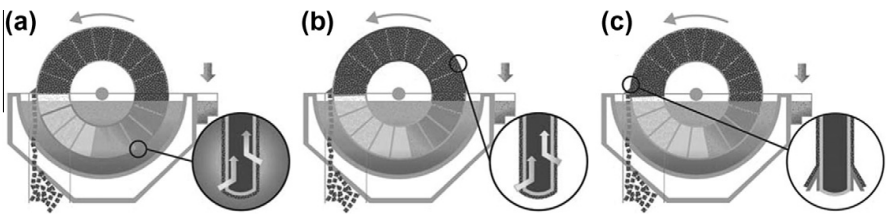

Fig. 1. Cake-formation (a), cake dewatering (b) and cake discharge (c) stages in rotary vacuum disc filters (Outotec, 2010).

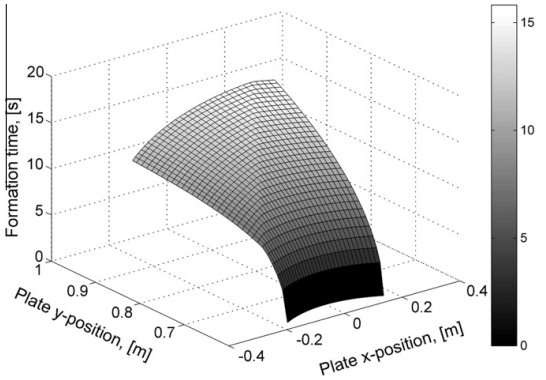

Fig. 2. Formation-time distribution on a ceramic disc filter plate when the rotation speed is 1 rpm and the slurry level in the basin is set to the minimum.

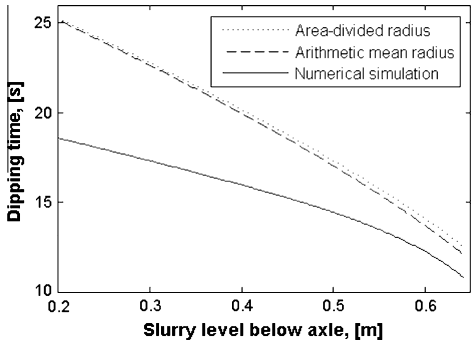

Fig. 3. Calculated dipping times for a ceramic disc filter leaf test. The rotation speed is 1 rpm and the slurry level in the basin is a variable.

- After a preset cake formation time, the filter leaf is lifted from the slurry, and the excess liquor is allowed to drain from the surface of the filter leaf. The filter leaf is then gradually rotated 180° to simulate the cycle of a rotary filter and to ensure that the filtrate drains into the filtrate receiver.
- After a preset dewatering/drying time, the vacuum is switched off and the test data is collected. Typical data includes the filtrate volume, filtrate quality, wet cake weight, cake thickness, and cake discharge characteristics. When collecting the cake sample, it should be remembered that the formation of filter cake on the edges of the leaf typically causes errors in the measured values of the dry mass of cake per filtration area (Thompson, 1993). This is due to the small filtration area of the test element, which means that the relative proportion of the edges of the overall area is considerably higher in the test leaf than in the full-scale element.
- At the end of the tests, the filter cake and the filter medium should be investigated for signs of cake cracking or blinding, wear, or chemical deterioration of the medium.

According to Osborne (1981), all published research papers relating leaf test data with either pilot-scale or full-scale operation suggest that overestimations of filter capacity are obtained with leaf tests. There are several possible reasons for this, but according to Thompson (1993), the most commonly observed sources of inaccuracy are errors in filtration tests, design and commissioning. This

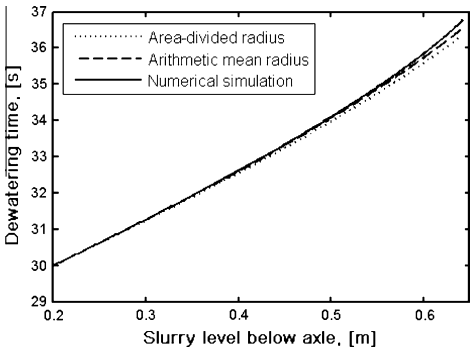

Fig. 4. Calculated dewatering times for a ceramic disc filter leaf test. The rotation speed is 1 rpm and the slurry level in the basin is a variable.

means that the tested sample has not been representative, the test procedure has been unsuitable or performed in an incorrect manner, incorrect safety margins and scale-up factors have been applied, or the auxiliary equipment has been poorly designed or installed. The inaccuracy can thus be mostly associated with human errors, and their role can typically be reduced by arranging an adequate training session, by repeating part of the measurements, and by creating detailed instructions for performing the test procedure in a correct manner. The effective permeability of the filter medium during the full-scale operation compared to the permeability of the medium used in leaf tests also seems to be an important factor. Long term use of the filter medium and repeated cycling tend to lead to an increase of medium resistance due to temporary or reversible medium blinding (Holdich, 1990). Filtrate flow through the filter medium can be high at the beginning of the operation, but it may decrease rapidly due to pore blocking by particles or due to salt deposition and the growth of micro organisms in the pores of the medium (Moritz et al., 2001). Therefore, the real permeability of the medium in process conditions can be significantly lower than the initial permeability that is normally realized in laboratory tests. The tendency of the filter medium to blind is rarely detected in laboratory scale, and pilot-scale tests are therefore advisable if the slurry is suspected to have some blinding properties (Thompson, 1993).

4. Test procedure for ceramic disc filters

The primary aim of the tests performed in this study was to evaluate the suitability of the typical leaf test procedure for ceramic disc filters, and to modify the procedure according to the test results to improve the accuracy of the tests. The experiments car-

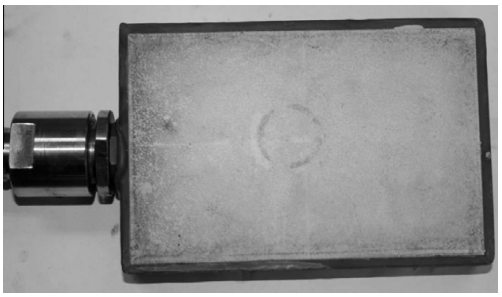

Fig. 5. Ceramic test plate for a capillary action rotary disc filter.

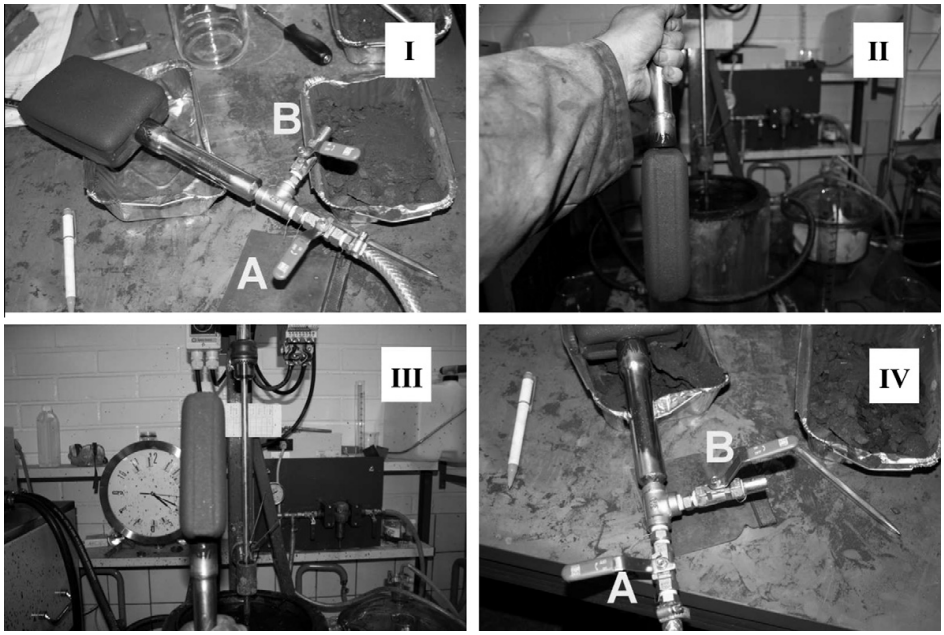

Fig. 6. Methods of cake drying and valve usage before moisture sampling.

ried out can be divided into three separate parts, which were preliminary leaf tests in laboratory conditions, leaf tests performed in process conditions, and the measurement of the performance of full-scale filters at three different field test locations. Some examples of the results achieved in each part are presented in this paper.

4.1. Formation and dewatering time calculation

The preset times for cake formation and cake drying are essentially important for avoiding errors in leaf testing. Both the formation time and drying time are dependent on the distribution valve timing, slurry level on the basin, rotation speed, and scraper position. Furthermore, it should be remembered that the test leaf area is generally 3–10% of the area of the production-sized filtration

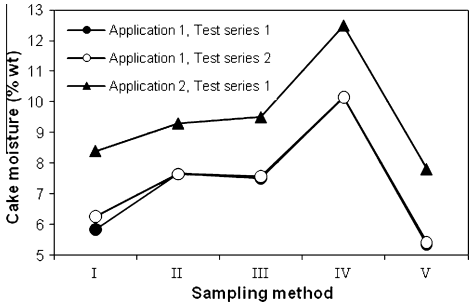

Fig. 7. Effect of different sampling and valve use methods on the residual cake moisture contents in leaf tests. Method I = the test plate handle main valve (A) was closed and sampling performed after 1 min. Method II = normal drying without turning the test plate. Method III = normal drying and the test plate was turned 180°. Method IV = the vacuum was released (valve B) and sampling performed after 1 min. Method V = over drying of the cake for 5 min.

disc sector plate. Traditionally, the formation and drying times are estimated from the radius that divides the disc sector plate into equal areas. A numerical simulation was used to calculate the formation and drying time in order to minimize the errors.

The leaf testing should be done in such a way that the results can be transferred into a larger operational filter and vice versa. This means that the slurry level in the basin as well as the rotation speed and filter diameter should be taken into account when calculating the formation and drying times for leaf testing.

When examining the filter disc sector in its operation mode, it can be noticed that the individual points on the disc sector radial do not spend an equal time in the slurry. Therefore, there is a time distribution that is not even throughout the disc sector area. The geometrical reason for the unevenness of the time distribution is the slurry level in the basin that is basically a chord that divides the disc. The time distribution on a filter disc sector would be simple if the sector element had the vacuum on before the disc is submerged into the slurry. In the normal operation mode of the disc filter, a single plate can be just partially under the slurry surface when the vacuum is set on by the distribution valve. This causes additional deviations in the formation time distribution on a sector plate. The maximum difference between cake formation times can be up to 16 s. between two single points of the disc sector plate when the rotation speed of the disc is 1 rpm, and the slurry level in the basin is set to its minimum.

In order to calculate the dipping times for leaf testing, the formation and drying time distributions have been calculated for the disc sector plate, and using this information, the dipping and drying times can be evaluated correctly. The disc sector plate is divided into a calculation grid with a one degree spacing angle and one centimeter radial spacing. The formation and drying times for each grid point are calculated separately. The grid points form a mesh with 'miniature' disc sector elements, and each of these elements has four corner grid points with associated formation and drying times. The sector elements are now associated with the

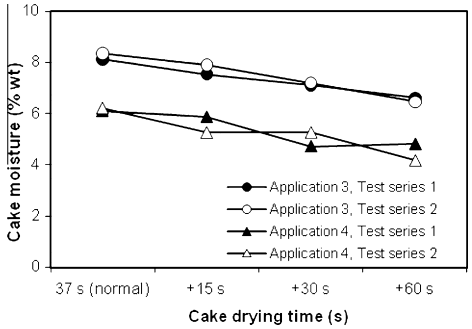

Fig. 8. Effect of overdrying of 15, 30 and 60 s on the residual cake moisture contents for two parallel test series of two different applications.

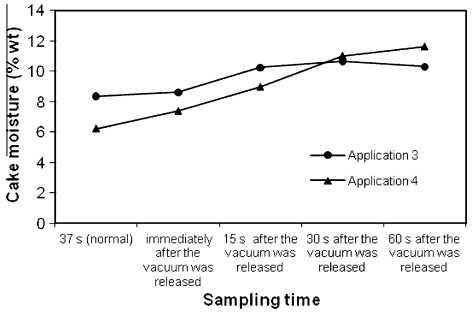

Fig. 9. Effect of vacuum release on the cake moisture content in leaf tests.

mean of the cake formation and drying time from the four corner calculation points, and this mean value is divided by the sector element area. These elemental timings are summed up and multiplied by the total sector area, which now gives the proper average for the timings used in leaf testing. An example of the calculated formation time distribution on a filter sector plate is presented in Fig. 2 when the rotation speed of a disc is 1 rpm and the slurry level in the basin is set to its minimum.

The traditional way of calculating the cake formation and drying time for leaf testing is usually estimating the residence time of a single point on the disc that is located on the radial line. The point is either the arithmetic mean of the sector plate inner and outer radii, or on a radius which divides the sector element into two equal size areas. This method does not produce realistic estimates for the timings used in leaf testing. The differences in the formation time and dewatering time are shown in Figs. 3 and 4.

As can be seen in Fig. 3, there is a noticeable error regarding the dipping times acquired either from the area-divided radius or the arithmetic mean radius. This kind of error seriously affects the estimated capacities of the ceramic disc filter and other responses, as well. On the other hand, the difference in the calculated dewatering time (shown in Fig. 4) is not as serious as in the case of the dipping time. It should be kept in mind that the errors created in the early stages of the testing procedure tend to accumulate when estimating the final performance of the filter, and therefore, it is essentially important to calculate the formation and dewatering times correctly.

4.2. Leaf tests in laboratory conditions

The target of the preliminary tests performed in laboratory conditions was to define the correct procedure for performing the actual tests and for collecting the cake samples, as well as to determine the influence of agitation conditions on the test results. An example of a test plate used in the leaf tests for ceramic capillary action rotary disc filters is presented in Fig. 5. The surface characteristics and internal structure of the ceramic plates are similar to the actual full-scale plates, and the dimensions of the plates are 150 mm $\times$ 100 mm $\times$ 2 sides, giving a total filtration area of approximately 0.03 m^2.

4.2.1. Influence of different ways of using valves and cake sampling

The first tests were carried out to study the effects of the different ways of using the valves in the plate handle (Fig. 6), and also to determine whether the test plate should be turned to an upright position during the cake dewatering stage, as suggested in the literature for conventional disc filters. The test series were carried out by using different metal concentrate samples that had been obtained from industrial processes as slurry. The first test series included five filtration experiments per series, and the following methods were used:

(I) The test plate handle main valve (A) was closed after the cake dewatering time, which means that the vacuum was discontinued but there was still a vacuum inside the test plate due to the nature of the ceramic material. A sample for determining the cake moisture content was scraped from the plate 1 min after valve A had been closed. Fig. 6(I).
(II) The cake moisture sample was scraped from the plate immediately after the dewatering time. The valves were not closed. The test plate was not turned to an upright position during dewatering. Fig. 6(II).
(III) The cake moisture sample was taken immediately after the dewatering time. The valves were not closed. The plate was turned to an upright position during the dewatering. This can be considered as the method that most accurately describes the operation of full-scale rotary disc filters. Fig. 6(III).
(IV) The main valve (A) was closed after the dewatering time and the deaeration valve (B) was opened. This means that atmospheric pressure was returned inside the plate. A sample for determining the cake moisture was taken 1 min after turning the valves. Fig. 6(IV).
(V) Reference point. The cake was dewatered for approximately 5 min to determine the minimum moisture content that can be achieved. No valves were used.

The results of cake sampling and valve use tests for two different applications are presented in Fig. 7. The applications 1 and 2 presented in Fig. 7 are magnetite and chrome ore concentrate slurries, respectively. The slurry densities used in the test filtrations were the same as in industrial process applications. It can be concluded from these results that the vacuum stayed inside the plate when the valve between the vacuum source and the plate was closed (sampling method I) and dewatering continued, resulting in a decreased cake moisture content that approached the values of overdrying (V) relatively fast. On the other hand, if the vacuum was removed from the plate (method IV), the cake moisture increased significantly, meaning that moisture was absorbed back into the cake from the pores of the ceramic filter medium. The results of methods II and III, where the orientation of the plate was changed during the dewatering stage, indicated that the plate orientation did not have a significant effect on the residual cake moisture contents. The repeatability of the results was very good, as can be concluded by comparing the results of the two parallel test series carried out for Application 1.

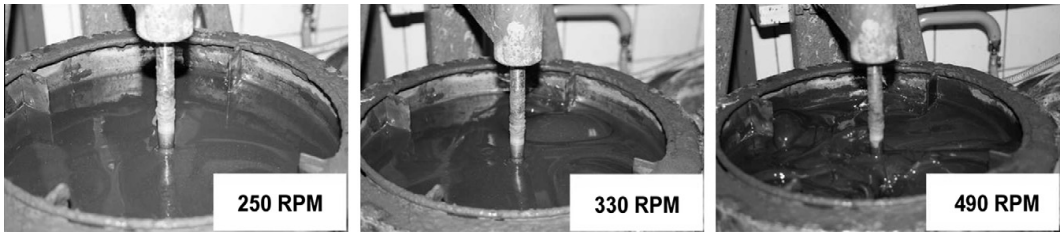

Fig. 10. Behavior of the slurry with different rotation speeds: 250, 330 and 490 rpm.

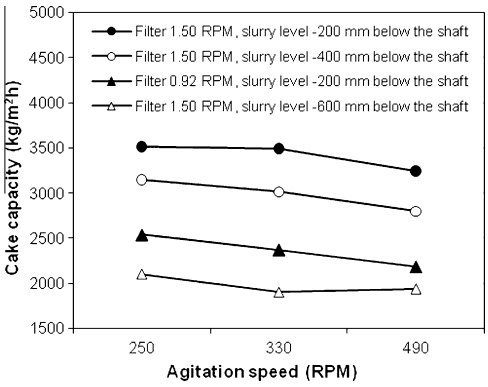

Fig. 11. Effect of pitched blade turbine rotation speed on filter cake capacity.

It can be concluded on the basis of the results presented in Fig. 7 that it is very important to analyse the moisture content of the cake sample immediately after the predefined dewatering time has been reached. Otherwise, the moisture content begins to change rapidly and the relative error in the obtained results can easily be as high as ±25%. The results also show that it does not make a difference whether the plate is held in an upright position during the dewatering or not. This is a clear difference compared to the procedure described in the existing literature, and it is most probably caused by the fact that the vacuum level with ceramic leaf is much higher than what can be achieved with conventional filter cloth. Typically, the pressure difference in the tests with ceramic leaf is between 0.90 and 0.95 bar. Methods I and IV showed that the cake moisture content began to change rapidly if the vacuum was either discontinued or released. The rate of changes in the cake moisture content was studied more closely by performing some additional test series with different test slurries. Fig. 8 shows the effect of overdrying on the cake moisture contents for two different applications. Two parallel test series were performed with both applications.

As can be seen in Fig. 8, the cake moisture content continues to decrease regularly after closing valve A. This can be explained by the vacuum that remains inside the test plate. Since ceramic filter material does not allow air to flow through the pores, the only route to the stabilisation of the pressure is through the flow of liquid from the cake into the plate. The effect of vacuum release (valve B) on the cake moisture content for two different applications is presented in Fig. 9. The applications 3 and 4 in Fig. 9 are from an iron pyrite process using two different ceramic plate types. These results show that the moisture content of the cakes begins to increase immediately after opening the valve. The only explanation for this is that the liquid from the pores of the plate begins to flow back into the cake. This could also be observed during the tests, as the surface of the plate wetted quickly after releasing the vacuum.

4.2.2. Influence of agitation characteristics

Laboratory leaf testing, and especially field leaf testing, is typically conducted in a vessel or a bucket with various ways of stirring, and the agitation intensity depends heavily on the person who is performing the tests and on the availability of different kinds of mixing equipment. This means that agitation can be a significant source of error and deviations in the test results. Bottom-

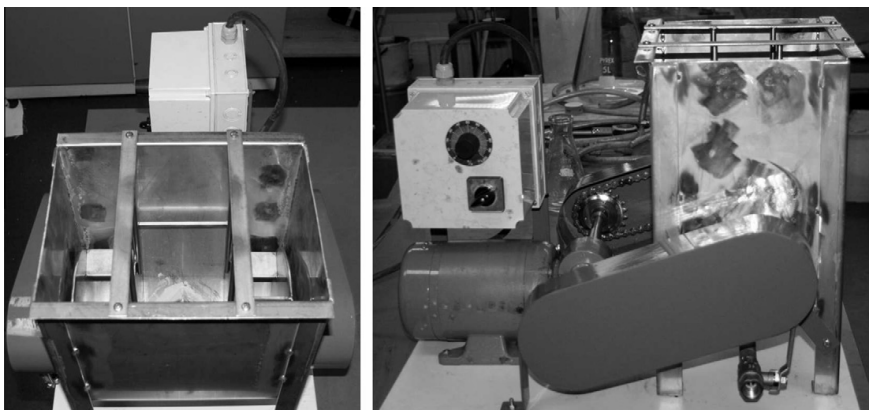

Fig. 12. Prototype of an agitation vessel with paddle wheel rotors (PWR agitator).

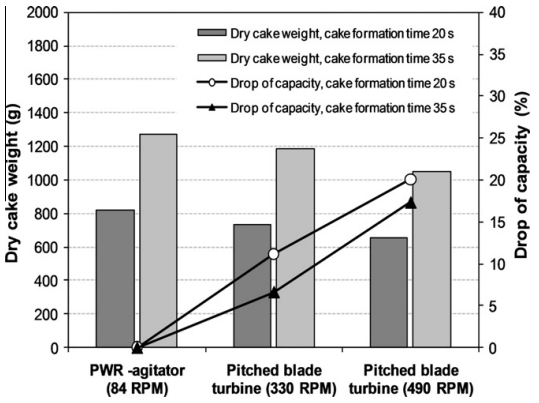

Fig. 13. Comparison of dry weights of the magnetite cakes obtained from leaf tests with the PWR agitator and conventional agitation with a pitched blade turbine.

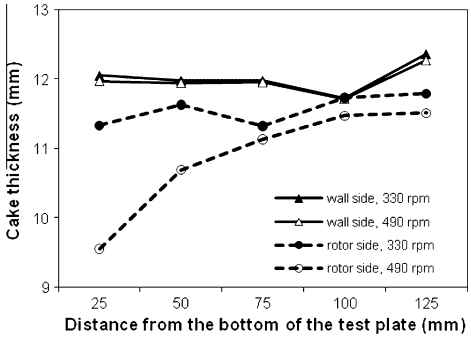

Fig. 14. Magnetite cake thickness profile on the rotor side and wall side of the plate with pitched blade turbine speeds of 330 and 490 rpm in a conventional agitation vessel with baffles.

feed rotary disc filters are often used for processing rapidly settling high concentration slurries, which means that the agitation intensity that is required for preventing the sedimentation of the solids and for maintaining the homogeneity of the slurry is typically quite high. The target of this part of the preliminary tests was to evaluate

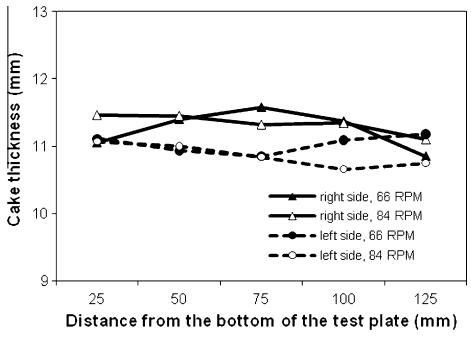

Fig. 15. Magnetite cake thickness profiles on different sides of the plate with the PWR agitator at rotation speeds of 66 and 84 rpm.

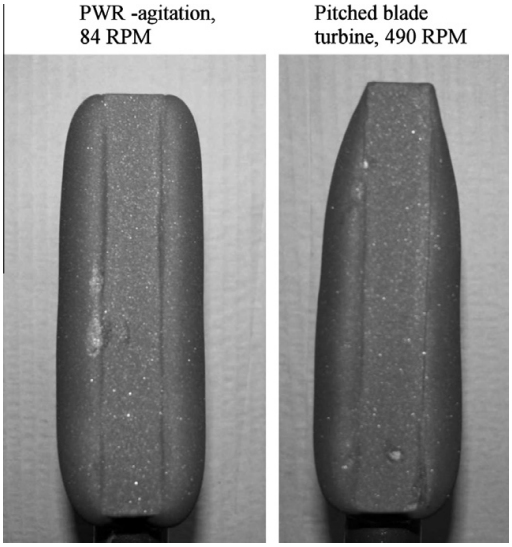

Fig. 16. Differences in magnetite cake thickness uniformity between the PWR agitator (on the left) and pitched blade turbine agitation (on the right).

the deviations caused by different kinds of mixing methods on the capacity of the filter and on the moisture content of the cakes.

Most commonly, the test slurry is agitated by using some kind of a rotor type agitator, which typically requires fairly high rotation speeds to form a homogeneous slurry. The conventional agitation vessel used in this study was equipped with a pitched blade turbine, a cooler jacket and four baffles which were mounted on the inner walls of the vessel to prevent the formation of laminar flow areas near the wall and to maximize the homogeneity of the slurry.

The preliminary leaf tests performed in this part of the study were carried out by using four different cake formation times and three different agitator rotation speeds, which were 250, 330 and 490 rpm. The slurry used in the tests was once again a highly concentrated (>60 w-%) mineral concentrate suspension (magnetite). Fig. 10 shows photos taken from the surface of the slurry with different rotation speeds. The rotation speed of 330 rpm represents a typical agitation intensity, and 250 rpm was near the minimum intensity applicable to the considered slurry because a clear liquid layer was noticed to form slowly on the surface of the slurry, which indicated weak agitation. Moreover, with the rotation speed of 250 rpm, the cake moistures were slightly higher when compared to cakes formed with higher speeds, and there were signs that the slurry composition changed over time due to settling. This is why the rotation speed of 250 rpm was omitted from the comparison. Rotation speed of 490 rpm caused high intensity agitation, and strong turbulence was observed, as can be seen in Fig. 10.

The filter capacities with different rotation speeds are presented in Fig. 11. As can be seen, the highest capacities were in all cases gained with the slowest agitation speed. The highest agitation speed gave on average a 10% lower cake capacity. The real agitation effect in a full-scale filter is naturally dependent on the filter basin agitation, but in addition, also the rotation of the disc itself causes relative motion of the slurry in the cake formation zone, affecting the cake loss. The adjustment of a proper agitation speed in leaf testing is very difficult, and a single agitation intensity for all applications cannot be generalized.

Based on the results presented in Fig. 11 and also on the results presented later in this paper (Figs. 13–15), it was concluded that

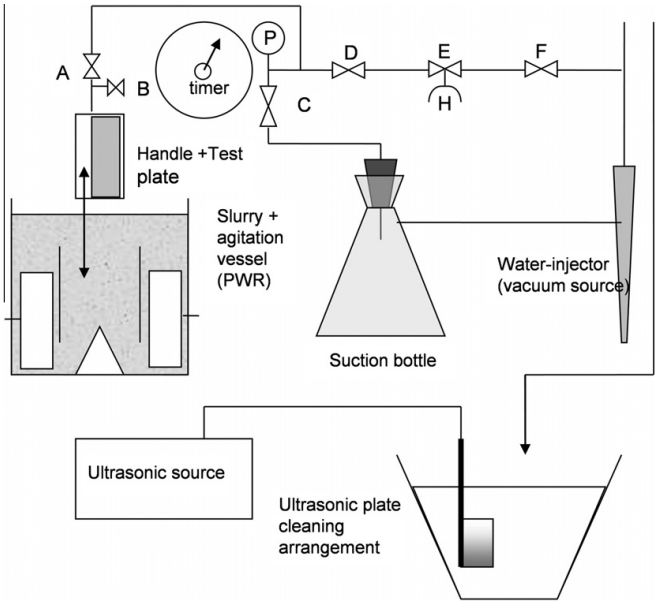

Fig. 17. Standard leaf test assembly used in tests performed in process conditions.

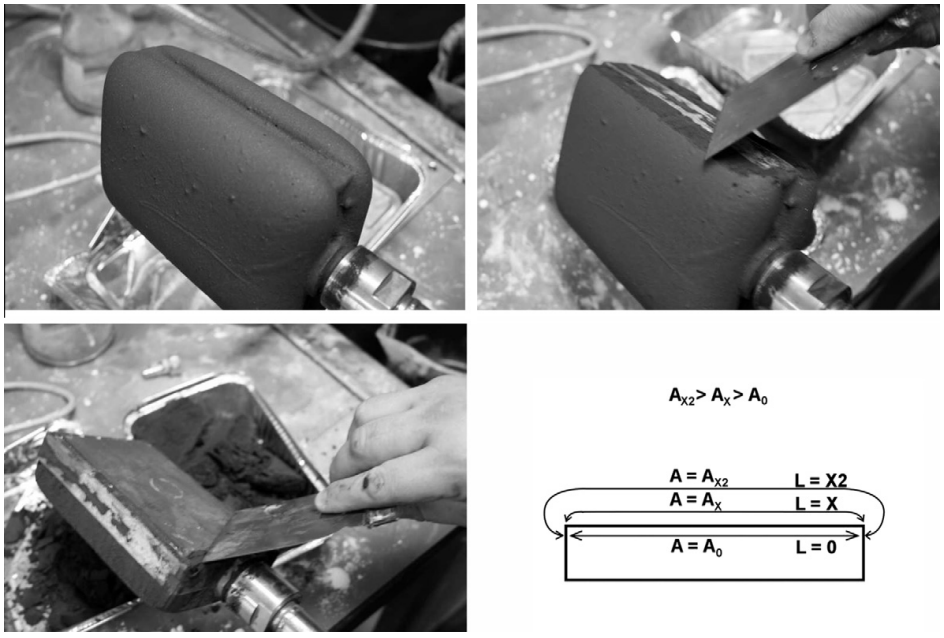

Fig. 18. Scraping of the cake formed over the edges of the test plate before the actual sampling.

agitation of the slurry with a rotor type mixer is not the optimal way regarding the comparison of the test results with full-scale filter units. For this reason, a new kind of agitation device was designed and tested. Extensive test series were carried out to compare the new mixing device with a conventional rotor agitation. A so-called paddle wheel rotor agitator (PWR agitator) was developed after a few unsuccessful attempts with other mixing devices. The main idea came from the fact that the agitation charac-

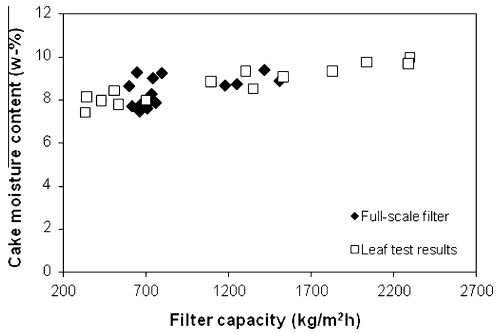

Fig. 19. Comparison between the results obtained from the leaf tests and the values measured from full-scale filters in the hematite process application.

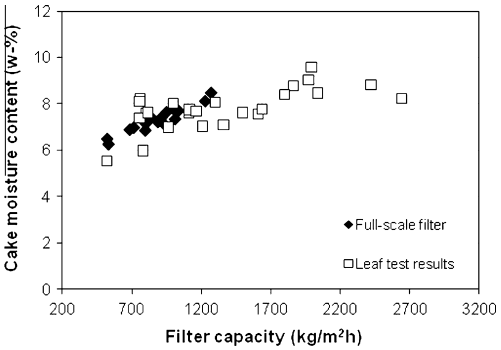

Fig. 20. Comparison between the results obtained from the leaf tests and the values measured from full-scale filters in the pyrite process test application.

teristics applied in the tests should correspond to the ones in the full-size filter (Thompson, 1993). The agitation in disc filters is commonly performed with cradle-type agitators mounted on a horizontal axis at the bottom of the slurry basin, and the discs rotate in the spaces between these agitator elements. This means that the direction of the slurry flow created by cradle-type agitators is mostly parallel with the surfaces of the discs, whereas a rotor type agitator creates a flow perpendicular to the surface of the test leaf. The general structure of the PWR agitator designed in this study is presented in Fig. 12.

The PWR agitator consists of a tank and two large vertical rotors which are installed in the tank symmetrically so that the space, where the leaf tests are performed is between the rotors. Due to the large diameter of the rotors, much lower agitation speeds can be used than with typical rotor mixers. The symmetrical installation of the rotors ensures equivalent flow patterns and slurry velocities on both sides of the test plate. One disadvantage of the new PWR agitator is that the required slurry volume is relatively high, approximately 15 dm^3. On the other hand, this will also be an advantage if the slurry is easily available, as with larger volumes the changes in the solid concentration of the slurry during the test series become smaller. The PWR agitator was compared to conventional rotor agitation with a pitched blade turbine. The tests were carried out using a pitched blade turbine with rotation speeds of 330 and 490 rpm and the PWR agitator with a rotation speed of 84 rpm. Fig. 13 presents the dry weights of the cakes obtained from the tests with different agitation methods/speeds and a relative

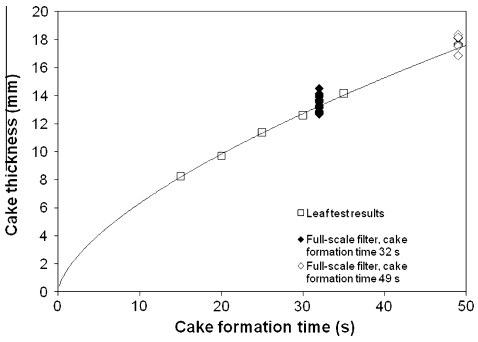

Fig. 21. Comparison between the results obtained from the leaf tests and the values measured from full-scale filters in the chrome ore process application.

capacity drop compared to PWR agitation. The tests were performed with a magnetite slurry using two different cake formation times.

As can be seen in the comparison presented in Fig. 13, the pitched blade turbine with a high agitation intensity (490 rpm) gave up to 20% lower capacities than the PWR agitator. It can be assumed that the PWR agitator is capable of creating the most optimal mixing conditions for the leaf tests. To verify this, another comparison was carried out to determine the influence of agitation conditions on the shape and homogeneity of the filter cakes. This was done by measuring the cake thicknesses at five different locations and from both sides of the test plates. These tests were carried out by using a pitched blade rotor with speeds of 330 and 490 rpm and the PWR agitator with speeds of 66 and 84 rpm. A cake formation time of 10 s. was used in all of the tests. Examples of the results are presented in Figs. 14 and 15.

Fig. 14 shows that with the pitched blade turbine, the agitation was most intense near the rotor, causing significant cake loss especially when the rotor speed was 490 rpm. The maximum difference in the measured cake thicknesses in the same tests was over 25%. It was observed that these cake losses occurred mainly on the rotor side and in the bottom parts of the plate. At the lower rotation speed of 330 rpm, the cake loss was clearly smaller on the rotor side, whereas on the wall side the differences between the different rotation speeds were near zero. When the PWR agitator was used, no differences were observed between the different rotor speeds, as can be seen in Fig. 15. However, small differences between the different sides of the plates were observed, which may have also been caused by the differences in the permeabilities of the different sides of the test plate. Examples of the shapes of the filter cakes are given in Fig. 16. As a summary, it can be concluded that PWR agitation can provide more uniform cakes than conventional mechanical agitation with pitched blade type rotors, thus improving the sampling and accuracy of the test results.

4.3. Leaf tests in process conditions

The main aim of the leaf tests performed in the process conditions at three different test locations was to compare the capacities and cake moisture contents obtained from the leaf tests with the corresponding values measured from the real full-scale filter. The leaf tests were always carried out using the basic correlations between the disc filter operation parameters and the values of cake formation and drying times corresponding to the operation of full-scale filters at the test locations. Simultaneously with the leaf tests, the cake thicknesses, filter capacities and residual cake moisture contents were measured from the operational full-scale filters

to compare them with the results obtained from the leaf tests. The leaf test assembly that was used in these tests is presented in Fig. 17. The equipment included the PWR agitator and the new test plate handle which were designed and tested previously. The standard leaf test assembly also included a suction bottle for filtrate collection, a control board including operating valves and a pressure gauge, an ultrasonic plate cleaning device, and a vacuum source with connecting hoses.

The leaf tests were performed according to the guidelines presented earlier. One possible source of inaccuracy in the test results is the edge effect, which is typical especially with slurries that filter quickly and form cakes over 15 mm in thickness. The edge effect was minimized in this study by scraping away the "extra" cake before collecting the actual cake, as shown in Fig. 18.

The cake growth can be thought to increase the filtration area, as presented in Fig. 18. When the cake thickness increases to $L_x = L_0 + dL$, the new filtration area is $A_x = A_0 + dA$. The filtration area growth dA is the same in the full-scale filter, but because $A_{0,full-scale\ plate} \gg A_{0,test\ plate}$, the effect on the capacity is negligible. Additionally, cake growth over the plate edge rarely exists in full-scale filters due to the wear between the moving edge of the plate and the slurry. Thus, basically, in leaf tests, an increase of the filtration area causes the observed cake growth and overestimation of the cake capacity. Because the change in filtration area is difficult to measure and estimate, the removal of the overgrown cake is the best solution. After each test filtration, the used test plates were regenerated with backwash and ultrasonics so that visible depositions of fine particles were removed and the plate surface became clean. A treatment of a few seconds per side was sufficient in most cases. After backwashing and ultrasonic treatment, the specific permeability of the plate was measured to ensure that the plate was cleaned successfully and that the next test was comparable to earlier measurements. The measurements were performed with three different test applications. Examples of the comparisons between the results obtained from the leaf tests and the values measured from full-scale filters are presented in Figs. 19–21. Fig. 19 shows the filter capacities and cake moisture contents for the hematite application, and as can be seen in this figure, the results obtained from the leaf tests are very similar to the performance of the full-scale filter. Similar results for the pyrite application are presented in Fig. 20, and the same conclusions can be drawn also according to these values.

Fig. 21 presents the measured filter cake thicknesses from the leaf tests and from the full-scale filter in the chrome ore application with two different cake formation times. The leaf test results obtained by using the procedure developed in this study can be successfully used to describe the real process behavior.

5. Conclusions

The suitability of the typical leaf test procedure for ceramic disc filters was evaluated in this experimental study in three different parts. The target of the preliminary leaf tests, which were performed in laboratory conditions, was to define the most significant sources of errors and their influence on the test results. It was found that for example the sampling and agitation of the slurry during the tests may cause considerable differences in the results. The leaf test procedure was modified according to the preliminary tests, after which three series of leaf tests were performed in process conditions by using the improved test procedure. The comparison of the results obtained from the leaf tests carried out in process conditions and the measured performance of the full-scale filters showed that the test procedure developed in this study could be successfully used for describing the real process behavior of ceramic capillary action disc filters.

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Article II

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EXPERIMENTAL STUDY OF THE INFLUENCE OF PROCESS VARIABLES ON THE PERFORMANCE OF A HORIZONTAL BELT FILTER

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This paper introduces an experimental study that was carried out to determine the influence of several different process variables on the performance of a horizontal vacuum belt filter. Laboratory scale tests were performed according to the multilevel full factorial design by using a conventional Buchner test unit. The investigated process variables were the volumetric feed rate of the slurry, dewatering time, amount of wash liquid used and solids concentration of the feed suspension. The parameters that were used for describing the performance of the process were the cake moisture content, production capacity of the filter and the purity of the washed filter cake. The results obtained from these tests were used to create different kinds of regression models for all of the studied responses. Several different kinds of test designs were also extracted from the initial full factorial design for defining the minimum number of tests required to obtain satisfactory results for the investigated application. Comparison of different models showed that the amount of test work could be efficiently reduced by utilizing the statistical design of experiments and empirical modelling tools.

INTRODUCTION

Sizing and preliminary running parameters for belt filters are usually gathered by conducting experiments with a conventional Buchner test unit. The test work is quite straightforward if the parameters used for describing the filter performance can be assumed linear within the process variable range. The amount of test work needed will become considerably larger if there are such performance parameters (i.e. responses) that are known to be nonlinear. One of these nonlinear responses that are typical for belt filters is the purity of the washed filter cake or washing efficiency. In this paper there are four process variables and three process responses used. Normally if the responses can be assumed to be sufficiently linear over the whole variable range one could do experimental design that is based on full factorial design having 2^4 = 16 experiments. This 2^4 design means that all four variables have only two levels (high and low) and the 16 experiment set-up can be minimized quite safely by applying fractional factorial designs. These fractional factorial designs like 2^{4-1} (= 8 experiments) can be used to minimize the experimental work load without the fear of losing information if only we are sure that the responses behave in a linear way.

Now, when responses like filter cake purity need to be taken into consideration it is not possible to use the linearity assumption when trying to minimize the amount of test work. Therefore, we concluded a full factorial design for four variables all having three levels (3^4 = 81 experiments). This design was complemented with 14 duplicate points and 18 additional experiments where the filter cake washing was not performed at all which means that the total number of experiments was 113. In order to gather the most accurate information for a belt filter with minimum experimental points one has to use an experimental design scheme other than full factorial design. In this work we separated several subsets of different designs from the full factorial design. The subset designs that are studied here are: Box-Behnken design (25 experiments), Central Composite design (25 experiments), Taguchi L16b orthogonal matrix (16 experiments) and Taguchi L9 orthogonal matrix (9 experiments).

The parameters that were used to describe the performance of the filtration process were the cake moisture content, production capacity of the filter and purity of the washed filter cake. The process responses of production capacity and cake moisture content were modelled with four different equations and the purity of washed filter cake was modelled with five different functions. All three responses were modelled with the linear, linear with interactions, pure quadratic and full quadratic functions. Furthermore, the cake purity was modelled with the modified exponential decay model[1].

MATERIALS AND METHODS

The test slurries were prepared from commercial native wheat starch Latitec 1000 R of Ciba Specialty

Chemicals Oy (Anjalankoski, Finland). The slurries were prepared by mixing the starch powder with water and homogenizing before the filtration experiments. Three different solids concentration slurries were used namely 35, 40 and 45 wt % ($kg_{Dry\ solids}$ / kg_{Slurry}). Three different slurry volumes of 100, 200 and 300 ml were used. Chloride ions were used as tracers for filter cake purity measurements and the chloride content of each slurry was adjusted by the addition of sea salt in such way that the chloride content was 20,000 ppm. The filtered cakes were washed with water so that the amount of wash water used corresponded to the wash ratios of 2, 5 and 10 (kg_{Water} / $kg_{Dry\ solids}$). The air dewatering times used were 0, 30 and 60 seconds.

The filtration experiments were conducted with a conventional Buchner test unit provided by Larox Corporation. The filtration area of the Buchner unit was 0.01 m^2. The cake purity was monitored by taking a sample of the washed cake, reslurrying in deionized water and allowing to settle. Conductivity of the supernatant liquid was measured with a Knick Konduktometer 702.

MODEL FUNCTIONS AND EXPERIMENTAL DESIGNS

The functions that were common for all of the measured process responses were linear (equation (1)), linear with interactions (equation ((2)), pure quadratic (equation (3)) and full quadratic (equation (4)) functions such that

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 \quad (1)$$

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_{21}x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 \quad (2)$$

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{44}x_4^2 \quad (3)$$

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_{21}x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{22}x_4^2 \quad (4)$$

where y is the measured response, x the process variable and b the coefficient. The subscripts have the following meanings: 0 ≡ constant term, 1 ≡ solids concentration, 2 ≡ slurry volume, 3 ≡ wash ratio and 4 ≡ drying time. Further, the cake washing efficiency was modelled with the function

$$y = b_1x_1 + b_2x_2 + \exp(-b_3x_3) + b_4x_4 \quad (5)$$

Here, the exponential term represents the exponential decay model as presented by Salmela and Oja[1]. The original exponential decay model has been supplemented with linear terms of variables other than wash ratio.

The experimental designs used were based on our original experimental setup which consisted of a full factorial design for four variables all having three levels (3^4 = 81 experiments). This design was complemented with 14 duplicate points and 18 additional experiments (without filter cake washing) which means that the total amount of experiments in the final design was 113. The subsets of experimental designs that were taken out from the full set were: Box -Behnken design (25 experiments); Central Composite design (25 experiments); Taguchi L16b orthogonal matrix (16 experiments); and Taguchi L9 orthogonal matrix (9 experiments). When considering the experimental design and the model function to be used with the design one should always pay attention to the number of parameters that need to be resolved and to the number of experiments that are to be included in the design.

The Box-Behnken and Central Composite designs are response surface designs that can fit a full quadratic model and both designs use three levels for each variable. The Central Composite design used in this paper is the so-called Central Composite Faced design. Both Box-Behnken and Central Composite Faced (CCF) designs use just three variable levels. The difference between the Box-Behnken design and CCF is that Box-Behnken design can be expected to be poorer in prediction ability close to the corners of the cube that encloses the design, because unlike CCF designs they do not include points at the corners of that cube[2].

The Taguchi arrays were tested because traditional ways of creating multi level fractional factorial designs tend to lead into experimental designs that are larger than the response surface designs. Therefore, we took the Taguchi arrays into consideration. In the Taguchi L9 array all four variables have three levels and in the L16b array one variable has four levels and the remaining three variables have three levels. The Taguchi arrays are orthogonal experimental design arrays as factorial designs, but they are also saturated design arrays that makes the interaction estimation difficult to resolve. The Taguchi arrays are very attractive due to their small amount of experiments. Table 1 summarizes the characteristics of the different functions and experimental designs considered in this study.

The quality and reliability of the experimental designs and model functions can be evaluated by comparing the coefficients of determination values (R^2), root mean squared error (*RMSE*) and root mean squared error of prediction (*RMSEP*) values. These can be calculated with the following:

$$R^2 = 1 - \frac{\sum_i (y_i - f_i)^2}{\sum (y_i - \bar{y})^2} \quad (6)$$

$$RMSE = \sqrt{\frac{\sum_i (y_i - f_i)^2}{n}} \quad (7)$$

$$RMSEP = \sqrt{\frac{\sum_i (y_i - \hat{y}_i)^2}{n}} \quad (8)$$

where f_i is the modelled response value, n the number of observations and $\hat{y}$ the predicted response value. The *RMSE* is used for the data points that are included in the model while the *RMSEP* is used for evaluating the external validation data, i.e. data which has not been used in the modelling phase.

RESULTS

The variation in the results obtained from the experiments carried out in this study was:

- Filter capacity: 5 to 81 $kg_{Dry\ solids}$ / m^2 h
- Cake moisture content: 34 to 65 wt %
- Dimensionless scaled conductivity: 0.004 to 1.

In Table 2 are the R^2-values for each model used when filter capacity is the modelled response. Similar values are shown for the cake moisture content model in Table 3 and for cake purity in Table 4. Tables 5-7 contain *RMSE*- and *RMSEP*-values for the measured process responses. The *RMSE* is used for the models that contained all the experimental values and *RMSEP* is for models which have been tested with those data points that had not been used in the modelling phase.

Figure 1 presents an example of the prediction ability of the different models. Here, the capacities, which were predicted by using four different models created for the Full design (95 experiments), are plotted against the experimentally determined filter capacities. If the predictions given by the models were perfect, all of the points would lie on the diagonal line. These kinds of figures are very useful for estimating the quality of the models, for detecting possible nonlinear trends and also for estimating the maximum prediction error of the models. The correlation coefficients for the different kinds of models, which were also presented in

Function	No. of parameters	Design	No. of experiments
Linear	5	Full design	95 / 113
Linear + interactions	11	Box-Behnken	25
Pure quadratic	9	Central-Composite	25
Full quadratic	15	Taguchi L16b	16
Exponential decay	4	Taguchi L9	9

Table 1: The number of parameters in the functions used in the modelling and the number of experiments in the different experimental designs considered.

Function	R^2 (-)				
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	0.79	0.80	0.78	0.60	0.86
Linear + interactions	0.85	0.85	0.86	0.79	
Pure quadratic	0.88	0.93	0.90	0.88	1.00
Full quadratic	0.95	0.97	0.98	0.98	

Table 2: The R^2-values for different models and experimental designs with filter capacity as the modelled response. Full design consists of 95 experiments.

Function	R^2 (-)				
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	0.92	0.91	0.96	0.99	1.00
Linear + interactions	0.93	0.94	0.97	1.00	
Pure quadratic	0.93	0.94	0.98	0.99	1.00
Full quadratic	0.94	0.96	0.98	1.00	

Table 3: The R^2-values for different models and experimental designs with filter cake moisture as the modelled response. Full design consists of 95 experiments.

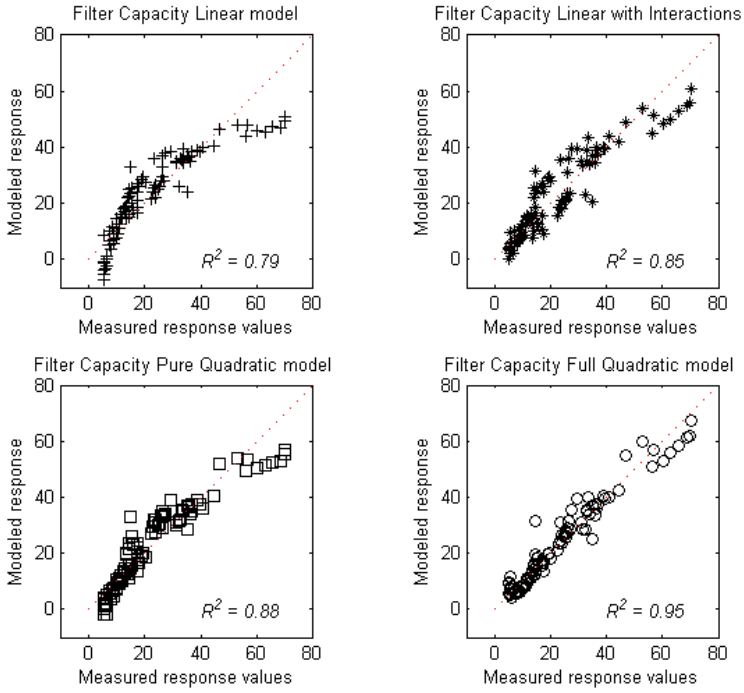

Figure 1: Linear model, linear with interactions model, pure quadratic model and full quadratic model vs. the measured values for filter capacity.

Table 2, are also given in these figures.

DISCUSSION AND CONCLUSIONS

As can be seen from the values presented in Tables 2-4 one should be extra careful when thinking of minimizing the number of experiments. Full design is in all cases the dataset that contains the largest number of experiments and this should therefore be considered as the point of comparison for the rest of the designs. The accuracy of the models created for the full design naturally increases as more components are added into the models. The full quadratic model results in the highest correlations and lowest prediction errors in most cases. An exception can be seen in the case of the cake purity models where the exponential decay model gives by far the highest correlation and the lowest error. This is a promising result since the equation that was used for creating the model is quite simple (equation (5)). Generalization of this behaviour, however, requires more cake washing experiments to be performed with various test materials.

The behaviour of the models created for Box-Behnken and Central Composite designs is rather similar in all

Function	R^2 (-)				
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	0.32	0.53	0.69	0.47	0.66
Linear + interactions	0.33	0.53	0.70	0.56	
Pure quadratic	0.73	1.00	0.98	0.83	1.00
Full quadratic	0.74	1.00	1.00	0.97	
Exponential decay	0.98	1.00	0.98	0.99	1.00

Table 4: The R^2-values for different models and experimental designs with cake purity as the modelled response. Full design consists of 113 experiments.

cases. For the capacity and cake moisture models, the correlations and prediction errors of both of these designs are about the same as those for the Full design. Significant differences can be observed when the models for the cake purity are compared since here the use of quadratic models results in 100% prediction accuracy but in extremely high prediction errors. It is thus quite obvious that the structure of the data in these designs is not suitable for the quadratic models. The exponential decay model seems to perform very well for both Box-Behnken and Central composite designs.

The Taguchi L16b array is a special case when looking at the filter capacity models and cake moisture models. The correlations are quite high but also the prediction errors are large especially for the more complicated quadratic models. One reason for this is that the Taguchi L16b array has the wash water volume variable in four levels. It means that the modelled process responses also have information from some of the unwashed cake samples.

The Taguchi L9 array definitely has too few experiments for anything other than linear models. This is true especially if one cannot be sure of what type of function should be used when modelling the particular phenomena. Mathematically, one can always use a pure quadratic model to create a model for four variables but the end result is that the degrees of freedom will be used up and the model fits perfectly to the experimental points. This does not, however, necessarily mean that the model would be usable for describing the additional data points of the process response. Tables 5-7 present the prediction errors for all models and as can be noticed from these values, the Taguchi L9 array seems to give fairly nice models with low prediction errors in most of the cases.

The overall conclusion that can be made according to the results presented in this paper is that although different kinds of experimental designs can be efficiently used for reducing the amount of experiments, it is important to always verify the validity of the obtained results and models. Comparisons

Function	RMSE ($kg_{Dry\ solids}$ / m^2 h)	RMSEP ($kg_{Dry\ solids}$ / m^2 h)			
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	7.80	8.57	7.49	46.56	9.47
Linear + interactions	6.81	7.94	6.57	76.32	
Pure quadratic	6.03	6.92	6.16	41.54	8.90
Full quadratic	4.24	5.41	4.97	333.44	

Table 5: The *RMSE*- and *RMSEP*-values for different models and experimental designs with filter capacity as the modelled response.

Function	RMSE (w-%)	RMSEP (w-%)			
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	1.15	1.65	1.25	1.22	1.31
Linear + interactions	1.16	2.28	1.27	1.25	
Pure quadratic	1.10	2.31	1.30	1.26	1.21
Full quadratic	1.11	2.44	1.32	5.86	

Table 6: The *RMSE*- and *RMSEP*-values for different models and experimental designs with filter cake moisture as the modelled response.

Function	RMSE (-)	RMSEP (-)			
	Full design	Box-Behnken	Central Composite	Taguchi L16 b	Taguchi L9
Linear	0.30	0.69	0.67	0.32	0.61
Linear + interactions	0.31	0.70	0.67	0.41	
Pure quadratic	0.19	2.17	2.34	0.21	2.08
Full quadratic	0.20	2.20	2.34	2.71	
Exponential decay	0.05	0.05	0.04	0.05	0.06

Table 7: The *RMSE*- and *RMSEP*-values for different models and experimental designs with cake purity as the modelled response.

between the obtained models also showed that significant reductions in the amount of experiments can be achieved if enough attention was paid to the selection of the type of the model equation. This could be seen especially in the models created for cake purity.

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EVALUATION OF CONSOLIDATION-SEDIMENTATION PROPERTIES FOR THE BATCH GRAVITY SEDIMENTATION OF CONCENTRATED SUSPENSIONS

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Consolidation-sedimentation behaviour of consolidated sediments under the action of gravity was investigated using highly concentrated suspensions of titanium dioxide particles under conditions of various pH, initial height and initial concentration. The average consolidation ratio of the consolidated sediment was analyzed on the basis of the simplified analytical solution obtained using the modified Terzaghi model with the moving Lagrangian coordinate system. The modified average consolidation coefficient increased in almost direct proportion to the total volume of solids per unit cross-sectional area, which changes the driving force of consolidation-sedimentation due to the overlying weight of the solid particles. The variations with time of the height of the consolidated sediment were adequately modelled using the analytical solution describing the average consolidation ratio with the aid of the relation that the equilibrium height was represented by a power function of the total volume of solids for a specified pH.

INTRODUCTION

Gravity sedimentation processes are of great importance to many industries, ranging from wastewater treatment to mineral separation. Thus, knowledge of the kinetics of sedimentation of suspended solids in liquid media provides valuable information for the design and operation of settling equipment.

Colloidal particles generally form clusters of particles with enclosed liquid called flocs or aggregates due to the interaction between particles. As flocs settle from concentrated and flocculated suspensions they deposit at the bottom as the sediment layer. This layer may consolidate further under its own weight. Recently, the effects of consolidation-sedimentation of settled particulate beds on batch sedimentation and centrifugation behaviours have been the subject of extensive research[1,2]. Although most studies have been presented with a focus on the numerical approaches to modelling the consolidation-sedimentation behaviour, from a practical point of view there is also a real need for a more simplified analytical solution.

The key objective of this current work is to examine the dependencies of pH, initial concentration, and initial height on the consolidation-sedimentation behaviour of consolidated TiO_2 suspensions. Emphasis is placed on clarifying the consolidation kinetics of suspensions exceeding the critical concentration on the basis of consolidation theory. In addition, the behaviour of consolidation-sedimentations are compared with those of hindered settling for suspensions below critical concentration values.

EXPERIMENTAL

Materials

The particles used in the consolidation-sedimentation experiments were titanium dioxide (Katayama Chemical Industries Co.) of the rutile form. This particle is an ampholytic dioxide, which has an isoelectric point. Aqueous suspensions were prepared by suspending preweighed quantities of the particles in pure water. The particle charge was adjusted by pH control. The suspension pH was adjusted downward by the addition of 0.1 N HCl solution and upward by the addition of 0.1 N NaOH solution. The change of electrolyte concentration brought about by the pH adjustment was negligibly small compared with the critical concentration where coagulation occurs. Deionized water was prepared by an ultrapure water system for laboratory use (Milli-Q SP, Millipore Corp.).

Experimental Apparatus and Technique

Batch gravity sedimentation experiments were conducted using vertical Plexiglass cylinders of 5.0 cm internal diameter. Before an experiment started, the suspension was agitated sufficiently to ensure that the contents were well mixed, and then it was gradually poured into the graduated settling cylinder. The sedi-

Article III

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Empirical modelling of cake washing in a pressure filter.

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Empirical modelling of cake washing in a pressure filter.

The focus of this article is on empirical modelling of filter cake washing. The filtration experiments introduced in this paper were conducted by using a pilot-scale (0.1 m^2) filter press according to the basic principles of factorial designs. Five different variables of the filtration, pressing, cake washing and air drying stages were considered in the tests and the examined product characteristics were the overall capacity of the filter and the purity of the cake. The results obtained from the tests were used for creating different kinds of regression models for explaining the influence of the studied variables on the success of the cake washing process. The goal of the modelling strategy for the cake washing was to determine the simplest empirical models and compare these with theoretical equations complemented with linear terms. It was found that the empirical equation could model the results more accurately than the theory-based equations could.

Keywords: cake filtration; vertical filter press; cake washing; empirical modelling; experimental design

Introduction

Fundamental filtration research focuses on understanding cake formation and cake washing phenomena separately, and thus the variables used in filtration experiments may be selected and controlled in such a way that the basic principles governing the phenomena can be revealed. Filter manufacturers use test filtrations as a tool for providing information to their customers and for serving their own sizing and sales purposes, which is why the number of available variables used by manufacturers is larger than the number of available variables used in fundamental filtration research. The test filtrations can be divided into preliminary-, sizing- and pilot-scale tests. In some cases, the preliminary tests already provide enough information for sizing purposes, whereas pilot-scale tests are used for finding the optimum operational parameters for the current application.

The overall process of cake filtration can be divided into sub processes such as cake formation, washing, cake compression, and deliquoring. Depending on the filter type used and the requirements needed, the cake formation is followed by the aforementioned sub processes, but the order and number of the stages can be arbitrary. Due to the vast choice of different filtration apparatus, process conditions, and possible filtration cycles, the filter cake washing process has proved to be a challenging task to model comprehensively. The process

step preceding the cake washing affects the washing results by influencing, among other things, the cake saturation level, porosity and specific resistance. Even though the washing is merely one step in the whole filtration procedure, it can consume up to 80% of the filtration area and time, depending on the application (1). For example, in a vacuum belt filter, in order to achieve the same throughput, if a cake is washed, the filtration area needed is five times larger than the space requirements for an unwashed cake.

Collecting appropriate measurement data on the cake washing is a prerequisite for successful modelling. Basically, the washing measurement consists of measuring the solute concentration as a function of wash liquid consumption. Solute refers to either the impurity or the product that is in the liquid phase. This might present an oversimplified picture of the procedure, especially when there are conditions and variables that should be kept constant. According to Svarovsky (2), the conditions and variables known to affect the washing curve are as follows:

(1) Flow rate of wash liquid through the cake.
(2) Mother liquor and wash liquid properties.
(3) Solute to solvent diffusivity.
(4) Cake properties such as porosity, structure, initial saturation, homogeneity, and thickness.
(5) Washing inefficiencies such as cake cracking, channelling, bypassing, and wash liquid maldistribution.

The measurement data acquired from the washing process are usually averaged values of concentrations in the wash filtrate. To obtain actual data on the local concentration and dispersion coefficient values requires extraordinary measurement techniques, such as the ones presented in the article by Lindau et al. (3)

Regardless of the selected washing method or filtration type, i.e. vacuum or pressure filtration, the cake washing results are usually described by a washing curve. The washing curve normally has the dimensionless solute concentration of the wash filtrate plotted against the wash ratio, as presented in Figure 1a. There are, of course, other ways to present the data, but most of these are tied to the solute concentration in the wash filtrate being either retained or removed. When inspecting the figures showing the washing data, one should also take a close look at the definition of the wash ratio used in the figures. This is essential because the wash ratio can be expressed in different ways.

The wash ratio is the volume of wash liquid used divided by the volume of filtrate retained in the cake at the start of the washing. Sometimes the wash ratio is interpreted to be the volume of wash liquid divided by the void volume of the cake (4). The latter interpretation is by definition correct if the cake is fully saturated before the start of the washing. It should be noted that the curve in Figure 1a represents the initially fully saturated cake, and the curve in Figure 1b represents the retained solute concentration in the same cake. If the filter cake has been partially dewatered prior to the washing, the wash curve changes in such a way that the plug-flow plateau diminishes. In some industrial reports, the wash ratio has been replaced by the wash liquid volume divided by the mass of dry solids (5). This type of wash ratio is used for practical reasons when the interest is in process economics and in the changes of process conditions. Also, the wash liquid volume divided by the cake volume has been a ratio used for visualising the wash curve (6).

The cake washing models and theories in the literature (7-13) focus on the solute concentration in the wash filtrate. Possibly the most used washing model is the dispersion model and its modifications for different washing regimes. The dispersion model for the case where the cake is fully saturated and sorption of the solute onto the solid matter is negligible can be described as follows:

$$\frac{c-c_w}{c_0-c_w} = 1 - \tfrac{1}{2}\left\{erf\left[\frac{1-W_R}{2\sqrt{W_R}}\sqrt{D_n}\right] + exp(D_n)erf\left[\frac{1+W_R}{2\sqrt{W_R}}\sqrt{D_n}\right]\right\}, \quad (1)$$

where c is the concentration of the solute in the filtrate, c_w is the concentration of the solute in the wash liquid, c_0 is the concentration of the solute in the liquid in cake voids prior to washing, D_n is the dispersion number, and W_R is the wash ratio.

The definition of the dispersion number D_n is

$$D_n = \frac{uL}{D_L} = Re\ Sc\ \frac{L}{d}\frac{D}{D_L} = \frac{\rho u d}{\mu}\frac{\mu}{\rho D}, \quad (2)$$

where D is the molecular diffusivity of the solute, D_L is the axial dispersion coefficient, d is the particle diameter, L is the cake thickness, u is the superficial fluid velocity, μ is the viscosity of the filtrate, and ρ is the density of the filtrate.

The wash ratio W_R is defined as

$$W_R = \frac{V_w}{V_{f0}} = \frac{ut}{S\varepsilon L}, \quad (3)$$

where S is the cake saturation and t is the washing time, V_w is the volume of wash liquid used, V_{f0} is the initial volume of filtrate retained in the cake, and ε denotes cake porosity.

According to Wakeman and Tarleton (14), this model can be used in the predictive sense if the properties of the cake and liquid are known. The dispersion model has been further developed for cases in which the diffusion of solute takes place in micro-porous particles (8). The dispersion model and its derivatives are somewhat problematic for use outside the laboratory. This is mainly due to problems in estimating the axial dispersion parameter and in obtaining the correct value for the molecular diffusivity of the solute.

The exponential decay model by Rhodes (15) is a simple, elegant model which describes the solute concentration in the cake (as opposed to the dispersion model, which describes the solute concentration in the filtrate).

$$c_{Rt} = c_{R0}\, e^{-\frac{kut}{L}}, \quad (4)$$

where c_{Rt} and c_{R0} are the solute concentrations in the cake at time t and in the beginning of the cake washing process, respectively, and k is an experimentally defined parameter. This

model, with a slight modification, has been utilised by Marecek (16) and Salmela (17, 18). They replaced the exponential term with the wash ratio and thus incorporating the saturation and porosity into the model unlike in the equation (4). This modified exponential decay function is as follows:

$$c_{Rt} = c_{R0}\, e^{-k\, W_R} \tag{5}$$

The exponential decay equation has been used successfully to model the removal of ferrous sulphate from hydrated titanium dioxide (16) and the removal of sodium chloride from starches (18). The exponential decay model is based on the assumption that the solute concentration of the filtrate is in equilibrium with the solute concentration in the filter cake so that the solute concentration in the wash filtrate is directly proportional to the solute concentration in the cake.

Despite the availability of advanced cake filtration theories, sometimes a simple and robust empirical model is more desirable. Simple empirical models describe effectively how the variables affect the response and what the magnitude of the effect of each variable is within the selected variable range. The empirical models used in the test filtrations are utilised to describe the filter performance with a slurry that is not necessarily completely characterised. Therefore, the uncertainties with regard to the slurry composition may render the theoretical equations unusable. The time available for performing the test filtrations is also an important potential constraint to bear in mind. The test filtrations are tied to schedules that usually do not allow rigorous tests based on scientific theory. The empirical equations should be regarded as disposable models which are valid only for the case at hand, and the model must be reworked every time the slurry, the filter, or the variables are altered.

Experimental

The experiments performed in this study to collect the data for modelling were conducted by mimicking the conditions used in industry. The cake washing was monitored by taking

samples of the filtered cakes that were formed. This was seen as a more practical approach because the number of single filtration experiments was fairly high, and taking filtrate samples and analysing them would have been too laborious. Furthermore, the final purity of the cake and the effect of the selected variables on the cake purity were the primary points of interest; not the exact modelling of the washing phenomena. The cake purity was evaluated by using the conductivity of supernatant liquid of reslurried cake samples. Although the conductivity is not a direct measure of the impurity concentration, we used it because it has been directly proportional to the concentration of chloride within the concentration range used in the experiments and with the starch-water slurry system (17).

The material used in the experiments was wheat starch that was obtained as dry powder (Latitec 1000 R, Ciba Specialty Chemicals Oy, Kaipiainen). The suspensions for the filtration tests were prepared by mixing the dry starch powder with water and adding 20,000 ppm of $Cl^-/kg_{dry\ starch}$ in the form of sea salt. Chloride was added into the test slurries to increase the conductivity of the test suspensions and therefore act as a tracer, enabling the washing efficiency to be determined after the tests. The solid concentration of the test suspensions was 35.5 ± 1.0 w-% and the density of those was 1147 ± 3 kg/m^3. The conductivity of the suspensions during the tests was 13.9 ± 0.4 mS/cm, and all tests were carried out at 25.0 ± 0.2 °C. The size distributions of the starch particles from all batches were measured with a Coulter LS 13320 laser diffraction analyzer. A sample particle size distribution is shown in Figure 2. Significant differences between the size distributions from different batches were not observed, which implies that the properties of the test suspensions remained fairly constant throughout the test series.

The filtration and cake washing tests were performed with a laboratory-scale pressure filter (Larox PF-0.1) shown in Figure 3, in which the filtration area is approximately 0.1 m^2. The height of the filtration chamber in all the tests carried out in this study was 60 mm. The

test filter was equipped with data collection software that recorded the cumulative quantity of filtrate produced, the feed line pressure, the pressing line pressure, and the volumetric air flow rate once per second.

Experimental design

The experimental design was a two-level full factorial design for five variables, including nine centre points, four experiments with no washing and no drying, one overlong washing, and two replicates. The overall number of experiments was 48. The two-level full factorial design for five variables consists of 32 experiments. The basic design was extended by adding nine centre points. The basic two-level experimental design for five variables and nine centre points is schematically presented in Figure 4. The centre points in the factorial design indicate at the modelling stage whether it is valid to assume that the variables have a linear effect on the response. The four experiments with no washing served as the baseline for cake purification, and the conductivity results were scaled with these results into the range [0, 1]. The experimental design was randomised in order to minimise the time-dependent variations on the results.

Variable selection imitates the variable set that is typical for test engineer making a test case either for sales purposes or gathering additional data for sizing. Due to this mimicry the variables represent all of the aspects in the filtration cycle typical for pressure filtration of starch.

The variables selected to be used in the experiments are pumping time, washing pressure, washing time, pressing time, and drying time. The variables and constant values used in the experimental design are presented in Table I.

The filtration cycle was started by pumping the test suspension into the filtration chamber at a constant pressure (4.0 bar in these tests). This resulted in a constant pressure filtration period that continued for a predefined time segment (20 – 60 seconds), after which

the slurry feed line was closed and the washing stage was initiated. This was done by introducing wash water from a pressurized vessel into the filtration chamber. The washing pressure varied from 4.0 to 8.0 bar, and the duration of the washing was 30 to 120 seconds. Once the cake washing was completed, the pressing of the cake was started by allowing the pressurized water to flow above the rubber diaphragm that was located on the top of the filtration chamber. The pressing pressure applied here was kept constant at 12 bar, and the duration of the pressing stage varied between 60 and 180 seconds. The final stage of the filtration cycle was the dewatering of the cake by air drying. This was performed by feeding air into the filtration chamber at constant pressure (5.0 bar). The duration of the air drying stage was between 120 and 300 seconds. After the air drying, the filtration chamber was opened and the cake was discharged. The cake was weighed, and samples were taken from the cake in order to determine moisture content as well as the conductivities used to estimate the efficiency of the washing stage. The thickness of the cake was also measured.

The purity of the washed cakes was estimated by measuring the conductivities of the cake samples that were reslurried with de-ionized water. Although this technique cannot be utilised for measuring the absolute amount of impurities in the cakes, it can be utilised for comparing the purity of different filter cakes.

A common way of conducting cake washing in an automatic vertical pressure filter includes a diaphragm compression stage occurring between the slurry pumping and the washing stages. In these experiments, however, the wash water feed was introduced immediately after the pumping. This means that some slurry remained on top of the filter cake when the wash water was fed into the filter chamber; thus, the overall washing consisted of dilution, displacement, and mixing and mass transfer washing mechanisms. This type of washing is used in the starch industry, where pressure filters are employed. Furthermore, Salmela (17) has demonstrated that compression before washing slows down the washing

process and that the washing efficiency remains unchanged when filtering native wheat starch. It should be noted that the theory-based filter cake washing models require porosity values that describe the situation in the filter cake right after the washing stage, and that within our experiments, these values were calculated only after the filtration cycle had been completed. This means that no true value for the cake porosity could be used, because the filtration cycle incorporated the compression after the washing stage, which affects the cake porosity.

The precision of the experiments were estimated using standard procedure of error estimation (19). The cause and effect diagrams for recognising the error sources were created for both the capacity measurements and the dimensionless conductivity shown on Figure 5. The error sources for capacity were created in measuring masses of the cakes discharged from the filter, the wet and dry cake samples used for defining the moisture content, and defining the total time used for filtration cycle. The filtration area was not assessed because it remained constant throughout the filtration and the value used in calculations is based on the manufacturer information. The error in dimensionless conductivity measurement consists of errors in moisture content, water mass used in reslurrying the samples, and the relative error of the conductivity meter. Quantification of the uncertainty components in capacity and dimensionless conductivity measurements are shown in Table II.

The uncertainty of the capacity measurement was calculated as

$$\begin{aligned} Capacity\ u/x &= \sqrt{(u/x)_{Cm}^2 + (u/x)_{Cw}^2 + (u/x)_{Cd}^2 + (u/x)_t^2} \\ &= \sqrt{0.000025^2 + 0.000036^2 + 0.000055^2 + 0.0015^2} \\ &= 0.0015 \end{aligned} \quad (6)$$

where subscripts Cm, Cw, Cd, and t are cake mass, cake wet sample, cake dry sample, and time respectively.

Similarly the uncertainty of the dimensionless conductivity was calculated as

$$\begin{aligned} \textit{Conductivity } u/x &= \sqrt{(u/x)_{Cw}^2 + (u/x)_{Cd}^2 + (u/x)_{Cc}^2 + (u/x)_{Mw}^2 + 3(u/x)_{Co}^2} \\ &= \sqrt{0.000036^2 + 0.000055^2 + 0.00025^2 + 0.000028^2 + 3\times 0.005^2} \\ &= 0.009 \end{aligned} \quad (7)$$

where subscripts Cc, Mw, and Co are cake conductivity sample, reslurry water, and conductivity respectively. The relative uncertainties are less than 1% for both of the measured responses.

Results and discussion

The experimental design was also used for modelling other responses such as the overall capacity of the filter. The measured values of the filter overall capacity and dimensionless conductivities of the cake samples are shown in Figure 6. The experiments were conducted in random order, and those numbered from 42 to 45 are the experiments with the unwashed cakes. The retained impurity content in the filter cake as a function of the wash ratio is known to have a distinct shape that resembles an exponential curve (15). The linear model was applied at first to the variables used in the experimental design to see how well the linear approximation explains the response behaviour within the selected variable range. All of the data from the 48 experiments were used in the modelling. The strategy was to find a simple model function explaining the response variation, without the need to apply parameters that are difficult to evaluate, e.g. the molecular diffusivity of the solute. In this particular case the slurry consistency was known, but in general, the slurries in the industrial environment may contain additional chemicals, such as surfactants, which may affect the real diffusivity of the solute. The experiments were done by mimicking industrial practice where the cake purity data is obtained by taking cake samples and not by recording the solute concentration in the filtrate. The pressure filter used in experiments resembles production filters in the sense that some of the typical filtration data used in theory modeling is impossible to obtain. As an

example the cake thickness and porosity values right after the pumping time cannot be accessed in between the filtration and cake washing stages.

The linear model is the simplest one to calculate. It was known that the linear assumption works sufficiently well with responses such as the cake moisture content (20) and the overall capacity of the filter. Model 1 is linear and is described in equation 8:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_5 \,, \quad (8)$$

where response *y* is the scaled retained impurity concentration in the cake, β represents the parameter values, *x* represents variables, and subscripts ranging from 0 to 5 are the constant term, pumping time, washing pressure, washing time, pressing time, and drying time, respectively. The measured vs. modelled plot shows the quality of the fit in that if the model describes the measured response perfectly, all of the data points should lie on the diagonal line. The deviation from the diagonal shows whether the model over- or underestimates the response. The measured and modelled response values for the linear model are shown in Figure 7. The correlation coefficient value (R^2 = 71.2%) is not good, especially when one takes into account the structured trend of the data points.

The theoretical models related to filter cake washing show strong nonlinear behaviour. Because the measured response represents the retained solute concentration in the filter cake, the exponential decay model was selected as a basis for the model function. The second modelling was with the exponential decay term. In Model 2, the washing time is introduced into the exponential term, and the rest of the variables are kept linear.

$$y = \beta_1 x_1 + \beta_2 x_2 + e^{-\beta_3 x_3} + \beta_4 x_4 + \beta_5 x_5 \quad (9)$$

This model shows significant improvement both in terms of the correlation coefficient value (R^2 = 92.9%) and of the data point structure, as shown in Figure 8.

Model 3 includes both the washing pressure and the washing time in the exponential term, and the rest of the variables have linear effects.

$$y = \beta_1 x_1 + e^{-\beta_2 x_2 x_3} + \beta_3 x_4 + \beta_4 x_5 \quad (10)$$

The exponential term in this equation could be regarded as closely related to the exponential decay model as proposed by Rhodes (15). The washing pressure is included in the exponential term because it has a direct effect on the wash water flux through the filter cake. The correlation coefficient (R^2 = 97.9%) is even better when compared to the previous model, as are the data point locations. The soundness of the fit can be seen in Figure 9. The model function 3 is in actuality simpler in comparison to the previous models. This model only has four parameter values to be estimated, whereas the linear Model 1 has six parameters, and Model 2 has five parameters. The rule of thumb is that if a simpler model works as well as a more complicated model, then the simpler model should be selected.

Model 4 is an exponential decay model with linear terms, as in the previous model. However, in this case the pumping time is inserted into the exponential term, because the pumping time is directly proportional to the cake thickness and, therefore, the pumping time is located as a divisor into the exponential term similarly to how the cake thickness is expressed in Equation 3.

$$y = e^{-\beta_1 \frac{x_2 x_3}{x_1}} + \beta_2 x_4 + \beta_3 x_5 \quad (11)$$

The modelled vs. measured plot in Figure 10 demonstrates that this is not as good as Model 3. The correlation coefficient value drops down to 89.3%, and some of the experimental points start to spread out from the diagonal.

Model 5 is an exponential decay model with linear terms, like in the previous model, but in this case the washing pressure and time are replaced with the wash ratio. The wash ratio was calculated afterwards from the filtration data. The model function is as follows:

$$y = \beta_1 x_1 + e^{-\beta_2 W_R} + \beta_3 x_4 + \beta_4 x_5 \quad (12)$$

Note that this model has the washing time within the wash ratio term, but the washing pressure is not directly included. The correlation coefficient for this model is good (R^2 = 95.4%) but not as good as Model 3 (Equation (10)). The modelled vs. measured plot is shown in Figure 11. The washing pressure has an effect on the wash water flow, but so does the

pumping time. The effect of the washing pressure on the wash water flow is direct: the higher the pressure, the greater the flow. On the other hand, a longer pumping time increases the cake thickness, and thus the cake permeability becomes smaller. Within the experimental setup, the highest wash ratio was 7.695 on a pore volume basis, and the lowest wash ratio was 0.398. The high wash ratio value was obtained with a low pumping time, high washing pressure, and high washing time.

Model 6 was tested for the same reasons as the previous model, but this time the pumping time is also discarded in the modelling phase. In this model, the wash ratio is used to replace the pumping time, the washing pressure and the washing time. Basically, this is valid because one should not use variables that are dependent on each other, and the previously mentioned variables do have an effect on the wash ratio. The problem in using the wash ratio as a variable stems from the fact that the wash ratio cannot be controlled in a precise manner. The wash ratio can be used in the modelling stage, but the interpretation of the results becomes difficult. The model function is

$$y = e^{-\beta_1 W_R} + \beta_2 x_4 + \beta_4 x_5 \quad (13)$$

The modelling results regarding Model 6 are shown in Figure 12. The correlation coefficient for this model is 94.0%. It should be noted that some of the experimental points begin to segregate into groups. This segregation is similar to the results obtained from Model 4 (Equation (11)) and can also be seen in Figure 10.

The measured dimensionless conductivities and the modelled conductivities as a function of the wash ratio are shown in Figure 13. The model function used in Figure 13 is Model 3, as its correlation coefficient is 97.9%. This clearly shows that Model 3 fits well with the measured values.

Conclusion

In this article, the test filtrations that have been conducted are laboratory-scale experiments. The experimental setup and data represents typical situation met in industrial environment

where the filtrate concentration data is either missing or difficult to obtain, which in turn makes the connection to the more state of the art cake washing models difficult. These experiments were used to gather information on two responses that are typical of solid- liquid separation processes. The measured responses consisted of the overall filter capacity and cake purity. The focus of this article is on cake washing modelling even though the experimental setup was not optimised for this. The aim of the modelling strategy in the cake washing was to determine the simplest empirical models and to compare these with the theoretical equations complemented with linear terms. A summary of the employed model functions and correlation coefficients is shown in Table III and the residual case order plots for model functions are shown in Figure 14.

The disadvantage of using the empirical models is that they are valid only for the variable space that has been used in the experiments, and no further extrapolation should be undertaken beyond that. The advantage of using the empirical models is that the experimentation can be done in a straightforward way, and problems in definition (e.g. the molecular diffusivity of the solute) can be avoided. The empirical models are usually simpler and directly show what the relationship between the variable and the response is. The simple empirical equations are easily introduced to people who may not have comprehensive knowledge of filtration theory.

Models 3 and 5 are related, and these model functions provide the best experimental fit. The relation of these equations to one another lies in the exponential term. Model 3 (Equation (10)) contains the washing pressure x_2 and washing time x_3 in the exponent, and in Model 5 (equation (12)), the exponential term is similar to Equation 5, where the wash ratio is in the exponent. The similarity exists because the wash ratio contains the superficial velocity of the wash liquid in the cake, which can be considered to be directly proportional to the wash liquid pressure x_2 when the cake properties are kept constant. Furthermore, the wash

time x_3 is in the exponent in both equations. An interesting result is that the purely empirical Model 3 (Equation (10)) yields a slightly better correlation coefficient as compared to Model 5 (Equation (12)). This can be explained by the fact that the wash ratio is calculated after the filtration and it is based on the data, which always contain more error sources than the directly measured pressure and time values.

When comparing the modelled vs. measured plots in Figures 10 and 12, obtained with Models 4 and 6, Equations (11) and (13), respectively, the figures show an interesting segregation of some experimental results into groups. Models 4 and 6 are the most closely related. The exponential term in Model 4, Equation (11) contains the following variables: pumping time x_1, washing pressure x_2 and wash time x_3. The reason why the pumping time x_1 has been inserted into the exponential term is that the pumping time is directly proportional to the cake thickness, which is one of the parameters used in the exponential decay model, Equation (4). Model 6, Equation (13), is basically an exponential decay term supplemented with linear terms. The pressing time variable x_4 and the drying time variable x_5 have been included in the model functions, because these variables were included in the experimental design, and even though they may not affect the cake purity much, it is better to model their behaviour than not to consider them at all. When filtration experiments are conducted for sizing and sales purposes, often the variables used are of such combinations that the theoretical filtration aspects are not always observed. Therefore, although purely theoretical equations cannot be used, they should instead be supplemented with the variables used in the experimental design.

Using the wash ratio in the model functions 5 and 6 does show that the exponential decay model yields good estimates of cake purification, but at the same time it shrouds the direct effect of the control variables such as the pumping time, the washing pressure, and the wash time. Wash ratio however is not a controllable variable and it must be emphasised that

Models 5 and 6 have been used as an example on combining the theoretical model with empirical terms. The effect of the variables is clear and easily visualised when using empirical models. The models in which the wash ratio is used as a variable show that the exponential decay model is valid in expressing the cake purity, and it is also shown that the empirical models perform slightly better. This slight difference in model performance can be explained by the fact that the cake is not completely formed when the wash water is introduced into the filtration chamber, and the exponential decay model is designed for cases where the cake thickness is known.

The empirical models supplement the theoretical knowledge of filtration, even if the scope of the test filtration is on the practical aspects and not on phenomenal modelling. Consequently, the results from the practical filtration tests reveal the relevancy of the underlying filtration theory.

Nomenclature

c	concentration of the solute in the filtrate, kg m^{-3}
c_{Rt}	concentration of the solute retained in cake at time instant t
c_{R0}	concentration of the solute retained in cake prior the washing
c_w	concentration of the solute in the wash liquid, kg m^{-3}
c_0	concentration of the solute in the liquid in cake voids prior to washing, kg m^{-3}
D	molecular diffusivity of the solute, m^2 s^{-1}
D_n	dispersion number, -
D_L	axial dispersion coefficient, m^2 s^{-1}
d	particle diameter, m
k	experimentally determined parameter, -
L	cake thickness, m
Re	Reynolds number, -
S	cake saturation, -
Sc	Schmidt number, -
t	time, s
u	superficial fluid velocity, m s^{-1}
V_{f0}	initial volume of filtrate retained in cake, m^3
V_w	volume of wash liquid used, m^3
W_R	wash ratio, the amount of wash liquid passed through a cake per unit amount of liquid retained in the cake prior to washing, -

ε cake porosity, -
μ viscosity, Pa s
ρ density, kg m^{-3}

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Table I The variables and constants used in the filtration experiments.

			Low	High
Variables:	**X_1**	**Pumping time**	**20 s**	**60 s**
	X_2	**Washing pressure**	**4.0 bar**	**8.0 bar**
	X_3	**Washing time**	**30 s**	**120 s**
	X_4	**Pressing time**	**60 s**	**180 s**
	X_5	**Drying time**	**120 s**	**300 s**
Constants:		**Pumping pressure**	**4.0 bar**	**4.0 bar**
		Pressing pressure	**12.0 bar**	**12.0 bar**
		Drying air pressure	**5.0 bar**	**5.0 bar**

Table II Quantification of the uncertainty components faced in capacity and conductivity measurements. Mass and time values are average of the individual measured values.

	Subscript	Value, *x*	Standard uncertainty, *u*	Relative uncertainty, *u/x*
Cake mass	Cm	4061 g	0.1 g	0.000025
Cake Wet sample mass	Cw	274 g	0.01 g	0.000036
Cake Dry sample mass	Cd	181 g	0.01 g	0.000055
Time	t	682 s	1 s	0.0015
Cake cond. sample mass	Cc	40 g	0.01 g	0.00025
Reslurry water mass	Mw	360 g	0.01 g	0.000028
Conductivity	Co	-	-	0.005

Table III The model functions used in the modelling stage and the correlation coefficients of different model functions.

Model	**Function**	**Correlation coefficient R^2 , %**
1	$\boldsymbol{y} = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_5$	**71.2**
2	$\boldsymbol{y} = \beta_1 x_1 + \beta_2 x_2 + e^{-\beta_3 x_3} + \beta_4 x_4 + \beta_5 x_5$	**92.9**
3	$\boldsymbol{y} = \beta_1 x_1 + e^{-\beta_2 x_2 x_3} + \beta_3 x_4 + \beta_4 x_5$	**97.9**
4	$\boldsymbol{y} = e^{-\beta_1 \frac{x_2 x_3}{x_1}} + \beta_2 x_4 + \beta_3 x_5$	**89.3**
5	$\boldsymbol{y} = \beta_1 x_1 + e^{-\beta_2 W_R} + \beta_3 x_4 + \beta_4 x_5$	**95.4**
6	$\boldsymbol{y} = e^{-\beta_1 W_R} + \beta_2 x_4 + \beta_4 x_5$	**94.0**

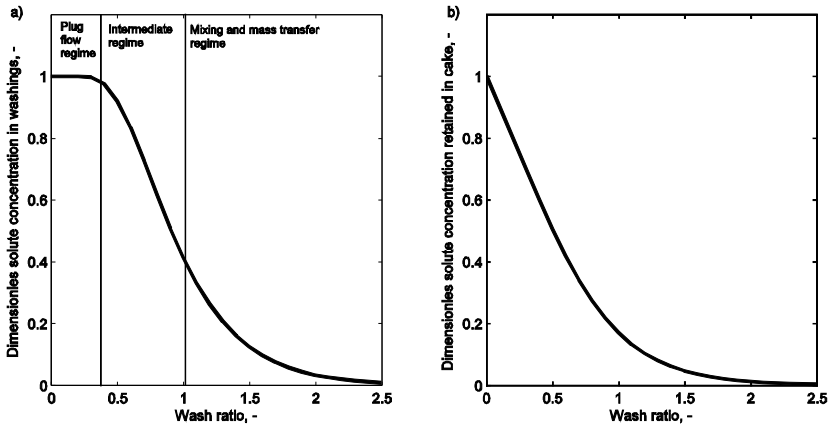

Figure 1. a) A typical wash curve obtained when the solute concentration of the filtrate has been measured. b) The wash curve for the retained solute concentration in the cake.

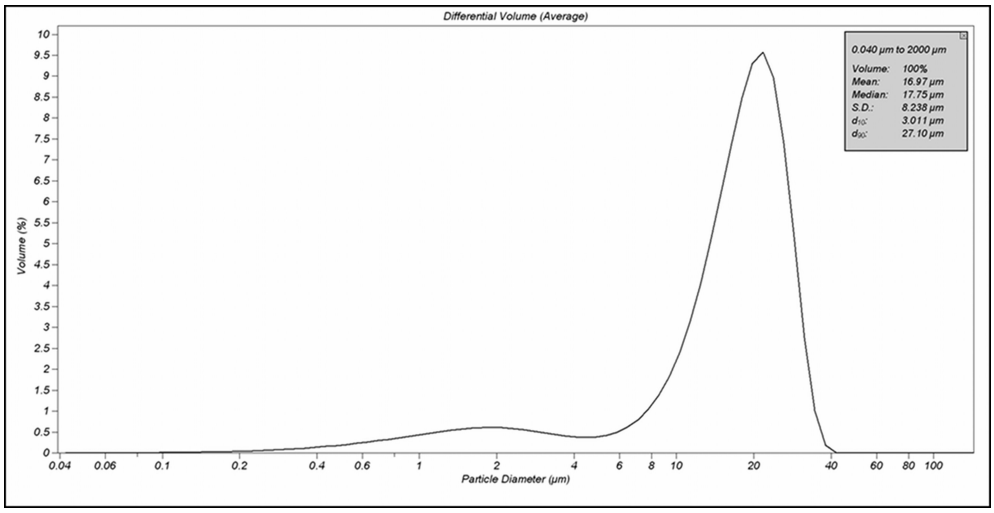

Figure 2. Particle size distribution of native wheat starch used in filtration tests.

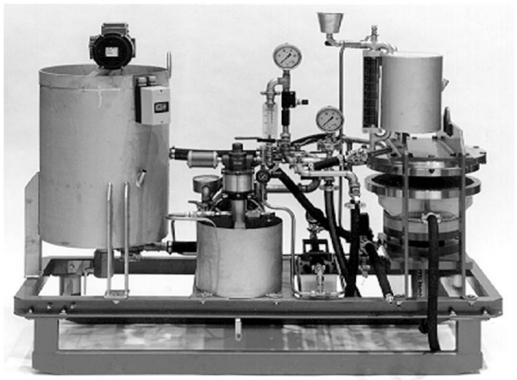

Figure 3. The pressure filter used in the experiments.

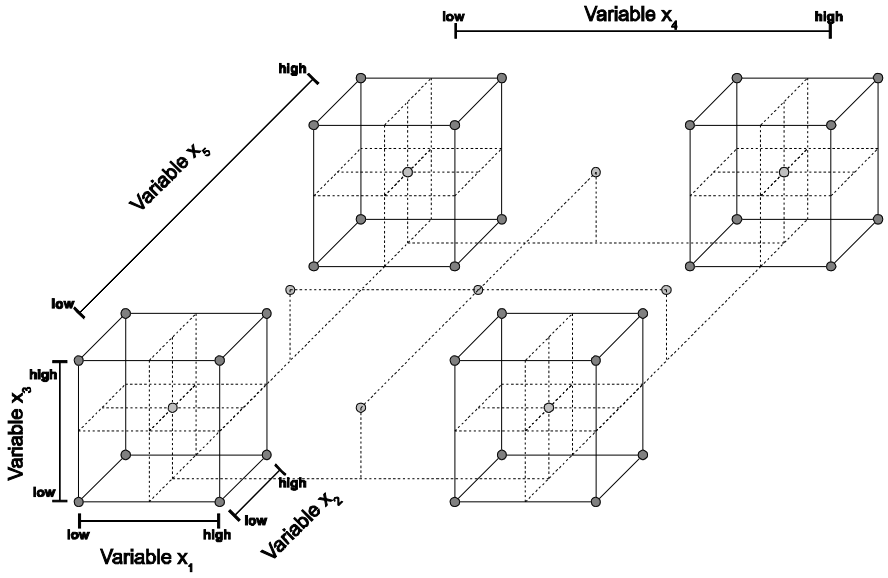

Figure 4. Full factorial experimental design supplemented with nine centre points.

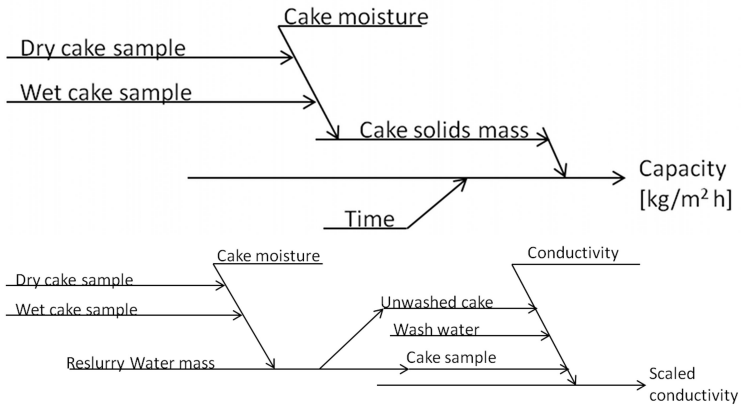

Figure 5. The cause and effect diagrams for both the capacity and the dimensionless conductivity measurements.

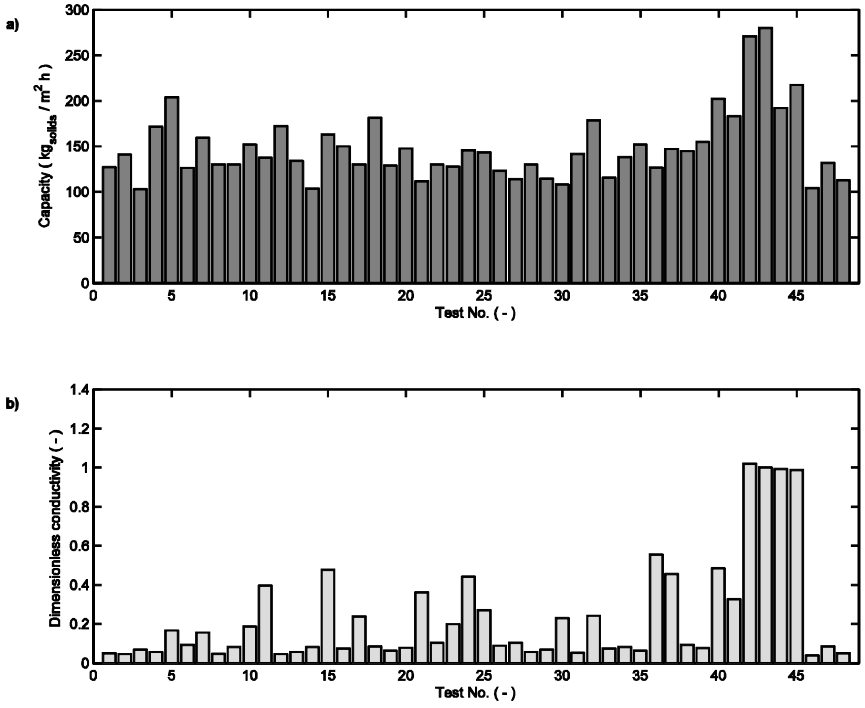

Figure 6. The measured response values of a) the filter overall capacity and b) dimensionless conductivity. The experiments 42-45 were without washing and drying.

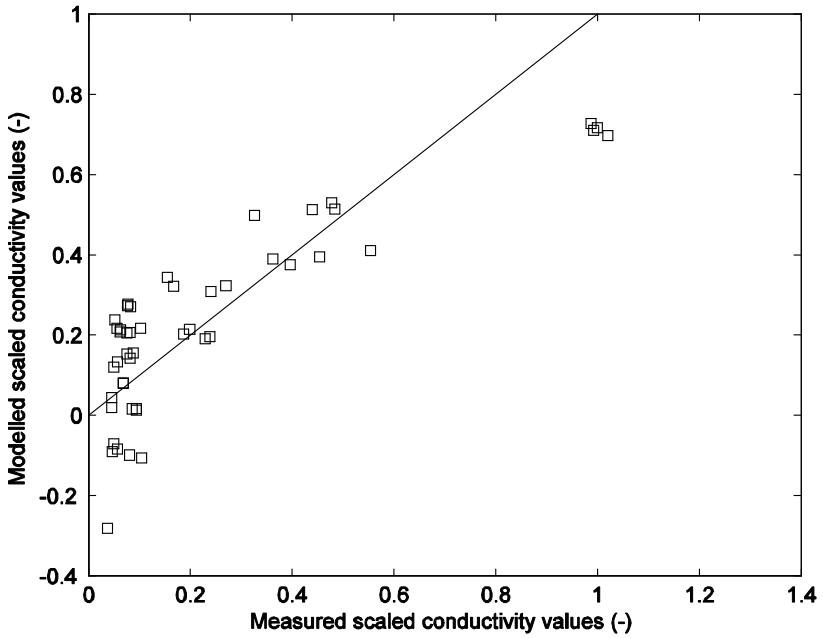

Figure 7. The measured vs. modelled plot of the linear Model 1. The correlation coefficient for this pure linear model was 71.2%.

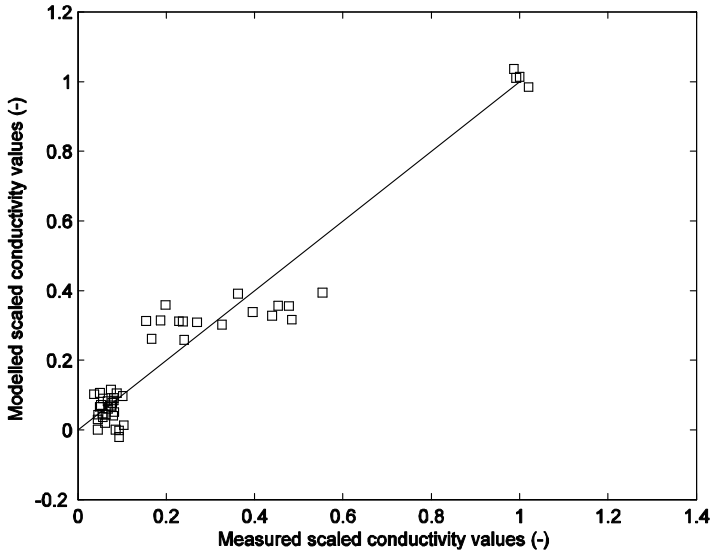

Figure 8. The measured vs. modelled plot of Model 2. The only variable in the exponential term is the wash time. Other variables are modelled to have a linear effect. The correlation coefficient for Model 2 was 92.9%.

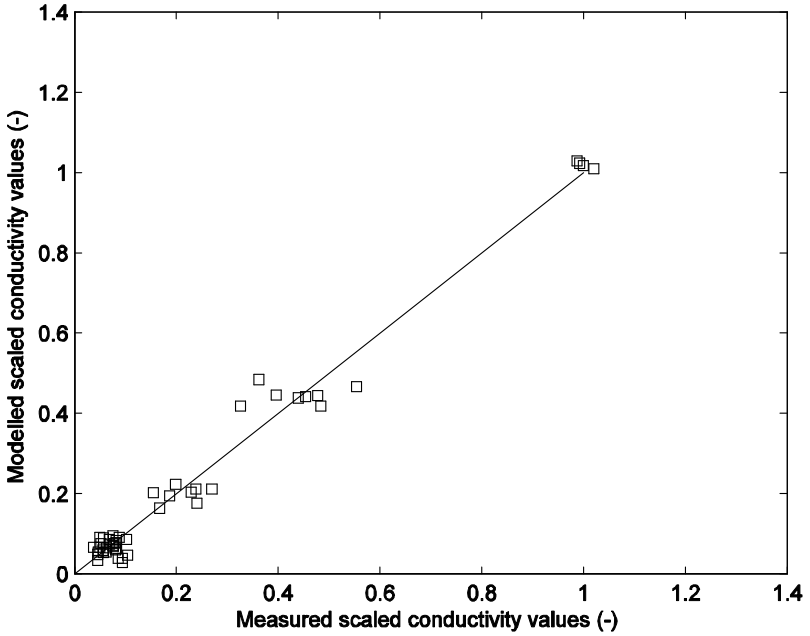

Figure 9. The measured vs. modelled plot of Model 3. The exponential term contains the washing pressure and wash time terms. The pumping time, pressing time, and drying time have a linear effect. The correlation coefficient for Model 3 was 97.9%.

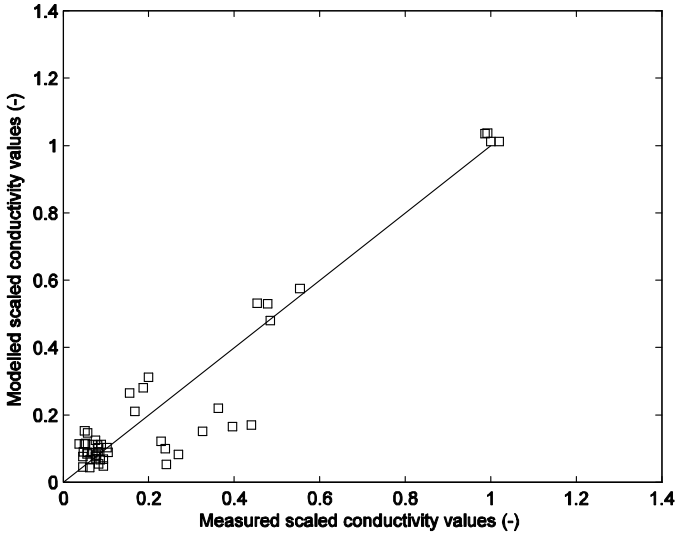

Figure 10. The measured vs. modelled plot of Model 4. The pumping time, washing pressure and wash time are in the exponential term. The correlation coefficient for Model 4 was 89.3%.

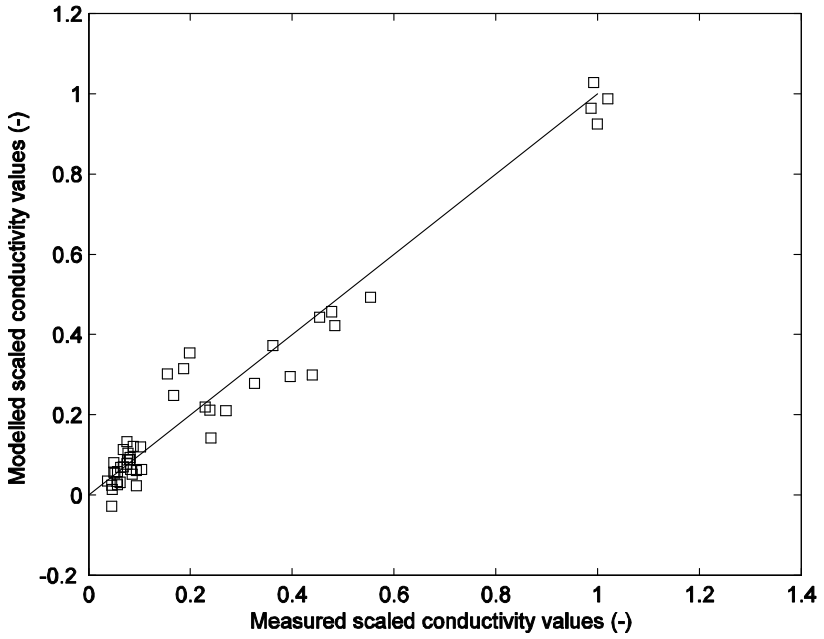

Figure 11. The measured vs. modelled plot of Model 5. The washing pressure and washing time are replaced by the wash ratio. The correlation coefficient for Model 5 was 95.4%.

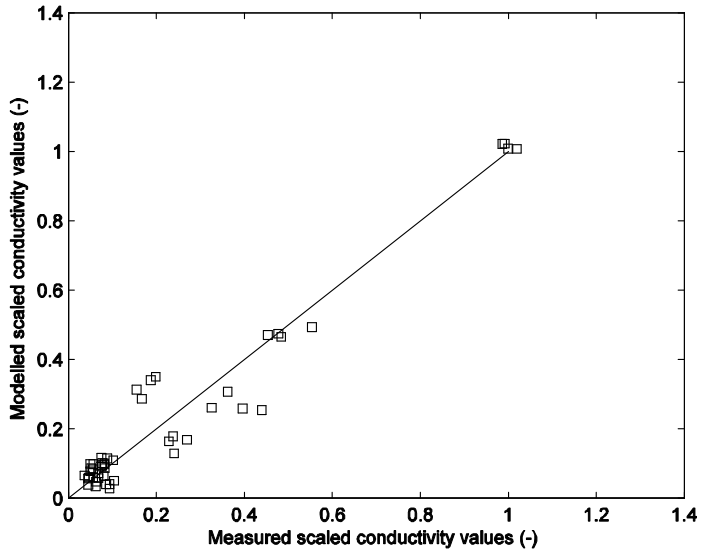

Figure 12. The measured vs. modelled plot of Model 6. The pumping time, washing pressure and wash time are replaced by the wash ratio. The correlation coefficient for Model 6 was 94.0%.

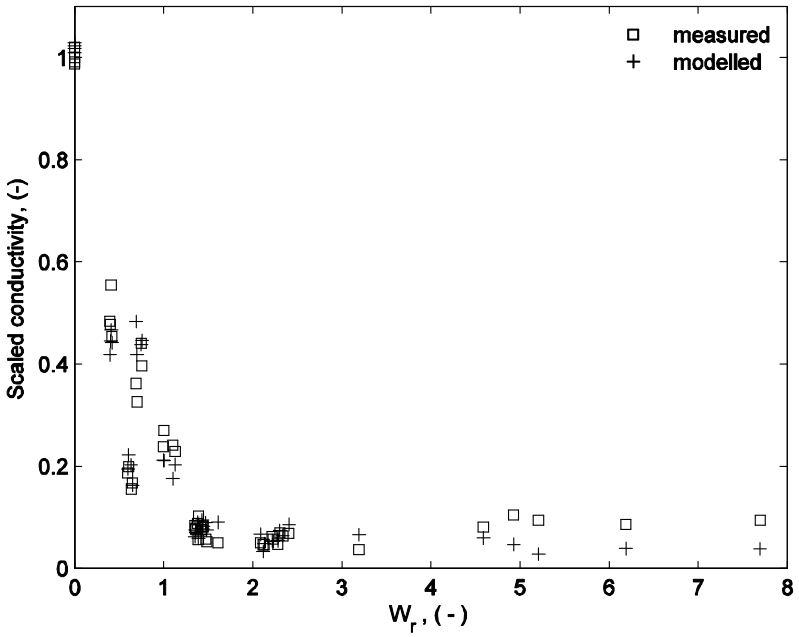

Figure 13. The measured dimensionless conductivities and the modelled conductivities plotted as a function of the wash ratio. The model function used is Model 3, having the correlation coefficient of 97.9%.

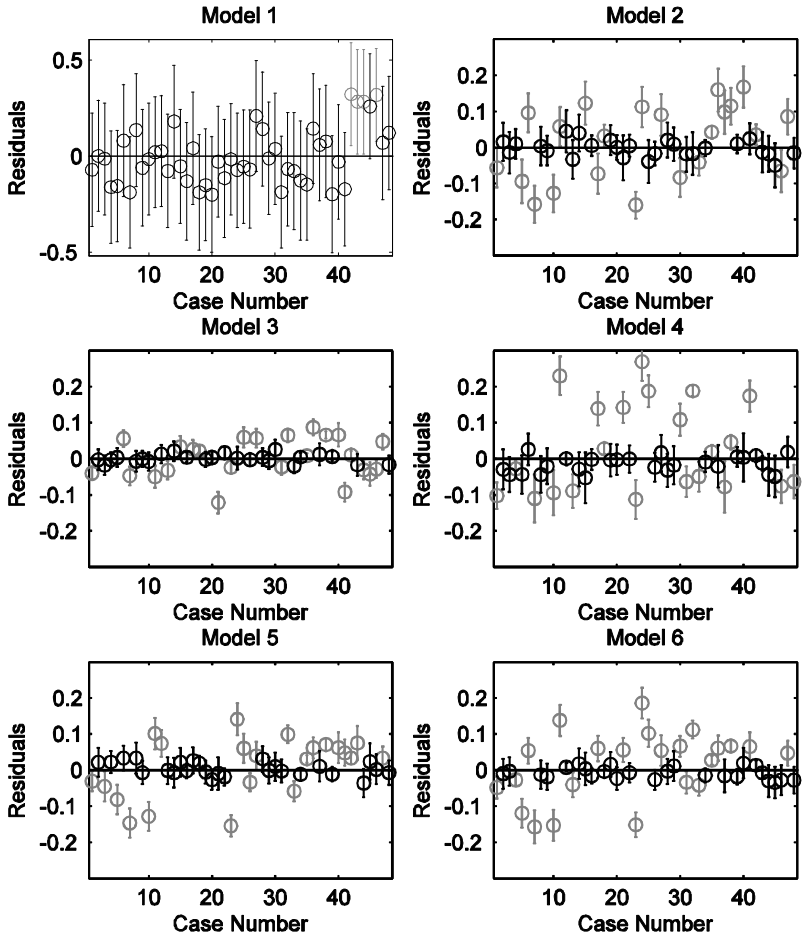

Figure 14. Residual case order plot for each of the models.

Article IV

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SOFTWARE FOR STATISTICAL DESIGN OF EXPERIMENTS AND EMPIRICAL MODELLING OF CAKE FILTRATION

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This paper introduces software developed to improve the efficiency and quality of typical test filtration tasks and to simplify and rationalize the interpretation of test results. The software consists of two different modules. The first module creates experimental designs according to initial information provided by the user. The test designs are created by applying the basic principles of factorial designs in such a way that the variations in the investigated ranges of the desired process variables are taken into account systematically. The utilization of different kinds of factorial designs also means that the amount of experiments needed to achieve the required accuracy can be minimized. The second module of the software analyzes the experimental results by utilizing standard multivariate data analysis techniques. The meaningful information from a given table of experimental data is extracted and used to create regression models that quantify the relationships between the studied process variables. These models can be further applied to predict new values, for visualizing the relationships between the different variables, or for selecting the variable combinations in order to reach the predefined process objectives.

The current software is capable of creating experimental designs for five different kinds of filters, namely the automatic vertical pressure filter, double-sided automatic vertical pressure filter, horizontal membrane filter press, horizontal vacuum belt filter, and the ceramic capillary action vacuum disc filter. The suitability of the applied techniques for each filter type has also been verified by performing a large number of experiments. Some of the experimental results with the automatic vertical pressure filter are presented in this paper to illustrate the procedure developed and also to demonstrate the quality of the final results obtained by the applied methods.

INTRODUCTION

The selection, design, and sizing of filtration equipment for a new application always requires tests to be performed. Theoretical filtration models can be used in some cases for estimating the performance of different filters based on experimental results, but the prediction of filtration characteristics of unknown suspensions on a completely theoretical basis is still unreliable. In addition to this, the interactions between the different stages of the overall filtration cycle, such as cake compression, washing and air drying, are difficult to predict and therefore need to be determined experimentally[1].

The pilot scale experiments performed in the real process environment are typically very expensive and often require test equipment that is not always readily available. Experiments may also be performed in a test laboratory by using samples collected from the target process. The problem here is that the sample amount is often fairly small and the properties of many samples are influenced by aging during transportation and during the test work. For these reasons it is important to be able to perform the required experiments as quickly as possible with as small an amount of the sample suspension as possible.

Test filtration tasks involving many variables can be labour intensive and time consuming if such conventional experimentation methods such as 'one variable at a time' are used[2]. The number of tests may become excessive when the influence of several variables needs to be investigated. The operation cycle of a filter consists of multiple stages which all influence the cake properties and therefore also the success of the other stages. Consequently, theoretical optimization of the whole cycle of a filter is difficult[3].

The use of statistical design of experiments and empirical modelling tools can often reduce the amount of experimental work significantly. When applying statistical techniques for designing the experiments, changes in the studied variables are made in a structured way so that possible interactions between the variables can also be taken into account. These techniques can also be applied to ensure that the final data contains enough information concerning the investigated phenomena and that the structure of the data table is suitable for further analysis[4].

The utilization of empirical modelling techniques can considerably simplify and rationalize the evaluation of experimental results. The experimental models do not

necessarily require any theoretical source information about the considered phenomena and can be created by using standard multivariate data analysis techniques, such as Multiple Linear Regression (MLR). These techniques aim to extract the meaningful information from a given table of experimental data and use this information to fit a model to the observed data in order to quantify the relationship between the studied process variables. The fitted empirical models can be applied to predict new values, to visualize the relationships between the different variables, or to select the variable combinations in order to reach the predefined process objectives[5].

It is therefore possible to replace many of the costly and time consuming measurements with the utilization of these kinds of empirical models. When the empirical models are made according to the data obtained from experiments designed using the statistical techniques, it is normally possible to obtain models which are stable and robust, and the significance of the individual effects as well as their relevance on the whole model can be easily evaluated.

The objective of the project introduced in this paper is to create a user friendly software package for reducing the amount of experimental work and costs in typical test filtration applications. The primary target audience for the software is testing engineers, but it is also suitable for such research purposes as the simplification of more complicated case studies. The produced software package is easy to use and has been programmed in such a way that it can be used without any prior experience or knowledge about the details of experimental design, multivariate modelling, or optimization methods. The software has been created in the MATLAB® programming environment.

STRUCTURE OF THE SOFTWARE

The LabTop software consists of two different modules. The first module (LTDoE) creates experimental designs for different kinds of test filters according to initial information provided by the user. The second module (LTReaD) analyzes the experimental results by utilizing multivariate data analysis techniques and creates a standard test report that contains the parameters of the models as well as some user defined illustrations.

LTDoE – Module

The LTDoE module is used to create experimental designs for the given filter type. As an output, the LTDoE produces an Excel file containing the created design. In this case, the experimental design involves a set of experiments that are meant to be carried out in the same order in which they are presented in the Excel file. After the experiments have been carried out, the results will be written into the same Excel file where the original design is found. The user may write in as many responses as needed, but most commonly the responses that are of interest are the overall capacity of the filter and the residual moisture content of the cake. The typical sequence when the user creates an experimental design using LTDoE is as follows:

1. Select the filter type
2. Select the appropriate filtration cycle
3. Select the variables used in the experiments and the corresponding variable limits
4. Select the extent of the experimental design
5. Create the experimental design and save it as an Excel file.

As the list states, the first step is to select the type of the filter that will be used in the tests. The current version of the software is capable of creating tailor-made test designs for five different kinds of filters manufactured by Outotec (Filters) Oy. These include the automatic vertical pressure filter (PF), the double-sided automatic vertical pressure filter (DS), the horizontal membrane filter press (MFP), the horizontal vacuum belt filter (Pannevis), and the ceramic capillary action vacuum disc filter (Ceramec). In addition to these, the user can also create experimental designs for virtually any purpose by selecting the 'Experimental' option. Procedures for creating designs for other kinds of test filters will be included into the software in the future. Even though the work has been done with Outotec Larox filters, the same principles are valid for any other manufacturer's filters.

After the filter has been selected, the user needs to define the different stages of the overall filtration cycle. This means that the user informs the software through dialog boxes as to whether the actual filtration stage is followed by one or more cake compressions, air drying, or washing stages. The user also defines the order of these stages. This is done by using letter codes, as can be seen from Figure 1, which presents screenshots of the different steps as an example. Here, a one-sided pressure filter (PF) has been selected to be the test filter, and the user has inputted that the filtration cycle consists of a pumping (i.e. filtration) stage, a mechanical squeezing of the cake and an air drying of the cake.

When the filter cycle has been defined, the software opens a new dialog box containing a list of possible variables for the selected test filtration task. An example of this is also shown in Figure 1. The first three variables on the list are the most typically used slurry variables, i.e. pH, temperature, and solids concentration of the slurry. In addition to these, there are at least two variables available for each stage of the overall filtration cycle. In the case of pressure filters, these variables are typically the duration of the stage

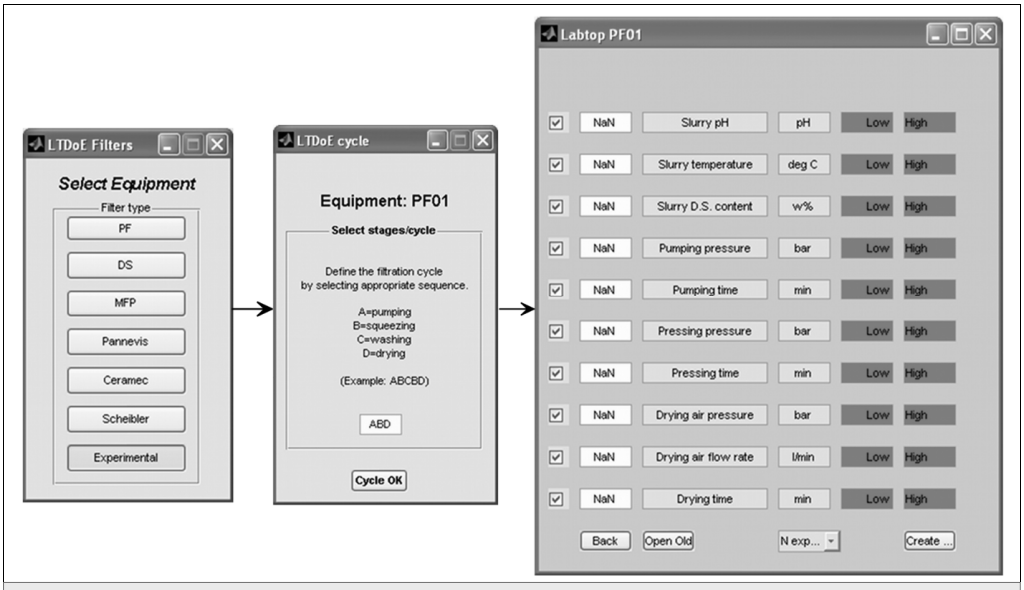

Figure 1: Filter type, filtration cycle, and variable selection dialogs.

and the pressure applied during the stage. The user may, however, freely modify the names and units of these variables. The structure of the variable table from left to right is as follows:

- A checkbox indicates whether the variable will be maintained as constant during the tests or whether the user prefers to use this parameter as one of the variables in the experimental design. If the checkbox is selected, then the current field is considered to be constant.
- The user may write the value of the constant variable in the first edit box, but this is not obligatory. If the background colour is white, then the current field is constant, and the value is written into the experimental design.
- The constant or variable name field can be edited according to user preferences.
- In the constant or variable unit edit box, the units can be edited according to user preferences.
- There are two variable range edit boxes. If the background colour is white, the edit box on the left is reserved for the lower limit and the edit box on the right for the upper limit of the desired range of the variable. A red background colour indicates that the field is constant and the values in these boxes are not in use.

By default, all of the variables are marked initially as constants. Unselecting the checkbox makes the parameter a design variable, which enables the user to provide the limits of the range that will be investigated in the tests.

An example case of creating an experimental design is presented in Figure 2. In this example there are now six variables selected in total and the preferred ranges for each of these variables are displayed in the variable range edit boxes. Before proceeding to the next step, the user must still select the appropriate number of experiments that will be performed during the tests. This can be done by selecting one of the options available in the drop down menu. The highest number on the list always denotes a full two-level factorial design complemented with a certain amount of centre points.

Other options on the list are the fractional two-level designs of different resolutions, starting from the ½-factorial and followed by smaller fractional designs such as the ¼-factorial and so on. In the example case, there are four different designs available and the number of experiments that the user will carry out is 71, 39, 23, or 15. The smallest value presented in the drop down menu is the absolute minimum number of experiments needed for evaluating the interactions of the selected variables. The centre points are added into the basic two-level factorial designs in order to reveal the possible non-linear behaviour of the investigated process during the modelling stage.

By clicking the ‘Create Plan’ button the user will even-

tually arrive at the save file dialog, where the name for the experimental plan Excel file is defined. There is only one possible side step, which is dependent on the variables that the user has selected. This is the slurry variable ranking which enables the user to define a rank order that is used to describe the nature of the slurry variables. For example, in the case presented in Figure 2, there are now two slurry variables selected (slurry pH and slurry temperature).

Now the user is prompted with an additional dialog box which requests the user to select the order of these variables in decreasing order of difficulty. Based on the slurry variable ranking, the experimental design table is divided into blocks that minimize the workload of the experimenter due to the change of slurry characteristics between the experiments. Normally the experimental runs are completely randomised, the blocking in experimental design reduces the randomisation level for slurry variables and the experimental runs are restructured so that the changes needed in slurry composition is minimised according to user preferences. Table 1 shows an example experimental design both with and without blocking of the first variable.

The experimenter can also open previously created experimental designs. The possibility to do this helps the user to create similar types of designs and flexibly modify them without the need to rewrite the variable limits, if those have proved to be good for the case at hand. The Excel file is for storing the experimental design, for writing in the real variable values used in the experiments, and for storing the measured response values. Sheet1 in this file is used only to show the experimental design, and Sheet2 is reserved for writing in the test conditions as they have been realized in the actual experiments. On Sheet2, the user will also write in (input) the results (responses for the models) that have been obtained from the experiments. There are no restrictions for the type, name, or number of the responses.

LTRead – Module

The LTRead module is used for creating models describing the influences of the test variables on the studied responses. The LTRead module reads the input data directly from the Excel file where the results of the experiments have been inputted. Mathematical models are automatically generated for each of the measured responses that the user has entered into the experimental design table. After the modelling and statistical calculations are completed, the user is provided with a graphical interface for creating additional figures for data evaluation and reporting purposes. There is also the possibility of creating an autogenerated report which contains the mathematical models with statistical data and additional figures that the user has created with the LTRead module. The modelled responses and parameters of each model are written into the Excel file as separate sheets.

There are two predefined figures that are always created for each of the modelled responses. These figures are the 'Residual Case Order plot' and 'Modelled vs. Measured plot'. In other words, if there are two responses that have been modelled, then there will be four predefined figures in total. These plots give the user an opportunity to examine the quality of experimental data and the reliability of the models. The Residual Case Order plot may be used for estimating whether there are any clear outliers in the experimental data. The errorbars show the 95% confidence

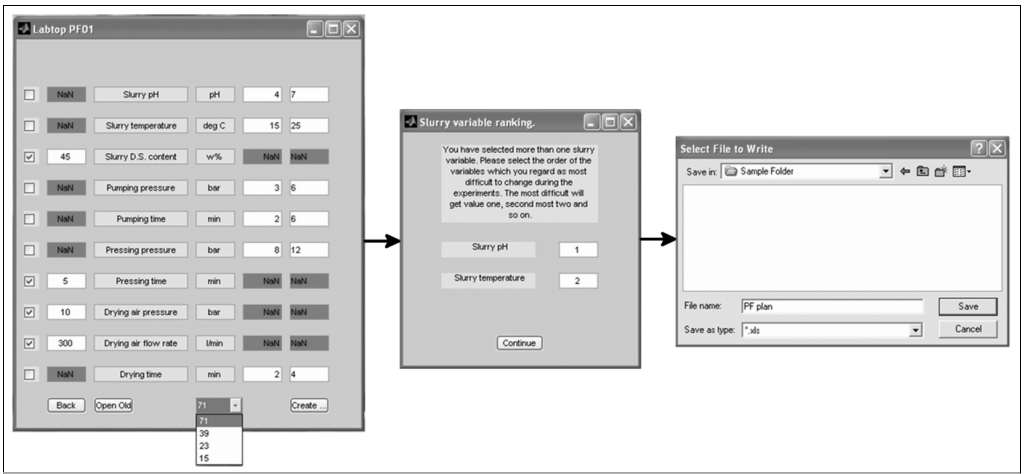

Figure 2: Variable information input, selection of the experimental design, slurry variable ranking, and file save dialog.

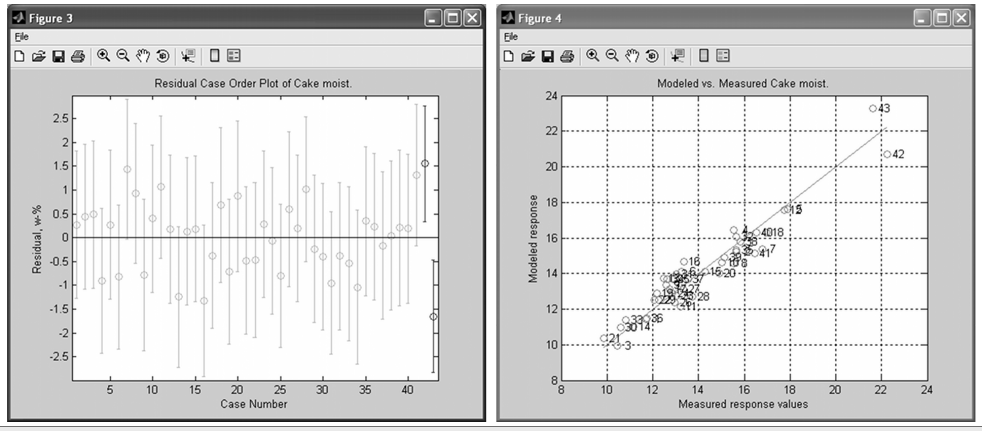

Figure 3: Residual Case Order plot and Modelled vs. Measured plot created for each of the responses.

intervals on the residuals. Similarly, the Modelled vs. Measured plot can be used for evaluating the overall quality of the models and also for detecting whether the built-in assumption for the linearity of the models is valid for the studied process within the measured variable range. Examples of the predefined plots are shown in Figure 3.

After all the models have been created, the user is given a graphical user interface for creating additional figures and for visualizing the relationships between the different variables. The user interface is divided into four different tabs. These four tabs are 2D, 3D, Responses, and Reporting. The user interface is mainly for previewing and modifying the figures that the user is able to create. The user selects the desired variables and responses and clicks the 'Draw Now' button to create a new figure. Later on the user may save these figures into separate .jpg image files. Each of the tabs has a unique task. The 2D tab is for creating ordinary graphs where one response is plotted against one variable. The 3D tab can be used for creating three-dimensional figures where there are two axes for the two variables and one axis for one response. The Responses tab is for creating figures where two responses are plotted against each other, a typical example being a plot where the residual cake moisture is plotted against the achieved overall capacity of the filter. The final Reporting tab is used for saving all the images at once and for creating the autogenerated report file (.doc). An example of the 2D tab of the user interface is shown in Figure 4.

The user may select the variables and responses that are to be displayed in the additional figures from the drop down menus. The preview window shows the axes of choice before the user makes the decision whether this figure is acceptable or not. The additional figures also contain the values of those variables that are included into the models, but have some fixed values in the figures. If there are some specific value combinations that the user wants to employ when drawing the additional figures, it is possible to define those values manually. The selection of the 'non free' values may be done by clicking the 'Slider 2D' button. This opens a new dialog window that can be used for setting the desired values, either by using the slider tool or by writing the values directly into the edit boxes. By default, the software creates the figures using the mean value of the range used in the experiments for each variable. The principle for creating the 3D images is similar to that for creating 2D images. An example 3D plot is shown in Figure 5.

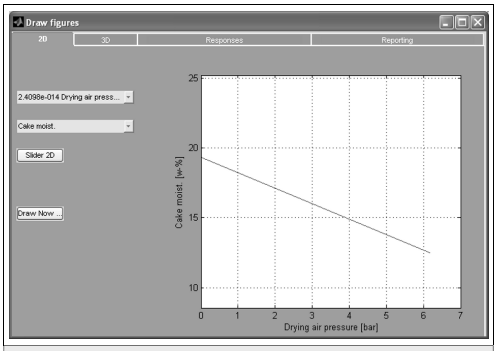

Figure 4: User interface of the LTRead module showing the 2D tab.

CASE STUDY

This case introduces an experimental study performed to determine the influence of five process variables on the performance of the vertical filter press. The tests were performed with a laboratory scale press according to the plans created by following the principles of factorial designs. The capacity of the press and the cake moisture content described the performance of the filter. Results were used to create regression models for both responses, and those were successfully utilized for discovering the most significant variables and for estimating the effects of those on the operation of the filter.

Several designs were created for defining the minimum number of tests required for obtaining satisfactory results. Comparison showed that the amount of tests could be reduced by utilizing the design of experiments and modelling. The number of tests could be reduced from 41 to 8 without losing much accuracy in predicting the influence of variables on the operation of the filter.

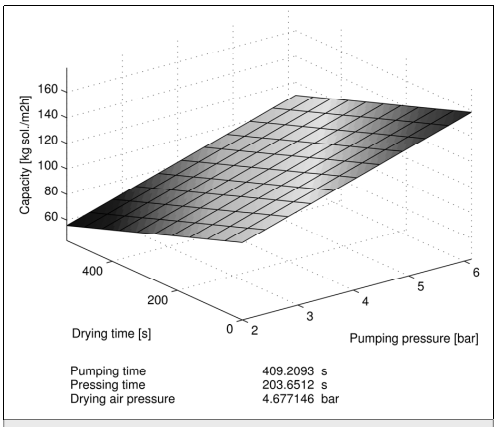

Figure 5: Example 3D plot showing the filter capacity as a function of drying time and pumping pressure, and the variable levels for the remaining variables.

Test Suspension

The material that was used in these tests was ground $CaCO_3$ (OMYACARB 10) and obtained as dry powder. The suspensions for the filtration tests were prepared by mixing the dry $CaCO_3$ powder with water in such a way that the solid concentrations of all test suspensions (8 batches in total) were 39.70±0.53 w% and the densities were 1333±4.5 kg/m^3. All tests were carried out at 25±0.2°C. The size distributions of the $CaCO_3$ particles from all batches were measured with a Coulter LS 13320 laser diffraction analyzer using an optical model created according to the optical properties of $CaCO_3$. The average of the measured size distributions, together with the SEM image of the particles, is presented in Figure 6. Significant differences between the size distributions from different batches were not observed, which implies that the properties of the test suspensions remained fairly constant throughout the tests.

Filtration Tests

The filtration tests were performed with a laboratory scale pressure filter (Larox PF-0.1) where the filtration area was 0.1 m^2 and the height of the filtration chamber was 45 mm in all tests. The test filter, which is shown in Figure 7, was equipped with data collection software that recorded the cumulative amount of filtrate produced, feed line pressure, pressing line pressure, and volumetric air flow rate once per second. The cloth in all tests was a multilayered polypropylene felt manufactured by Tamfelt (S5100-L1K4).

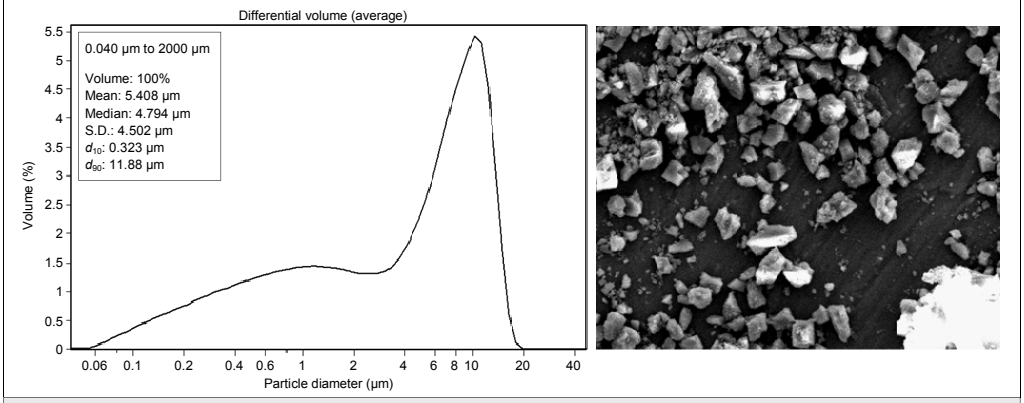

Figure 6: Particle size distribution of the test slurry and SEM image of the particles.

The filtration cycle was started by pumping the test suspension into the filtration chamber using feed pressure that varied between 2.0 and 6.0 bar. This resulted in a constant pressure filtration period that was continued for a predefined time (240-600 s), after which the feed line was closed and the pressing of the cake was started by allowing the pressurized water to flow above the rubber diaphragm located on the top of the filtration chamber. The pressing pressure applied in all tests was 15.0 bar and the duration of the pressing stage lasted between 120 s and 300 s. After the pressing stage was completed, the cakes were further dewatered by air drying. The duration of the air drying stage was between 180 s and 540 s, and the feed pressure of the drying air varied from 4.0 to 6.0 bar. After the air drying, the filtration chamber was opened and the formed cake discharged. The cake was weighed and samples were taken from the cake in order to determine its solids content. The thickness of the cake was also measured.

The examined filtration process consisted of 5 process variables and 1 constant. It was therefore decided that the base design for the tests would be 2^5 full factorial design. This means that each variable was given two levels (high and low), and every possible combination of these variable levels was included in the design. For five variables, this kind of design resulted in 32 tests. In addition to these tests, it was decided that a certain amount of centre points was needed in the design in order to detect the possible non-linearities during the modelling part. A general structure of this kind of 2^5 full factorial design with 9 centre points is illustrated in Figure 8, and the variable notations together with the selected variable levels are summarized in Table 2. The overall amount of tests in this design summed up to 41.

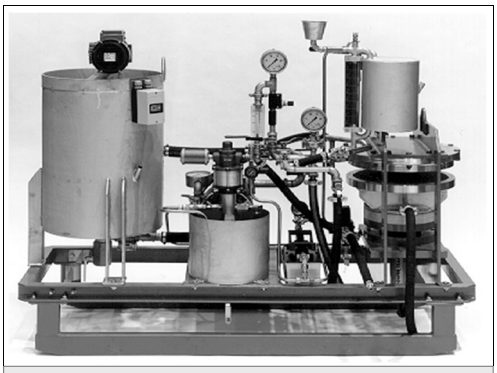

Figure 7: Laboratory scale pressure filter used in the tests.

In addition to the experimental design explained above, two tests were performed without the air drying stage to obtain reference values for estimating the overall influence of the air drying stage on the tested material. Therefore, the total amount of experiments carried out in this study was 43.

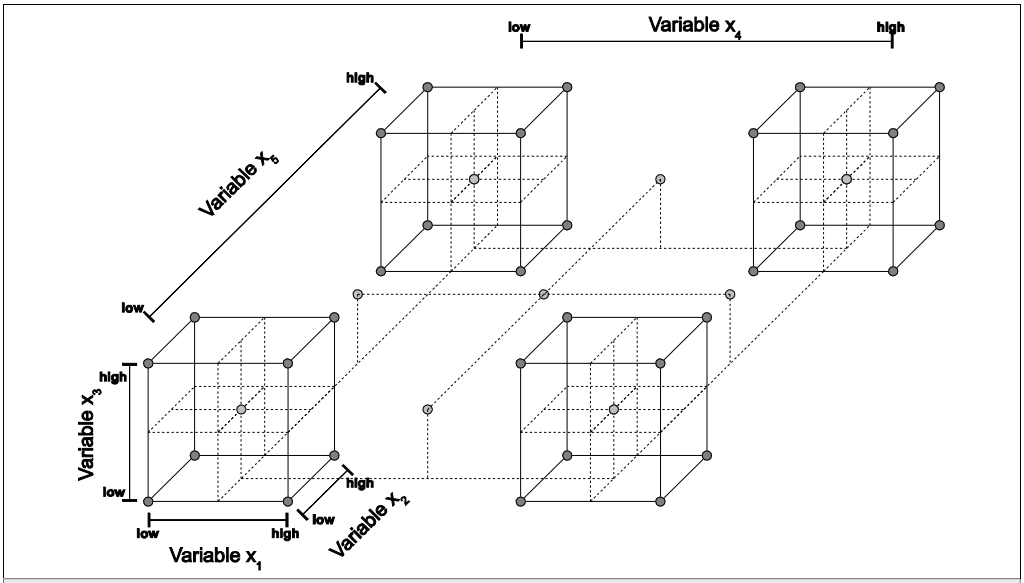

Figure 8: 2^5 full fractional design with 9 centre points.

	Basic randomized design				Blocked design		
	x_1	x_2	x_3		x_1	x_2	x_3
run	w%	bar	s		w%	bar	s
1	45	6	120		35	2	120
2	35	2	120		35	6	120
3	45	6	60		35	6	60
4	35	6	120		35	2	60
5	35	6	60		45	6	120
6	45	2	120		45	6	60
7	45	2	60		45	2	120
8	35	2	60		45	2	60

Table 1: Example of randomized and blocked design showing the reduction of randomization.

Results

The main target of this study is to determine the influence of the five filtration process variables on the moisture content of the filter cakes and on the capacity of the filter. The experimentally determined cake moisture contents and the calculated capacities for the 43 tests are shown in Figures 9 and 10. Although the exact conditions applied in the tests cannot be seen from these figures, the graphs clearly show the variation between the obtained results. It can also be noticed that the experiments have been performed in randomized order since obvious trends do not seem to exist between the results.

As Figure 9 shows, the variations in the cake moisture contents are quite large, ranging from about 9.9 to 22.2 w%. As expected, the highest moisture contents are obtained from those tests that were carried out without the air drying. The effect of the air drying stage on cake moisture content appears to be between 7 and 12 w%, depending on the levels of the other variables. The differences in the calculated capacities are also fairly large as can be observed from Figure

			Low	High
Variables:	x_1	Pumping pressure	2.0 bar	6.0 bar
	x_2	Pumping time	240 s	600 s
	x_3	Pressing time	120 s	300 s
	x_4	Drying air pressure	4.0 bar	6.0 bar
	x_5	Drying time	180 s	540 s
Constant:		Pressing pressure	15.0 bar	15.0 bar

Table 2: Variables and levels for the 2^5 full fractional design.

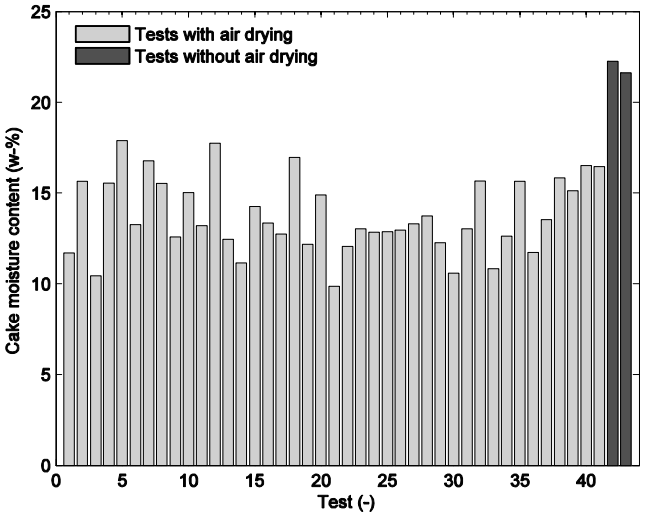

Figure 9: Cake moisture contents for the 43 tests.

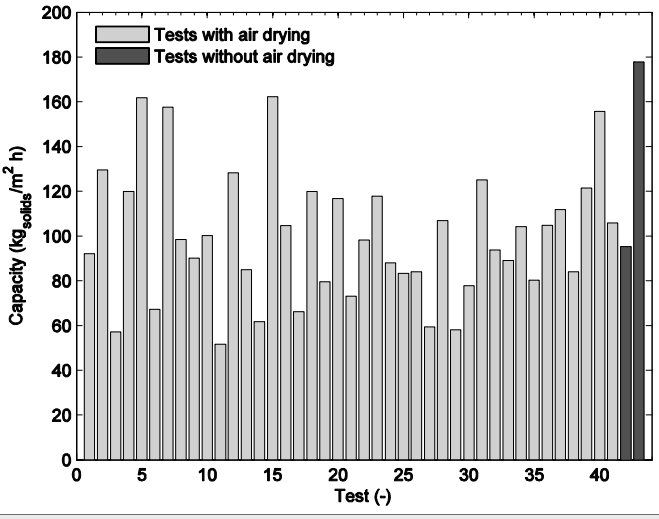

Figure 10: Capacities for the 43 tests.

10. The highest capacity (177.7 kg_{solids}/m^2 h) is obtained from Test 43, which did not contain the air drying stage at all. The lowest capacity (51.1 kg_{solids}/m^2 h), on the other hand, was obtained from Test 11. Figure 11 shows the relationship between the measured moisture contents and capacities for all tests. It is obvious that lower cake moisture contents are achieved by employing test conditions that result in low overall capacities.

Regression Models

The experimental results obtained from this study were used to create empirical models that describe the influence of the studied process variables on the cake moisture contents and capacities. These models were created by regression analysis, which find the optimal coefficients for the variables of the model in such a way that the maximum fit between the predicted and measured values is achieved. The general form of a linear regression model where only the main variables are included can be written as:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \ldots + \beta_n x_n \quad (1)$$

where $\beta_{0,1,2\ldots n}$ are the regression coefficients, $x_{0,1,2\ldots n}$ are the variables, and y is the measured response. For the filtration process introduced in this study, the main effect model is of the following form:

$$y = \beta_0 + \beta_1 \Delta p_{pumping} + \beta_2 t_{pumping} + \beta_3 t_{pressing} + \beta_4 \Delta p_{airdrying} + \beta_5 t_{airdrying} \quad (2)$$

Figure 12 illustrates the predictions from the models that were created by using all the experimental data obtained from the 43 filtration tests. These figures show the moisture contents and capacities that were predicted by applying the regression models (equation 2) against the actual, experimentally obtained, values. If the predictions given by the models were perfect, all the data points would lie on the diagonal line. The figures also show the correlation coefficients (R^2), which are fairly good for both models. Furthermore, all of the data points are distributed evenly on both sides of the diagonal, which implies that the models are stable and significant outliers do not exist.

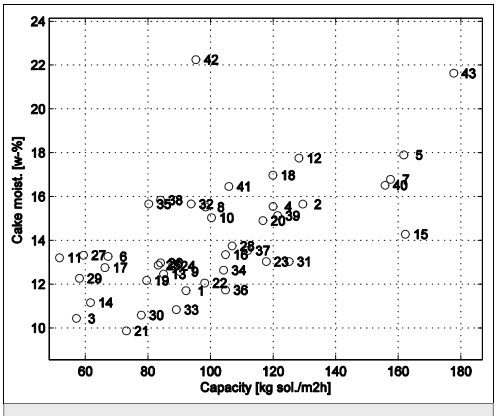

Figure 11: The relationship between the measured moisture contents and capacities for the 43 tests.

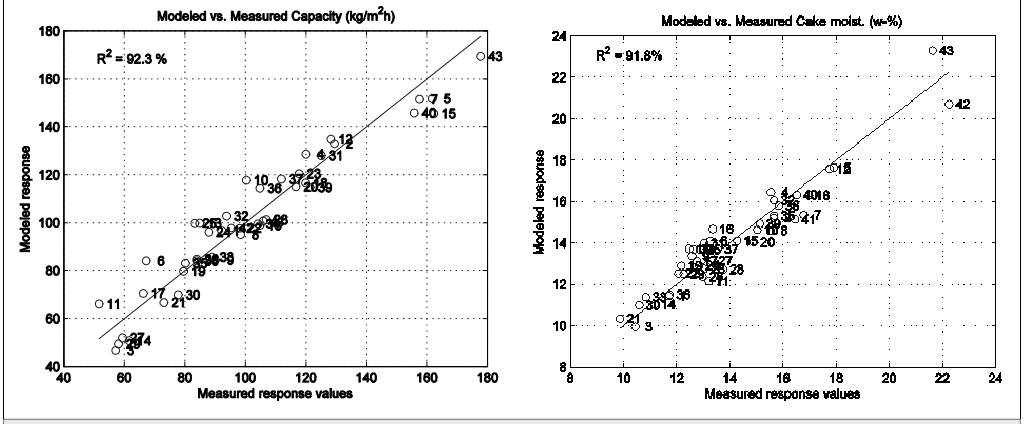

Figure 12: Measured vs. Modelled diagrams of the linear moisture and capacity models for all 43 tests.

The regression models also allowed the main effects of the different variables in the investigated range of the two responses to be estimated. This was done simply by using the coefficients of the main effect models. The main effects of the studied variables on both responses are presented in Table 3.

Comparison of Different Kinds of Designs

Regression models were also used in this context for comparing the suitability of different kinds of experimental designs for these types of filtration studies. The overall number of tests that were carried out was fairly high, 43, and it was therefore possible to divide this set of experiments into many smaller experimental designs which were created according to the different types of fractional factorial designs[4,5]. The main purpose of these comparisons was to define the smallest amount of experiments needed to obtain the required level of accuracy in the results and predictions regarding the examined process.

It can be seen from Table 4 that the amount of experiments required for the different designs varies significantly. All of the introduced designs are, however, based on the assumption of linear relationships, and they should all therefore result in similar models for a system where all the relationships are perfectly linear. This means that comparison of the different designs in fact produces information about the linearity of the studied process. At the same time it should be remembered that there are no experiments lacking in experimental error. This means that the experimental error should be taken into account when considering experimental points showing non-linear behaviour. The possible non-linearities can be most clearly observed by comparing the fit of the centre points with the fit of the other points of the designs.

The smallest number of experiments in the compared designs is 8, which is less than 20% of tests in the full factorial design. For estimating the suitability of this design for the investigated filtration process, new models for cake moisture content and overall filter capacity were created by utilizing the results only from these 8 tests. The results from the remaining 35 (= 43 – 8) tests were used as an independent test set for estimating the prediction capability of these models. By using this kind of external test set, the practical accuracy of the model can be easily and independently estimated.

Variable	Range	Main effect on filter cake moisture (w%)	Main effect on filter capacity (kg/m^2 h)
Pumping pressure	2.0 → 6.0 bar	+1.60	+52.8
Pumping time	240 → 600 s	+1.03	+4.4
Pressing time	120 → 300 s	–0.03	–17.3
Drying air pressure	4.0 → 6.0 bar	–2.22	–1.3
Drying time	180 → 540 s	–2.95	–32.4

Table 3: Main effects of the studied variables on the cake moisture contents and filter capacities.

Model 1	All tests included, even the ones without air drying (43 experiments)
Model 2	Full factorial design with 9 centre points (41 experiments)
Model 3	Full factorial design without the centre points (32 experiments)
Model 4	Fractional factorial design (1/2) with 9 centre points (25 experiments)
Model 5	Fractional factorial design (1/2) without the centre points (16 experiments)
Model 6	Fractional factorial design (1/4) with 9 centre points (17 experiments)
Model 7	Fractional factorial design (1/4) without the centre points (8 experiments)
Model 8	Fractional factorial design (1/4) without the centre points but with the tests without air drying (10 experiments)

Table 4: Different kinds of experimental designs.

The results obtained with the fractional factorial design (Model 7 in Table 4) are presented in Figure 13. This graph also shows the correlation coefficients R^2 for the calibration sets (i.e. the 8 tests in the design) as well as the correlation coefficients Q^2 for the external test sets (= 35 tests). It can be seen that even if the values of these correlation coefficients for the external test set (Q^2) are quite poor, the maximum prediction error is still fairly low in most cases. The poor correlation coefficients are therefore likely caused by experimental inaccuracy and somewhat non-linear behaviour of the investigated process. The small prediction errors, however, suggest that the models obtained by using only 8 tests are capable of providing reliable results.

CONCLUSIONS

There is consensus among filtration experts and professionals about the vital role of experimentation in the design and implementation of efficient and effective solid/liquid separation processes[6-9]. In practice, the test work is often done by personnel who do not have in-depth knowledge of the theoretical basis of solid/liquid separation processes. The task of estimating the overall effect of selected variables on the response requires the use of many different theories and models sequentially, since the filtration process contains sub-processes such as cake formation, compression, cake washing, and dewatering. Unfortunately, there is no unifying model which could integrate the entire process chain used, for example, in a pressure filter.

The LabTop software package was created for test engineers who have limited time and when many variables need to be tested. The software package guides the user in the creation of experimental designs that give structured information of good quality. LabTop provides a quick and easy way to establish the most important variables and to visualise the effect of selected variables on the response. The empirical linear

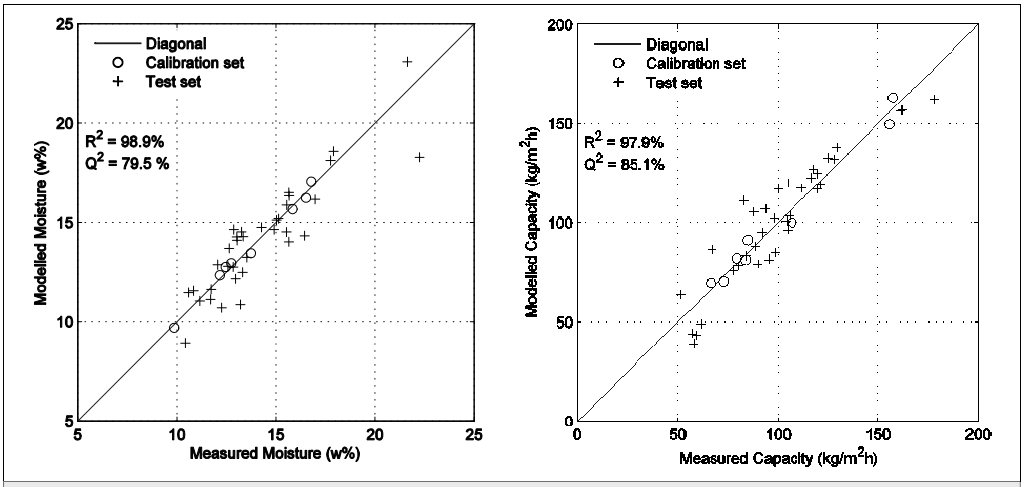

Figure 13: Measured vs. Modelled diagrams for the calibration set and for the external test set. Predictions have been calculated using Model 7 presented in Table 4.

models produced do not replace filtration theory, although they give a good overview of the overall filtration cycle behaviour. It should be remembered that the variable ranges used in this kind of experimental work are limited by the same factors as in industrial processes and therefore these models should not be used beyond the ranges specified.

The results presented in this paper clearly and reliably demonstrate that by using factorial experimental design, the number of filtration tests required to determine the main effects of five filtration process variables could be reduced from 43 to 8 without losing a considerable amount of information. The smallest experimental design this software creates could be used to model the behaviour of the sample case. It is the user's decision how small an experimental design could be used, and it has to be reconsidered on a case-by-case basis.

The examples also show that linear regression models could be quite successfully used to describe the studied filtration process. The experimental work done during the software project showed that filtration capacities and cake moisture content were well modelled with linear models. The results for some processes, for example the cake washing, which is non-linear in nature, need to be viewed with caution. If examined processes turn out to be strongly non-linear in their behaviour, or if considerable variable interactions exist, simple linear models often fail to give satisfactory results. In these cases, more complicated models have to be created with the introduction of additional components. The most commonly applied components for this purpose are direct variable interaction terms and squared variables. The addition of these components, however, renders the models more complicated and more difficult to interpret. For non-linear responses, the experimental design from LabTop provides a good starting point for further test design.

The LabTop software shows the strength of factorial experimental design and helps create measurement data that is structured for further analysis. It also works well in establishing the overall effects of selected variables on the response and provides tools for visualization of these effects.

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ROTATIONAL PARTICLE SEPARATOR: AN EFFICIENT METHOD TO SEPARATE MICRON-SIZED DROPLETS AND PARTICLES FROM FLUIDS

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The rotational particle separator (RPS) has a cyclone type housing within which a rotating cylinder is placed. The rotating cylinder is an assembly of a large number of axially oriented channels, e.g. small diameter pipes. Micron-sized particles entrained in the fluid flowing through the channels are centrifuged towards the walls of the channels. Here they form a layer or film of particles, material which is removed by applying pressure pulses or by flowing of the film itself. Compared to conventional cyclones the RPS is an order of magnitude smaller in size at equal separation performance, while at equal size it separates particles ten times smaller. Applications of the RPS considered are ash removal from hot flue gases in small scale combustion installations, product recovery in the stainless environment for pharmaceutical/food, oil water separation and demisting of gases. Elementary formulae for separation performance are presented and compared with measurements performed with various RPS designs.

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