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WASHING AND DEWATERING OF DIFFERENT STARCHES IN PRESSURE FILTERS

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ABSTRACT

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The main objective of this thesis was to generate better filtration technologies for effective production of pure starch products, and thereby the optimisation of filtration sequences using created models, as well as the synthesis of the theories of different filtration stages, which were suitable for starches.

At first, the structure and the characteristics of the different starch grades are introduced and each starch grade is shown to have special characteristics. These are taken as the basis of the understanding of the differences in the behaviour of the different native starch grades and their modifications in pressure filtration.

Next, the pressure filtration process is divided into stages, which are filtration, cake washing, compression dewatering and displacement dewatering. Each stage is considered individually in their own chapters. The order of the different suitable combinations of the process stages are studied, as well as the proper durations and pressures of the stages. The principles of the theory of each stage are reviewed, the methods for monitoring the progress of each stage are presented, and finally, the modelling of them is introduced. The experimental results obtained from the different stages of starch filtration tests are given and the suitability of the theories and models to the starch filtration are shown.

Finally, the theories and the models are gathered together and shown, that the analysis of the whole starch pressure filtration process can be performed with the software developed.

Keywords:
characterisation, cake filtration, filter media, cake washing, dispersion model, exponential decay model, compression, expression, Terzaghi model, Terzaghi-Voigt model, Shirato model.

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PREFACE

This study has been carried out in the Laboratory of Separation Technology, Department of Chemical Technology in Lappeenranta University of Technology during the years 1999-2005.

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Kuusankoski, October, 2006

Nina Salmela

CONTENTS

RESULTS

LIST OF PUBLICATIONS

This thesis consists of a summary of the following papers, which will be referred to in the text:

I. Salmela, Nina and Oja, Marja, 2000: **The Effect of Compressibility of Different Starches on Starch Washing and Dewatering**, World Filtration Congress 8, Proceeding Volume One, pp. 288-291, April 3-7, 2000, The Brighton Centre, Brighton, UK.

II. Salmela, Nina and Oja, Marja, 2004: **Filter Media Testing for Starch Filtration Systems**, *Filtration*, Vol.4, No. 3, 2004, pp.194-198.

III. Salmela, Nina and Oja, Marja, 1999a**: Washing and Dewatering of Different Starches in Pressure Filters**, *Fluid/Particle Separation Journal*, Vol. 12, No. 2, August 1999, pp.134-139.

IV. Salmela, Nina and Oja, Marja, 2006: **Monitoring and Modelling of Starch Washing**, *International Journal of Food Science and Technology*, Vol. 41, p. 688-697.

V. Salmela, Nina and Oja, Marja, 2005a: **Analysis and Modelling of Starch Dewatering**, *Filtration*, Vol. 5, No. 2, 2005, pp.134-145.

VI. Salmela, N. and Oja, M., 2005b: **Filtration of Starches in a Pressure Filter – methods and simple modelling**, FILTECH 2005, Proceedings, Vol. 1, L-session, pp. l-513-l-519, October 11-13, Wiesbaden, Germany.

Author's contribution to publications:

All experimental work in Papers **I-VI** has been planned by the author, and performed by, or under direct supervision of, the author.

The author was in charge of all the experimental data and modelling in Papers **I-VI**.

The author has been in charge of the preparation of all Papers **I-VI** and taken into account the comments of the co-author.

The software for the analysis and modelling of the starch filtration cycle, which was introduced in Paper **VI**, was planned by the author, and programmed by software designer Artturi Salmela with C++.

NOTATION

A	filtration area	m^2
a_p	actual area of the projected particle image	m^2
B	ratio between the liquid volume squeezed by the secondary consolidation and the total liquid volume squeezed during the entire expression	m^3/m^3
C_{av}	solids volume fraction in the original slurry	-
C_e	modified consolidation coefficient	m^2/s
c	apparent filtration concentration, i.e. mass of dry filter cake per unit volume of filtrate	kg/m^3
c_0	solute concentration in the retained filtrate	-
c_e	solute concentration in the wash effluent	-
c_w	solute concentration in the wash water feed	-
c'	concentration of the wash water at the beginning of the second washing	-
D	binary molecular diffusion coefficient for solute	m^2/s
D_L	axial dispersion coefficient	m^2/s
D_n	dispersion parameter	-
$d_{F(\max)}$	maximum Feret's diameter	m
$d_{F(\min)}$	minimum Feret's diameter	m
d_{Pamas}	average mean diameter given by the Pamas SVSS analyser	m
d_{ECD}	equivalent circle diameter given by the AnalySIS®	m
d_p	particle diameter, mean size of the particles in the cake	m
E	percentage of the solute removed from the cake with a wash ratio of unity	-
F	instantaneous concentration of solute in the wash effluent	-
F_s	fraction of solute removed from the cake	-
i	number of drainage surfaces involved in the dewatering process	-
K	constant in Eq. (5.5)	$m^2/Pa\ s$
K'	Kozeny's constant	-
k_v	flow consistency index	$Pa\ s^{n_v}$
L	cake thickness	m
L_0	original thickness of the slurry	m
L_∞	final cake thickness	m
L_{mix}	thickness of the solid/liquid mixture in the cylinder	m

L_{tr}	thickness of the solid/liquid mixture at the transition moment	m
l	length of the pore	m
m_{av}	mass of wet cake per unit mass of dry cake	-
m_c	mass of dry filter cake per unit area	kg/m^2
m_{cake}	mass of wet filter cake	kg
m_s	mass of dry solids in the cake	kg
m_{tr}	ratio of the mass of wet cake to the mass of dry cake at the end of the filtration stage	-
n	compactibility coefficient	-
n_v	flow behaviour index	-
p_p	perimeter of particle	m
p_t	treshold pressure	Pa
Q_w	interstitial flow velocity of the wash water through a straight capillary in a cake	m^3/s
q_w	average velocity of the wash water flow	m^3/m^2s
q	superficial flow rate	m/s
R_m	filter medium resistance	m^{-1}
R_s	fraction of solute retained in the cake	-
Re	Reynols number	-
r	inside radius of the pore	m
S	cake saturation	-
S_0	specific surface area of the particles	m^{-1}
S_∞	irreducible saturation	-
S_R	reduced saturation	-
Sc	Schmidt number	-
s	mass ratio of dry solids to slurry	kg_{solids}/kg_{slurry}
s_{cake}	solid content of filter cake	-
T_c	dimensionless consolidation time	-
t	time	s
t_c	compression time	s
t_s	time, where the filtration pressure over the cake has reached a constant value	s
t_{tr}	time, when the transition from filtration to consolidation occurs	s
t_w	washing time	s

t_w'	time from the beginning of the second washing period	s
U_c	consolidation ratio	-
V	volume of filtrate	m^3
V_0	volume of slurry in the piston press at the start of the test	m^3
V_l	volume of liquid in the pores of the cake	m^3
V_s	volume of filtrate collected at time t_s	m^3
V_{void}	void volume of the cake	m^3
V_w	volume of the wash liquid used	m^3
V_{f0}	volume of the residual filtrate retained in the cake at the beginning of the washing	m^3
W	void volumes of wash liquid used	-
W_R	wash ratio	-
w	total mass of solids deposited	kg/m^2
w_c	washing parameter	-
α_0	local specific cake resistance at unit applied pressure	m/kg
α_{av}	average specific resistance of the filter cake	m/kg
Δp	pressure difference	Pa
Δp_0	scaling pressure for local specific cake resistance	Pa
Δp_m	pressure below the filter medium	Pa
ε	porosity of the cake	-
ε_{av}	average porosity of the cake	-
Φ_1	shape factor	-
Φ_2	shape factor	-
$\Phi_{AnalySIS}$	AnalySIS® -shape factor	m
γ	shear rate	s^{-1}
η	empirical constant that characterises the creep of the material	s^{-1}
μ	viscosity of filtrate	Pa s
μ_s	shear viscosity	Pa s
μ_w	viscosity of wash liquid	Pa s
θ_1	time constant	s
θ_2	time constant	s
θ_3	time constant	s

ρ	density of the filtrate	kg/m^3
ρ_s	density of dry solids	kg/m^3
ρ_w	density of wash water	kg/m^3
σ	surface tension of the liquid	N/m
τ	shear stress	Pa
υ	consolidation behaviour index	-
ω_0	total volume of solids per unit filtration area submitted to the compression	m^3/m^2
ψ	ratio between the filter medium resistance and the filter cake resistance	m

1 INTRODUCTION

Commercial starches are obtained from seeds, tubers, or roots of different plants in which it serves as a food reserve for the plant. Starches from different plant types have their own, special properties. Additionally, it is also possible to modify native starches by altering their properties to make them more suitable for technical applications. However, it is a very common problem that the differences between the starch types are not sufficiently taken into account in the industrial applications of starches.

A broad assembly of processes and equipment are utilised in starch production. The well-known basic processes are easily maintained and improved, but new equipment and processes are rarely developed for a certain purpose, because it is expensive compared to the use of the already known equipment or processes (Besso, 1976). Traditional equipment in dewatering of starches are decanters and vacuum filters. In a traditional decanter, the filter cake can not be washed and, hence, the purity of the cake can not be maximised. In a vacuum filter the slurry has to be diluted, which increases the water consumption. Also the capacity of the filter is low which in turn slows down the production process.

In a pressure filter the starch slurry is pumped into a filter chamber. The driving force for filtration is the pressure difference in the filter chamber. The filtrate flows out from the chamber and the solid particles form a cake on the filter cloth. The filtration cycle is divided into different stages: filling of the filter chamber and filtration, pre-wash compression, pre-wash displacement, washing, compression, and displacement. Only the filtration and compression stages are necessary, but the use of the other stages depends on the quality requirement of the product.

1.1 MOTIVATION AND PROBLEM DESCRIPTION

Starch production is growing all the time, and at the same time, the quality requirements for starches are becoming stricter. Nowadays modified starches are more frequently used than native starches in the food industry as well as in the technical field. The new trend in starch processing is the processing techniques utilising minimal thermal

treatments and the favouring of the mechanical dewatering. Thermal techniques need to be explored, because of the formation of acrylamides in starchy foods at high temperatures. Acrylamides are carcinogens at doses greater than 0.5 ppm in water (Alavi, 2003). Additionally, during the last few years many new starch-based products have been developed. These are, for example, bio-based paper (Kemia-Kemi, 2002), bioplastics (Härkönen, 2004), and gene modified potato starch for fine grade paper coating (Räsänen, 2004).

Nowadays the pressure filter has become a competitive device in the starch industry, and can replace traditional devices if it is working properly. If a pressure filter is utilised in industrial starch production, the problem is usually that it is operated always the same way, which is not necessarily the most optimal one and does not always work properly with different starch grades. As a matter of fact, the choice of satisfactory operating conditions is difficult, because of the large number of combinations of satisfactory operations (Tiller, 1974). Therefore the comprehensive experimental work; the characterisation of the slurry to be filtered, and the optimisation of the parameters of the different filtration stages, is necessary. When the order of the process stages and the parameters of the different stages of the filtration cycle are selected correctly, the performance of the pressure filter can be guaranteed. The properly working pressure filter is easy to use and maintain, the obtained product will be homogeneous, and the operating costs will be low. The filtration cycles are easily repeated, and the quality of the product can be regulated depending on the demands for the end product, by changing the combination of the process stages and the process parameters.

The former approach to the performance of starches in pressure filtration has been experimental (Satoh, 1957 and Takai *et al*., 1987). The experiments and the modelling have covered only the filtration stage, whereas the dewatering or washing stages have received less consideration. However, nowadays the cake washing and dewatering stages are very important because of the economical point of view as well as because of the strict quality requirements for the starch products. The existing cake washing and compression dewatering models are developed for different types of filtration processes, but not very comprehensively applied to the experimental filtration data. In this work, the models for the different filtration stages are applied to the experimental starch filtration data and developed to be applicable for starch filtration processes. In addition,

the present development of the automatic pressure filters allows the conversion in the order of the filtration process stages, which can be utilised in regulating the product quality and in increasing the capacity of the filter. By changing the order of the process stages it is possible to obtain pure, high quality starches with minimum wash water consumption, and to maximise the solid content of the filter cake. Hence, the same filter can be easily used in filtration of different starch grades, if the order of the process stages is regulated depending on the starch grade.

1.2 OBJECTIVES OF THIS THESIS

The main objective of this thesis is to generate better filtration technologies for effective production of pure starch products, and thereby the optimisation of filtration sequences using created models, as well as the synthesis of the theories of different filtration stages, which are suitable for starches. Based on the above discussion, the following hypotheses are made, and hence, the general aims and the main tasks of this thesis are:

- *The origin of the starch affects on filtration.*
  TASKS: Characterisation of different starches and starch slurries in order to define the specific properties of different starches affecting the filtration behaviour. Distinguish the differences between starch grades.

- *The existing cake filtration theories can be applied to starch cake filtration and the filtration properties of different starches can be evaluated using them.*
  TASKS: To measure the specific filter cake resistances and compactibilities from the experimental test results of different native and modified starches. To perform the tests with a piston press laboratory pressure filter and to analyse the results using the existing cake filtration theories.

- *The order of the process stages in a pressure filtration process can be exploited in order to affect the efficiency of the process and the quality of the product.*
  TASKS: To measure the performances of the starch slurries with a small-scale pressure filter by changing the order of the process stages between the different

filtration tests and by comparing the quality of the product and the capacity of the filter between the test.

- *The testing of the filter media has to be easy, because the type of filter medium has a remarkable effect on the effectiveness of the filtration and the quality of the filtrate.*
  TASKS: To test two different types of filter media, which are used in commercial starch filtration applications. To find an easy way to compare the media in starch filtration applications.

- *Since several models are developed for describing the cake washing and compression dewatering stages in pressure filtration, at least some of them can be applied to the stages of starch cake filtration.*
  TASKS: To test the existing cake washing and dewatering models for starch filtration applications by dividing the process into different stages, modelling each stage individually, and measuring the typical parameters in each suitable model for different starch grades.

- *And finally, the most important task that will help in analysing the whole process cycle: the filtration cycle can be optimised for each starch type.*
  TASKS: To measure the model parameters of the different process stages under typical conditions of each stage for each starch type. To congregate the models of the different process stages for easy modelling of the whole starch pressure filtration process and to develop software for the analysis and modelling of starch filtration.

1.3 THESIS OUTLINE

This thesis comprises of six publications that deal with the different process stages in a starch pressure filtration cycle and unpublished results that complement the studies reported in Publications I to VI. The theory part is a general review of the literature concerning the pressure filtration cycle and serves as a basis for the experiments carried out in this study. The contents of this thesis are described as follows:

THEORY-part:

Chapter 2 introduces commonly the characteristics and the basic properties of different starch granules and slurries. **Chapter 3** presents the filtration fundamentals and the compactive cake filtration theory. **Chapter 4** is a review of the choice of filter medium. **Chapter 5** introduces the filter cake washing theory: methods for monitoring and analysing the cake washing stage in a filter press, and for easy modelling of that stage. Correspondingly, C**hapter 6** introduces the filter cake compression dewatering and displacement dewatering theories. The main emphasis is given to the theory and the modelling of the compression dewatering. Additionally, the measurement of the point at which the transition from filtration to consolidation occurs is critically examined. **Chapter 7** introduces the experimental arrangements used in the experimental part of this thesis.

RESULTS-part:

Chapter 8 introduces the analysed properties of different starch granules and slurries used in the experimental part of this thesis. The main emphasis is given to the properties which affect the filtration behaviour of these starches. Parts of the results are included in Publications I to VI.

Chapter 9 represents the results presented in **Publication I** (Salmela & Oja, 2000). It shows the application of the filtration fundamentals and the compactive cake filtration theory on the filtration of starches with a piston press test filter. The different starch types are classified according to their average specific filtration resistances and compactibilities.

Chapter 10 shows how the different filter media can be easily compared in normal starch filtration applications without any extra devices, and introduces the results shown in **Publication II** (Salmela & Oja, 2004). It is also shown how the choice of the medium affects on the filtration behaviour of the starch slurry.

Chapter 11 introduces the monitoring and analysing of the starch cake washing stage in a filter press (**Publication III,** Salmela & Oja, 1999a), and easy ways of modelling that stage (**Publication IV,** Salmela & Oja, 2006).

Chapter 12 introduces the measurement of the point at which the transition from filtration or washing to consolidation occurs, and the application of filter cake compression dewatering theories and modelling of the starch cake compression (**Publication V,** Salmela & Oja, 2005a).

Chapter 13 represents the principles of the software, which is developed for the easy processing and analysis of the data from the whole filtration cycle. The software is introduced shortly in **Publication VI** (Salmela & Oja, 2005b).

Finally, **Chapter 17** presents the summary and the conclusions of the whole thesis.

1.4 BENEFITS AND NOVELTY VALUES

- *Novel approach of characterisation:*

  The different materials can be characterised by measuring their particle size distribution, average specific cake resistance, and its dependency on the filtration pressure. For starches, the filtration characteristics are grade dependent, and hence the grade defines the optimal filtration cycle parameters. With the known characteristics, the starches are classified after their grade. This improves the understanding and the anticipation of the filtration behaviour of the different starches under different filtration conditions, and decreases the need for the experimental tests.

  The filter media can be characterised by filtering the pure filtrate through the medium in an existing filter, and measuring its specific resistance from the filtrate flow curve. This procedure enhances the comparison of the different media used in the filtration tests.

- *Novel application of the proven approach to model the cake washing and compression dewatering stages:*

  Starch filter cake washing can be modelled with the dispersion model or with the exponential decay model. Starch filter cake compression dewatering can be modelled with the Terzaghi model, Terzaghi-Voigt models, or with the Shirato model. The modelling of the process stages decreases the demand for experimental

work, and enables the calculation of the probable behaviour of the starch slurry during the filtration process, if the model parameters are known for a certain starch.

- *Novel approach of the filtration cycle:*
  In modern automatic pressure filters it is possible to change the order of the process stages. This increases the flexibility of the process and allows for regulating the quality of the product and the throughput of the filter.

- *Novel simulation program:*
  The models of the different process stages are gathered together in order to analyse the test data automatically from the first measuring point to the last. This improves the data handling and analysis, and decreases the amount of miscalculations. This program can be used for the analysis of the filtration of other materials too, especially if the material in question has similar dewatering properties to starches.

2 STARCH

The knowledge of the characteristics of the different starch types is essential in order to be able to interpret their filtration behaviour. Therefore, this chapter gives a review of the origin of different starch types, the uses of starches, the structure of starch granules, and finally the properties of starches affecting their filtration behaviour.

2.1 ORIGIN OF STARCH

Starch is a polymeric plant carbohydrate, with the chemical structure $(C_6H_{10}O_5)_n$. It is obtained from seeds, tubers, or roots of different plants. The main sources of commercial starches are maize, potato, wheat and waxy maize (Kearney and Maurer, 1990). Starch occurs in the form of tiny white granules at various sites in the plants. The diameter of the granules varies from one to 150 μm. Starch is usually sold commercially in a dry form, as a white, free-flowing powder. In plants, starch acts as a reserve-energy store, and it is classified into two types (Codd *et al*., 1975):

1. Starches with a transient existence. For example the starches, which are deposited in green leaves during periods of active photosynthesis and which are broken down to provide energy during periods of darkness.
2. Starches which retain their integrity over long periods of time. For example endosperm, tuber, and rhizome starches, which remain in the plant storage organs over periods of several months during which the plant is dormant. The starches are be broken down for energy at the initiation of new growth.

Starches of type two provide the starches of commerce. Another way of classifying starches is introduced by Jane *et al*., (1994). They divided starches according to their origin:

- Root and tuber starches
- Grain/seed starches
- Maize and related endosperm starches
- Bean and pea starches
- Fruit and nut starches

The seeds of all cereals contain 60-80% starch, which has basically a similar structure independent of the cereal. Starch is bound inside the seed by the endosperm, which contains aleurone layers and starchy endosperms (Codd *et al.*, 1975). For example in maize grains, most of the starch is loosely packed in the top part of the kernel and smaller amounts lie tightly packed in the heart of the kernel (Berkhout, 1976). In agriculture, the term 'corn' is used for various cereals. It describes the most commonly grown cereal in a particular area. Thus, in Europe, 'corn' may refer to various small grained cereals, like wheat, barley or oat. In the United States 'corn' refers mostly to maize.

In **tubers**, like potato and tapioca, which is also called cassava, manioc(a), yucca, mandioca, kaspe, ubi ketella or brazilian arrowroot, starch is located in the end part of the underground stem, for which the parenchymatic tissue has swollen because of starch deposits. The starch is unevenly distributed over the tubers and the starch contents vary between the separate tubers in the plant. A mean content of dry starch is between 15 and 30% in potatoes (De Willingen, 1976), and between 20 and 30% in tapioca (Radley, 1976).

In **roots**, like sweet potato, arrowroot or sago, starch is located in the rhizomes of the plant. In sweet potato, an enlarged root or root section contains about 10-30% starch. In arrowroot, starch is located along the length of the root, which contains about 25-30% starch. Sago starch is located at the pith of several kinds of palm trees. After seven or eight years of growing the palm flowers once and then dies. Just before the flowering takes place, the whole stem, which may be from half to one meter in diameter at the base and over nine meters in height, is loaded with starch. (Radley, 1976)

2.2 THE USES OF STARCHES

Starch is the major dietary component in all human populations and the consensus of recent opinion on healthy eating habits favours an increase in the portion of polymeric plant carbohydrates in the daily diet. Hence, starch constitutes an excellent raw material to modify food texture and consistency, and it is very commonly and increasingly used food carbohydrate (Bello-Perez *et al*., 1999). Starch is used for example in sugars and

syrups, sausages, conserves, bakery products, processed foods, spices and confectionery. Additionally, starch has many chemical and physical characteristics that give it numerous industrial applications too. The most common technical applications of starches are found in the paper industry. Starches are used for to produce specific surface properties required in a finished paper sheet (stuff sizing, web sizing, additive of coating slip), for paper converting (bonding agent), for the textile industry (warp thread sizing, printing), and for the adhesive industry (stamps, labels, cigarettes, sizes and glues). Additionally, starch is used in detergent, foundry, building material, plastic, cosmetic, hygiene and the pharmaceutical industry as well as in oil drilling and mechanical wood processing. (Mentzer, 1984, Moore *et al*., 1984)

The starches may be used in the native form or they may be modified by altering their properties to make them more suitable for certain applications. Most of the modifications are based on the ability of the hydroxyl-groups in the glucose-units of starch to form hydrogen bonds. These bonds are formed between the starch and water molecules, when starch particles are in water. The bonds are weak, but the viscosity of the suspension increases during the formation of the bonds. When starch dries, water evaporates and the hydrogen bonds form between starch and other media. Starch acts as a bonding agent.

Starch may be modified in numerous ways, both physically and chemically. Physical modification is made by pre-gelatinising the starch. Starch slurry is put in a trough between counter-rotating rolls, which are internally heated above the gelatinisation temperature by steam. Another physical modification method is to spray dry a gelatinised starch paste (Daniel & Whistler, 1994). The chemical modifications produce acid-modified, thin-boiling starch, oxidised starch, cross-linked starch, partially esterified or etherified starch, and cationic starches (Daniel & Whistler, 1994).

Pre-gelatinised starches are widely used as a food ingredient, whereas, for example, cationic starches are never encountered in food use. They are widely used for paper manufacturing, because they are attracted to the negatively charged cellulose fibres and fillers thus increasing the bonding (Snyder, 1984, Mentzer, 1984, Rojas & Neuman, 1999, Bobacka & Eklund, 1999, Merta, 2001). By modifying native starch, it can be used in many different applications. The modification of starches is introduced for

example by Codd *et al*., 1975, Radley, 1976, Rutenberg & Solarek, 1984, Knight & Olson, 1984, Rohwer & Klem, 1984, Mentzer, 1984, Moore, *et al*., 1984, and Mitrevej *et al*., 1998.

2.3 STARCH GRANULE STRUCTURE

The structure of the starch granules is very broadly studied. The knowledge of the structure was increased and sharpened widely during the last century (Tester *et al*., 2004). Hence, within this thesis work the structure of starch granules will only be introduced briefly.

2.3.1 Main Components: Amylose and Amylopectin

All starch grades are composed of a single type of sugar unit, D-glucosyl unit. Each starch granule is composed of two main polysaccharide components: amylose and amylopectin, such structures are shown in Figure 2.1. These components represent approximately 98-99% of the dry weight of starch (Tester *et al*., 2004). Amylose, with molar mass of 10^6 kg/kmol, is a linear molecule consisting of (1→4)-linked α-D-glucopyranose units. Amylopectin, with molar mass of 10^7-10^9 kg/kmol, is a highly branched molecule consisting of short chains of (1→4)-linked α-D-glucose with (1→6)-α-linked branches. These linkages appear at intervals of approximately 20 units (7 nm), depending on the starch source. Amylose and amylopectin molecules have very different physical properties. For example, when starch is mixed with water, amylopectin absorbs water and swells, whereas amylose forms a clear viscous gel (Knight, 2001).

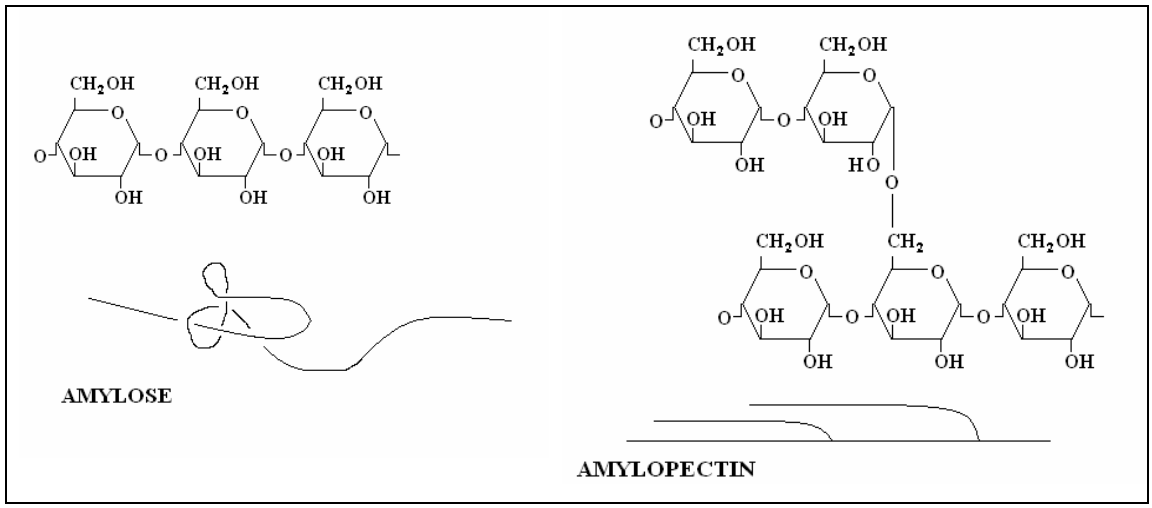

Figure 2.1 The structure of chains of two of the main components in starch granules.

According to Ahmad & Williams (1998) many common starches contain only 20-32% amylose. However, the amylose content and the amylose fine structure determine the properties of the starch granule. Plants can also produce starches with amylose to amylopectin contents outside the 'normal' range. Amylopectin alone can generate granules, which contain less than 15% amylose and are called 'waxy' starches. Respectively, the 'high-amylose' starches contain more than 40% amylose (Shannon & Garwood, 1984, Tester *et al*., 2004). For example maize starch can have the amylose content as high as 70% (amylo-maize) or as low as zero (waxy-maize). Such families of starches are useful for studying the roles of amylose and amylopectin within the granule (Oates, 1997). Table 2.1 shows the typical amylose contents of different starches.

Table 2.1 The typical amylose contents of different starches (Young, 1984).

STARCH	AMYLOSE CONTENT, %
Wheat	26
Barley	22
Waxy barley	0
Potato	20
Maize	28
Waxy maize	0.8
Amylo-maize	52-75
Sweet potato	18
Tapioca	17

2.3.2 Structural Features

The first growth layer of the starch granule, hilum, is initiated at the centre of the granule. The hilum contains a large portion of the reducing ends of the starch molecules and it is usually less organised than the rest of the granule. The non-reducing ends radiate out towards the surface of the granule. They allow the addition of further glucose residues, so that further growth occurs from the surface as the fresh cells of the amylopectin molecules are added, forming concentric rings, also known as growth rings. These structures are visible under a light microscope in large granules, like potato granules, as shown in Figure 2.2, but are seldom seen in small-granule starches, like barley and rice. (Oates, 1997, Gallant *et al*., 1997, Waigh *et al*., 1998, Nordmark & Ziegler, 2002, Tester *et al*., 2004)

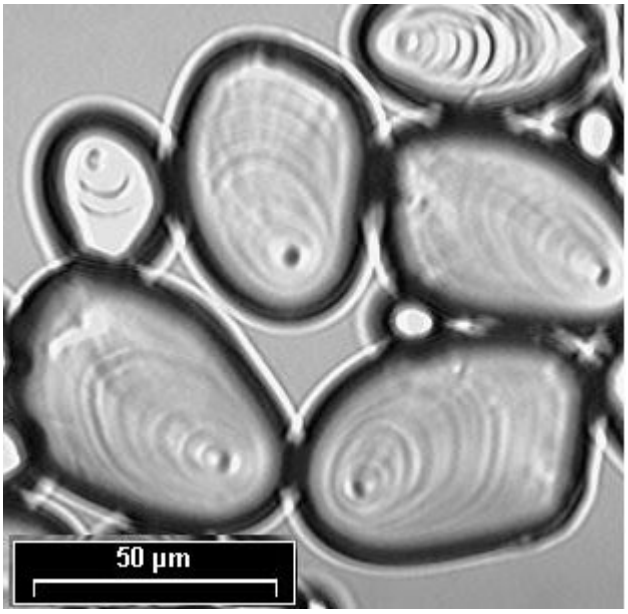

Figure 2.2 The hilum and the growth rings of the native potato starch granules measured with an optical microscope Olympus BHS connected with AnalySIS®Soft Imaging System via Hamamatsu C2400 camera unit.

A typical cereal starch granule involves amorphous and crystalline lamellae, called 'crystalline blocklets', in which amylose and amylopectin are embedded. According to Gallant *et al*., (1997) the amylopectin polymer is predominantly responsible for the starch granule crystallinity. The crystalline lamellae of amylopectins are about 9-10 nm thick and form the backbone of the granule. They are organised into larger, more or less spherical structures, stacks of lamellae. These hard layers are about 120-400 nm thick

on average depending on the starch type and on the location in the granule. Typically, natural starch granules range in degree of crystallinity from about 15% to 45% (Cheetman & Tao 1998). The amorphous fraction is composed of amylose, which is responsible for the elasticity of the system and which controls the variation in the granule volume due to its ability to absorb and release free water. Blocklets are found to have somewhat the same size distribution in smaller starch granules and in the centre of the larger potato starch granules (Gallant *et al*., 1997).

The starch granule crystallinity can be characterised into three main X-ray diffraction patterns: A, B and C type (for example French, 1984, Whistler & Daniel, 1984, Nordmark & Ziegler, 2002, Tester *et al*., 2004). These patterns are the result of different packing of the amylopectin side-chain double helices. In native granular forms, the A-pattern, monoclinic lattice, is associated mainly with the cereal starches, while B form, hexagonal lattice, is usually obtained from the tuber starches. The C pattern is a mixture of both A and B types, but also occurs naturally in the pea and bean starches. In addition to these types, the V-type is a result of amylose being complexed with aliphatic fatty acids, emulsifiers, butanol or iodine. The main difference between the A and B type is that the former adopts a close-packed arrangement with water molecules between each double helical structure. The B-type is more open, and there are more water molecules (Wootton & Bamunuarachchi, 1978). This difference was easily seen in the native starches used in the experimental part of this work: A-type starches wheat, maize and barley, which were stored in paper bags, contained 10% absorbed water, whereas the B-type potato starch contained about 20% absorbed water.

2.3.3 Granule Surface and Internal Structure

The porosity of starch granules to water or small, soluble molecules has commonly been based on the assumption that amorphous regions penetrate throughout the entire granule and that on hydration they reversibly swell, forming a continuous gel phase (French, 1984).

Huber & BeMiller (2000) showed the structure of the granules consisting of pores at the surface of the starch granules, internal cavities at the granule hilum from centre outward

and lateral channels between them. The tests were carried out by treating starch granules with an aqueous suspension colloidal gold and viewing by scanning electron microscope (SEM). No dependence between the channels and the cavities were found. The channels showed voids rather than regions occupied by amorphous material. The channels penetrated the granule to varying depths, and most extended to the central cavity. Channels within different starch grades appeared to differ in diameter and in number, and the populations of starch granules were heterogeneous in terms of granular microstructure.

Huber & BeMiller (2000) ascertained also the effect of granule re-hydration on the channel and cavity structure, because the industrial starch reactions are conducted in aqueous media under slightly swelling conditions. The cavities in never-dried maize starch granules were substantially smaller than those in both intermediate and completely processed granules. The channels appeared to remain open and to enlarge in water. The cavities could have been formed by crystallisation of the amylopectin molecules and concurrent shrinkage of the matrix as the granule grows and develops. Drying could then enlarge cavities.

Oates (1997) has shown the presence of surface openings, with a diameter of 0.1-0.3 μm and interior channels with diameter of 0.07-0.1 μm, with transmission electron microscopy (TEM) of dry starch granule sections. He was not able to prove whether the surface or interior holes were formed naturally or were they the result of drying during the processing or sample preparation. Conceivably, he claimed that pressures imposed on the granule by the removal of water during drying might have generated a hole at the weakest point, the hilum.

2.3.4 Granule Swelling and Gelatinisation

In its natural form, starch contains a lot of water. Most of the water is bound in the amorphous gel phase or at the surface of the crystallites (French, 1984). In water the amorphous phase is limitedly and reversibly swelled with the consequent swelling of the entire granule. Satoh (1957) found that the diameter of potato, sweet potato, maize

and tapioca starches became 1.12 times greater in the wet state, when the particles were allowed to swell in water for the duration of the day. According to Shannon & Garwood (1984), the starch granule can absorb water even up to 50% in weight and expand from 30% to over 100% in volume. In swollen starch granules the channels and cavities allow molecules smaller than 1000 g/mol to be admitted into the granule to varying extents. Drying causes the granule to shrink and crack, and also partial realignment and reorganisation of the polysaccharides occur. Dried starch granules are insoluble in cold water. However, the amorphous phase undergoes a limited reversible swelling again with consequent swelling of the entire granule when exposed to liquid water or water vapour at 0-40°C. On re-hydration, the dried starch granules do not resume their fully native form.

The amount and distribution of water within the starch granules is critically important. It affects the physical properties and chemical reactions of starch, as well as the understanding of the role of water in the industrial processing of starch. The amount of water bound by starch is expressed as the percent of the total weight or add-on weight to the dry starch. When the moisture value is given it should be expressed on how it has been determined.

When the aqueous starch suspension is heated above a certain temperature, more swelling and dissolution occurs (Ahmad & Williams, 1998). In the beginning of starch granule swelling the structural changes within the granule are just starting to occur. This is the stage where the amorphous regions have been swollen due to the water absorption and the crystallite melting is just starting to occur (Gallant *et al.*, 1997). The point, where the whole starch slurry begins to thicken and show an increase in viscosity is called the pasting temperature (Snyder, 1984), whereas the gelatinisation temperature is the temperature at which the crystalline structure of the granules is totally disrupted (Ahmad & Williams, 1999).

Every starch grade has its own typical gelatinisation temperature, which varies from 60 to 85°C depending on the source of the starch, the relative amounts of the amylose and the amylopectin, and the amount of moisture available for hydration (Alavi, 2003). Each individual starch granule gelatinises quite sharply, but they do not gelatinise at the same temperature (Snyder, 1984). The variance of the gelatinisation temperature of each

starch grade is from 8 to 10°C. This is due to the differences in internal bonding forces within the individual granules. The gelatinisation of the starch suspension by heat treatment or by ultra high pressure treatment, in which the pressure is greater than 400 MPa, can be measured from the loss of a birefringence, which is characteristic for native starches. It is seen in the form of a 'Maltese cross', a characteristic dark cross seen on starch granules at the point at which the hilum is centred, when the granules are viewed under polarized light.

Stapley *et al*. (1999) have studied the use of NMR tomography and differential scanning calorimetry (DSC) to examine the diffusion of water into the wheat grains. The images of boiled grains show that water penetrates into the grain as a well-defined moisture front with a marked difference between the moisture content near the surface and at the centre of the grain. In contrast, the images of steaming show essentially uniform moisture profiles across the grain. When steam is taken up by the grain it is ultimately absorbed as liquid water. The steamed grains show a much narrower gelatinisation temperature range than the boiled grains.

2.3.5 Impurities in Starch and in Starch Slurry

Normal starch granules contain some impurities, like protein (<0.5%), fat (<1%), ash (<0.4%), and phosphorous (<0.06%). The cereal starches contain internal lipids and surface lipids, whereas potato starch does not contain lipids. Also the protein content in potato starch is lower than that in cereal starches. Additionally, all starches contain small quantities of minerals, such as calcium, magnesium, potassium and sodium. Other starches, except potato, generally contain little phosphate (Tester *et al*., 2004). The contamination of starch from storage proteins, lipids, is commonly identified by measuring the nitrogen content of starch. (Vasanthan *et al*., 1999)

Some impurities in starch suspension may have effects on the characteristics of the suspension. Ahmad & Williams (1999) have shown that salts have a significant effect on gelatinisation, swelling and rheological properties of starches. Salts cause an elevation or depression of the gelatinisation temperature and enthalpy, and similarly increase or decrease the rate and the degree of gelatinisation and retrogradation, which

is an inverse process to hydration and leads to a more organised structure. The effects are due to the polymer-solvent interactions, which are influenced to a great extent by the anions, or due to the formation of complexes and the disruption of polymer chain aggregation caused by the interaction between the cations and the hydroxyl groups of the starch molecules. Both, the starch lipids and proteins have the potential to moderate the starch functionality. The minerals are of little functional significance, except phosphorus. Knight (2001) has shown that highly soluble components, like electrolytes (sodium chloride) or non-ionic materials (polysaccharides), reduce the thermodynamic activity of water in contact with the starch granules immediately after mixing with water. For a short period, this inhibits the swelling and dissolution of the granules, if the soluble material coats, or is intimately mixed with the starch granules. The material is effective only for a short period before it diffuses or disperses into the bulk solution.

2.4 PROPERTIES OF STARCHES AFFECTING FILTRATION

The properties of starches alter depending on the plant source as well as on the place of growth. Particle size distribution, particle shape, density, and surface properties can be considered as primary particle properties. Knowing them will help to predict the secondary properties like the settling velocities of the particles or the specific resistances of filter cakes. These secondary properties are critical in understanding the filtration behaviour of different starch slurries during filtration (Svarovsky, 1977).

2.4.1 Particle Size and Particle Size Distribution

The granule size range gives frequently useful information, because it is directly related to the rate of filter cake formation in filtration. Additionally, the particle size data is useful in monitoring the effect of various processing changes or modifications of the granular starch product.

Variations in starch granule size, shape, and size distribution reflect the botanical origin. Starch granules range in size from sub-micron elongated granules of chloroplasts to the relatively large (>100 μm) oval granules of potato and canna. Also the granules isolated

from the different parts of the same plant may be entirely different in size and shape (French, 1984). The particle size distribution may be uni-modal or bi-modal (Stapley *et al*., 1999, Manelius & Bertoft, 2002, Tester *et al*., 2004). For example Andersson *et al*., (1999) have shown that the barley starch exhibits a bi-modal particle size distribution with large lens-shaped granules of an A-type X-ray diffraction pattern (10-25 μm), and small spherical B-type granules (<6 μm). The A-granules make up 90% of the starch volume even though the B-granules are more numerous, up to 95% by number. According to Vasanthan *et al*., (1999) the potato starch has a narrow size distribution, but the mean particle size may differ from 36 μm to 46 μm depending on the potato variety. Also the proportion of small (<26 μm) and large (>26 μm) granules differ substantially among potato starch varieties. Mota *et al*., (1999) studied the effect of the fine particles on the porosity of the starch. They found that the introduction of a fraction of fine particles into the binary mixture of the starch particles led to a dramatic decrease in the porosity. For a volume fraction of smaller particles as low as 0.06-0.10 in the total volume of particles, the porosity was 20-30% of the mono-sized porous media. According to Tester *et al*., (2004) there is still a lack of understanding why there is a distribution of granules of different sizes in the starches.

2.4.2 Particle Shape

The shapes of the starch particles can be studied with the optical microscope or with the scanning electron microscope (SEM). The light microscope provides the advantages of colour through selective staining as well as the details on the internal structure. These advantages are not available by SEM, which in turn has two major advantages over the use of a light microscope: it has hundreds of times greater depth of focus than the optical microscope, and the resolution is on the order of 7 nm or less depending on the instrument used. So SEM permits the study of fine particle surface structure of starches (Snyder, 1984, and Fitt & Snyder, 1984, Jane *et al*., 1994). Jane *et al*. (1994) have proposed that the magnification of ×1500 is suitable for most of the 54 starches they studied, and can be generally used in studying of starch particle shapes.

Some qualitative terms of the nature of starch particle shape are published (French, 1984, and Fitt & Snyder, 1984, Singh *et al*., 2003). Generally, SEM of undamaged or

unmodified starch granules shows that the granule surface is relatively smooth and free from pores, cracks, or fissures. Jane *et al*., (1994) measured the shape of 54 starch types, and classified them based on their botanical origin. Generally, root and tuber starch granules are large and smooth, grain starch granules are thicker, and have a round or pancake-like shape, maize-type starches are irregularly shaped polygons with sharp edges and their smoothness increases with increasing amylose content, bean and pea starches are thick disks with cut ends, and fruit and nut starch particle shapes varies according to their origin. With some cereal starches, there are indentations thought to be impressions of spherical protein microbodies, which have impeded the local growth of the starch granule. When the starch granule is damaged or eroded, either by acidic or enzymic treatment, the granules are weakened so that many granules crack open and expose a pronounced layer structure (French, 1984). The classification of the shapes of the starch particles is shown in Table 2.2.

Table 2.2 Examples of typical shapes and features of starch particles based on the measurements made in this study and the studies by French (1984), Fitt & Snyder (1984), and Jane *et al*., (1994).

Starch	Typical particle shape	Typical features
Wheat	Small: spherical Larger: round, disk, lens-shaped	Smooth, striated and dimpled
Maize	Polyhedral - Grounded (from floury endosperm) - Angular (from horny endosperm)	Grounded: smooth Angular: grooved, dimpled Crater-shaped impressions (caused by zein bodies pressing into the soft endosperm of the growing maize kernel)
Potato	Oval, round 'Oyster shell' (hilum & lamellation)	Smooth
Barley	Round Disk-shaped (flattened centre)	
Tapioca	Round Truncated Egg-shaped / cap-shaped	Smooth Fractured sides - dimpled - more textured
Rye	Disk, round	
Rice	Irregular: polygonal 'sharp stones' Polyhedral	
High-amylose maize (55%)	Elongated irregular filamentous Rods, 'snakes'	Smooth

The light microscope gives information on the shape and size of the particles, as well as the centric or eccentric position of the hilum and the brilliance of the interference cross under polarized light. It also provides a useful and rapid way of identifying the various native starches. In ordinary light, dry starch granules are clear and colourless. French (1984) has shown that large hydrated granules show growth rings, which represents concentric cells or layers altering in high and low refractive index, density, crystallinity,

and resistance to chemical or enzymatic attack. The rings are due to the deposition of polymer layers of increasing organisation around the water-rich central region. They are visible, because the refractive index of water is 1.33, whereas that of starch is 1.53 (French, 1984). Thus, because of the contrast in refractive indices, the layers of higher and lower starch content give visible rings. During drying this region shrinks more than the rest of the granule leaving a void or an air pocket. With small starch granules, the growth rings are seldom seen by optical microscope, but with bigger granules, like potato, they are easily seen.

The shapes of the particles can be compared with the shape factors. The shape factor, Φ_1, provides information about the shape of the particle. For a spherical particle the shape factor is one, and for other particles, the shape factor smaller than one. The first shape factor measured describes slimness of the particle, and it is calculated (Oja, 1996)

$$\Phi_1 = \frac{d_{F(\min)}}{d_{F(\max)}} \tag{2.1}$$

where $d_{F(\min)}$ is minimum Feret's diameter and $d_{F(\max)}$ is maximum Feret's diameter.

The AnalySIS® -shape factor does not describe any specific shape. It is given by the AnalySIS® Soft Imaging System, and it helps in comparing the different starch grades analysed with the software. It is calculated as follows:

$$\Phi_{AnalySIS} = 4\pi \frac{a_p}{{p_p}^2} \tag{2.2}$$

where a_p is the actual area of the projected particle image and p_p is its perimeter.

The third shape factor, Φ_2, compares the stability of the particle position during the particle size measurement. It can considered that the average mean diameter given by the Pamas SVSS analyser, d_{Pamas}, is measured at an unstable position, whereas the equivalent circle diameter given by the AnalySIS® Soft Imaging System, d_{ECD}, is measured at a stable position. However, they can be considered to describe the same dimension, and hence the shape factor is calculated as follows:

$$\Phi_2 = \frac{d_{Pamas}}{d_{ECD}} \tag{2.3}$$

2.4.3 Densities of Starches

The density of particulate material affects the settling rate of the particles in water. Hence, the density has to be taken into account in planning of the filtration, especially if dilute suspensions are to be filtered. The density of starch depends on its botanical origin, prior treatment, and method of measurement. The measurement can be made by adding a weighed amount of starch of known moisture content to a calibrated volumetric flask or pycnometer, making up to volume with water, and obtaining the combined weight of the starch plus water. For example, dried granular maize starch increases the volume by 0.611 cm^3 per gram of anhydrous starch (French, 1984). This specific volume corresponds to an apparent density of 1637 kg/m^3. For potato starch, the corresponding value is 1617 kg/m^3. Typically, the apparent specific volume of starch dissolved in water is between 0.61 and 0.63 cm^3/g (French, 1984). If starches are air-dried, which means that the starch contains absorbed water, the densities are:

- potato (which contains 16% water) 1450 kg/m^3, and
- maize (which contains 10% water) 1500 kg/m^3 (French, 1984).

2.4.4 Settling of Starch

In a properly performed filtration test, the effect of sedimentation in the filter should be removed. However, in the gravitational field, it is difficult to cancel the effect of sedimentation by any experimental procedure when the dilute solutions are to be filtered. The formation of starch cake by filtration at pressures below one bar is found to accompany the sedimentation (Takai *et al.*, 1987). After the completion of sedimentation the liquid flow is by permeation. When the particles settle to form the cake, they are mutually influenced by each other. The structure of the cake may be different in different parts of the cake.

The objective of a settling test is to determine the initial constant rate of settling, clarity of the supernatant liquid and the final proportion of the sediment layer. First the solid content of the slurry is measured. Then a one litre graduated measuring cylinder is filled to the one litre mark with the sample slurry, which is agitated to ensure uniform dispersion of the particles. The cylinder is placed on a rigid surface and the slurry is allowed to settle. The height of the suspension-supernatant interface is recorded at suitable intervals from the graduation on the cylinder and the elapsed time is noted at the same time. The settling is allowed to continue until it is finished. The settling rate is determined by plotting the suspension-supernatant interface height versus time and calculating the gradient of the line (Rushton *et al*., 1996, Wakeman & Tarleton, 1999, Besra *et al*., 2000).

For example in effluents, which contain small amounts of starch particles the settling may be a problem. The starch particles in dilute suspensions tend to settle very rapidly due to their relatively high specific gravity. This characteristic can be utilised in the removal of starch from the liquid carrier, but it can also create difficult mechanical handling problems when settled. The settlement forms a stiff cake-like material which has to be stored in a tank or container. In this state starch is difficult for example to pump. Liquid velocities in pipework have to be high enough to prevent plugging. The piping has to be self-draining for that reason, and the slopes of the tanks, pits, chests, etc. have to be adequate in their design. (Banks, 1978)

2.4.5 Flow Behaviour of Starch Slurries

The material handling problems in filtration can be minimised by testing the rheology of the materials at all conditions that will arise in the plant (Carleton & Heywood, 1983). Rheology is a science of material flow and deformation (Binder, 1962, Massey, 1979). The measure of viscosity describes the ability of a fluid to flow, and it is proportional to the flow time (White, 1974). A suspension is Newtonian if the shear viscosity, μ_s, does not vary with shear rate, γ. However, in the vast majority of all cases, the viscosity decreases with increasing shear rate, and then the behaviour is non-Newtonian, shear-thinning behaviour. If the viscosity increases with increasing shear rate, the behaviour is

non-Newtonian, shear-thickening rheology (Barnes *et al*., 1989). Viscosity is calculated as follows:

$$\mu_s = \frac{\tau}{\gamma} \tag{2.4}$$

where μ_s is shear viscosity, τ shear stress, and γ shear rate. The shear stress versus shear rate curve is a straight line for Newtonian suspensions, whose viscosity is independent of shear rate. According to Crochet *et al*., (1984) for most non-Newtonian systems, μ_s is found to be a monotonic decreasing function of γ representing shear-thinning behaviour. In this case the curve showing the viscosity against the shear rate is usually first falling from a zero shear rate 'first-Newtonian value' μ_0 to a 'second-Newtonian value' μ_2 at very high shear rates, which can be as much as several orders of magnitude lower than μ_0. The higher constant value is called the 'zero-shear viscosity' (Barnes *et al*., 1989). The decreasing part of the curve between these values is usually well approximated by the Ostwald-de Waele power law model for starch slurries/pastes (Natarajan and Suppes, 1997, Sopade & Kiaka, 2001)

$$\tau = k_v \, \gamma^{n_v} \tag{2.5}$$

$$\mu_s = k_v \, \gamma^{n_v - 1} \tag{2.6}$$

where k_v is the flow consistency index and n_v is the flow behaviour index. For Newtonian materials $n_v = 1$ and for shear thinning substances, n_v is less than unity. If $n_v < 1$ the slurry is *pseudoplastic*, and its μ_s decreases with increasing γ. If $n_v > 1$ the slurry is *dilatant*, and its μ_s increases with increasing γ.

2.5 SUMMARY

This chapter gives an overview of the different types of starches and their typical properties, which have to be taken in account in filtration. First the starches were classified according to their origin and to the way they store energy. The main uses of starches in the food industry and in technical fields were reviewed as well as the methods for modifying native starches by altering their properties. The starch granule structure was introduced briefly in order to understand the filtration behaviour. First, the

main components, amylose and amylopectin, and their ways to form the polymer chains were introduced (Figure 2.1). Then the structural features, like hilum and growth rings (Figure 2.2.) as well as crystallinity and crystalline blocklets, and their characterisation were discussed. An understanding of the starch particle behaviour during filtration was deepened by considering the surface and the internal structure of the starch particles, and the property of the starch slurry to swell and gelatinise during heating. Also the typical impurities and their contents in starch granules were introduced.

Next typical properties which affect the pressure filtration process of starch slurries were introduced. The particle size and shape, and the particle size distribution were shown to differ depending on the origin of the starch. The definitions for the shapes of the particles were shown in Table 2.2. Also the shape factors were introduced in order to show how to get quantitative information about the shapes of the starch particles (Equations 2.1-2.3) and how to compare the shapes of the different starch grades. The measurement of the density of starch powders and slurries were introduced in order to show their possible effects on the filtration. Also the effect of the settling of starch particles during filtration and the effect of the viscosity of starch slurry at its typical filtration concentration were discussed. Finally, the power law –model curve for the measurement of the effect of changes on the slurry viscosity in filtration was introduced.

3 FILTRATION STAGE

In cake filtration particles are separated from a suspension by the use of a medium, which is permeable to liquid flow but does not allow the passage of the particles. The medium is placed into the one side of the filter chamber. The chamber is closed and the slurry is pumped into the chamber, above the filter medium. The filtrate flows through the filter medium, but the solid particles are retained on the surface of the medium. This causes the formation of a filter cake above the filter medium. The driving force for filtration in a pressure filter is liquid pressure developed by pumping or by the force of gas pressure in the feed vessel. Sperry (1916) has listed the factors affecting the filtration process: the filtration pressure, the time of filtering, the rate of deposition on the mixture, the resistance of the material, the concentration of solids in the mixture, the resistance of the filter medium, the temperature of the mixture, the pressure to allow for the squeezing together of non-rigid solids as the pressure increases, and the influence of gravity through the agency of settling or sedimentation.

The formation of a filter cake in a filter press is a basis of the success of the following filtration cycle stages. The washability as well as the dewatering properties of starch depend on the structure of the formed filter cake. The cake structure may be described by the porosity, the permeability, and the average specific resistance of the cake, which are in turn affected by the cake formation rate, the filtration pressure, and the duration of the filtration stage. The commonly known basic theories of the filtration stage are reviewed here, since the filtration stage in starch cake filtration tests is performed according to these theories. The filtration stage is optimised and the best filtration conditions for every starch type are determined.

3.1 CAKE FORMATION AND GROWTH

At the beginning of the filtration the whole pressure drop available is across the medium itself since as yet no cake is formed. As the pores of the medium are normally small and the rate of filtrate flow is slow, the flow conditions are almost invariably laminar (Tiller, 1974, Svarovsky, 2000). When the particles are retained at the surface of the medium,

they form a cake. As soon as the first layer of the cake is formed, the subsequent filtration takes place on the top of this cake and the medium provides only a supporting function. A greater proportion of the available pressure drop is taken up by the cake itself. This results in an effective increase in the bed resistance thus leading to a gradual drop in the filtrate flow rate in constant pressure filtration. This theory, based on Ruth's works (1933a, 1933b, and 1935) is commonly known as the conventional filtration theory or as the two-resistance theory, since the total resistance in filtration is supposed to comprise of a series of the resistances of a medium and of a cake (Lee & Wang, 2000).

Bonding forces between the cake and the cloth are dependent on how the first layer of the cake is formed. So it is necessary to ensure that the solids concentration, the state of flocculation, and the filtering velocity in the laboratory scale are the same as in the industrial scale. It is also advantageous if the filtration tests are conducted at the same pressure as used in the plant-scale operation whereby complications due to cake compressibility are avoided (Marecek & Novotny, 1980, Carleton & Heywood, 1983). A common technique in industry is to start filtration with a gradual increase in the pressure drop (Satoh, 1957). This allows the particles in the initial layers to form with more open packing and lower flow resistance, and minimises the entrainment of air in the cake. Trapped air bubbles can cause the resistance to increase and to make an otherwise incompressible or non-clogging cake appear to be compressible or clogging (Chase & Willis, 1991).

When the decision of the suitable magnitude of the filtration pressure is made, some important aspects have to be taken into account: The use of high pressure drop is often advantageous, leading to higher outputs, drier cakes, or greater clarity of the filtrate. However, if the solid material is compressible, an increase in the pressure drop leads to a decrease in permeability of the cake and an increase in the specific cake resistance. The change in the cake structure and thickness, in turn, affect the filtration performance. This reduction may be so significant as to nullify the advantage of using high pressures. (Shirato *et al*., 1986b, Svarovsky, 2000)

In many cases, when studying cake formation, the overall approach to the cake formation is sufficient. However, the relevant information about the local properties of the cake and of the build-up of the cake is studied by many authors. For example, Lu & Hwang (1993a and 1993b) studied the mechanism of cake formation under constant pressure by adopting the concept of the critical friction angle of the particles. The packing structure of the spherical particles was found to differ depending on the friction angle between the deposited particles and the depositing particle. Stamatakis & Tien (1991) made the analysis of the theory of the cake formation and growth in cake filtration. They predicted empirically the cake growth, the filtration rate as well as the structure and the permeability of the cake based on the operating conditions and the material characteristics. The cake was assumed to be formed when the local volume fraction of solids, the so called solidosity, exceeded a certain critical value, and then to grow in thickness as a function of time. At the same time the formed cake was assumed to be subjected to a compressive stress such that the structure of the cake underwent a continuous change. This change was, in turn, manifested by a continuous change in the local solidosity as well as the local permeability. This in turn affected the flow of the liquid through the cake and therefore the cake growth rate. Chase *et al*., (1994) observed the particle packing and cake formation directly with a microscope and video. The images showed particles in the slurry colliding with and sometimes adhering to other particles when forming the cake. In 1995 Fathi-Najafi & Theliander developed an apparatus with which they measured the hydraulic pressure distribution in the filter cake during its formation and hence examined whether the formation was smooth or not. In 1996 Theliander & Fathi-Najafi developed a computer program describing the build-up of a cake. The filtration process was divided into equal-sized filtrate volume intervals, and hence called the ‘layer by layer’-model. It allowed the calculation of the cake height, the pressure profile, and the local specific filtration resistance. Keinänen *et al*., (1994) studied the number and the location of feeds, and the shape and the height of the filter chamber in a Larox pressure filter and found that the structure of the chamber and feeds affected significantly on the structure of the cake.

3.2 FILTRATION THEORIES

The classical filtration theory is developed by Ruth *et al*., (1933a and 1933b) and Ruth (1935). It is based on the assumption of the constancy of the filter medium resistance and the cake resistance, so that the total resistance in the filtration comprises of a series of resistances of the medium and that of the cake (Lee & Wang, 2000). After that theory, various investigators have suggested ways of estimating the rate of liquid flow through filter cakes exhibiting compressible behaviour, as well as estimating the specific resistance of those cakes. For example Tiller and his co-workers have made extensive work within the field (for example Tiller, 1955, Tiller & Cooper, 1960, Tiller & Shirato, 1964, and Tiller & Green, 1973, Tiller, 1974, Tiller & Crump, 1977, Tiller & Yeh, 1987). Then many other investigators have based their work on the work of Tiller's. Only two papers, except the author's own, concerning the filtration of starches are published. In 1957 Satoh reformed the Ruth's constant pressure filtration equation based on the assumption about the gradual variation of the starch cake resistance coefficients. The specific resistance of filtration was assumed to have a distribution along the thickness of the cake and to vary with the progression of constant pressure filtration operation. It was assumed to have its maximum in the initially formed cake on the surface of the filter medium, and its minimum in the newly formed cake on the surface of the already deposited cake. The result was an empirical equation which needed several experimental parameters. Takai *et al*., (1987), filtered 10 w-% sweet potato, maize and wheat starch slurry, and 20 w-% potato starch slurry at pressures between 0.19 and 1.15 bar and found that under these conditions the starch filtration was accompanied by sedimentation. They developed an equation expressing the filtration of starches in the presence of sedimentation by modifying Ruth's (1935) equation. The same type of equation was also developed by Bockstal *et al*., (1985). However, usually the sedimentation can be neglected if the settling velocity is very slow. Lately, Mayer (2000) has generally emphasised the importance of Tiller's empirical theories and especially their suitability for many industrial applications too.

Also the Kozeny-Carman equation is used in filtration calculations, because it relates permeability of the cake to the porosity and the specific surface area of the cake (Grace, 1953a, Satoh, 1957). However, its use is limited, because it cannot be used for

compressible cakes (Grace, 1953a, Tiller, 1974). In 1957 Satoh assumed a gradual increase or decrease of the resistances with the thickness of the cake for the progressive filtration of starches. He did not obtain satisfactory results when he applied the Kozeny-Carman equation to the filtration of starches.

Commonly, the classical filtration equation is a basis of most filtration equations and models. It is presented in many forms, of which one of the simplest and the most suitable for analysis purposes is the so-called general resistance form (Tiller, 2002/1970, Leu and Tiller, 1983, Oja, 1996)

$$\frac{\Delta p}{\mu q} = \alpha_{av} m_c + R_m \tag{3.1}$$

where Δp is the pressure difference, μ the viscosity of filtrate, q the superficial flow rate, which is obtained from the volume vs. time data according to Tiller (2002/1970), α_{av} the average specific resistance of the filter cake, m_c the mass of dry filter cake per unit area, and R_m the filter medium resistance. The term on the left-hand side represents the instantaneous total resistance of the filtration and the terms on right-hand side are the cake and the medium resistance, respectively. This classical filtration equation may be used under different process conditions and it can be applied to any kind of cake filtration: constant rate or pressure, as well as variable pressure or flow rate.

Constant pressure filtration is the most common method for collecting experimental filtration data. For data interpretation Eq. (3.1) is rearranged and presented in the differential form (Grace, 1953b, Svarovsky, 2000)

$$\frac{dt}{dV} = \frac{\mu c \alpha_{av}}{A^2 \Delta p} V + \frac{\mu R_m}{A \Delta p} \tag{3.2}$$

where t is time, V the volume of filtrate, c apparent filtration concentration, and A the filtration area. The reliability of the experimental data is easily controlled by plotting dt/dV or t/V versus V. If the plot is not a straight line, it indicates that the equation is not suitable for that case, and that there might happen to be, for example, sedimentation or pressure changes in the process (Sedin, 2003b).

Equation (3.2) can be integrated with the assumption that the concentration, the average specific cake resistance, and the filter medium resistance are constant after a certain start-up period t_s. It is recommended to ignore this in the initial stage of the calculations, because often high initial flow rates through a clean medium cause penetration of solids through the medium and the assumption of a constant filter medium resistance do not hold true during this period (Svarovsky, 2000). After the start-up period the pressure difference over the cake remains constant and the following equation holds true:

$$\frac{t-t_s}{V-V_s}=\frac{\mu c \alpha_{av}}{2A^2\Delta p}\left(V+V_s\right)+\frac{\mu R_m}{A\Delta p} \tag{3.3}$$

where t_s is the time, where the filtration pressure over the cake has reached a constant value, and V_s is the volume of filtrate collected at time t_s. The apparent filtration concentration, c, is defined as the mass of dry filter cake per unit volume of filtrate. It can be calculated from the measured mass of dry cake, and from the known volume of filtrate collected at the end of the filtration, or from the equation (Wakeman & Tarleton, 1999)

$$c=\frac{s\rho}{\left(1-m_{av}s\right)} \tag{3.4}$$

where s is the mass ratio of dry solids to slurry, ρ the density of the filtrate, and m_{av} the mass of wet cake per unit mass of dry cake.

3.2.1 Determination of α_{av} and R_m

The essential solid-liquid separation parameters of the filtration resistance (or permeability) and porosity must be obtained for every material. It is critical for solid-liquid separation system success (Tiller, 1974, Tiller & Crump, 1977, Mayer, 2000). The average specific cake resistance, α_{av}, and the filter medium resistance, R_m, can be measured by plotting $(t\text{-}t_s)/(V\text{-}V_s)$ against $(V+V_s)$ according to Equation (3.3). α_{av} is measured from the slope of the curve and R_m from the intercept between the curve and y-axis. During the filtration period the curve should give a straight line. Non-linearities can often be seen towards the end of filtration, where the curve starts to rise sharply. The reason is that the filter chamber becomes full of cake (Wakeman & Tarleton, 1999). The applicability of the determination of α_{av} is tested for example for two industrial

sludges of extreme filterability conditions by Bartolomeu *et al*., (1989) and proved to be suitable for both. The slurries are introduced in Table 3.1.

Table 3.1 The two industrial sludges and their properties tested by Bartolomeu *et al*., (1989).

Slurry	Origin	Average particle size, μm	Specific resistance, m/kg
Easy-to-filter one	sludge from the scrubber of a blast furnace	13.7 μm	At 0.25 bar: 1.9×10^{10} At 0.75 bar: 4.8×10^{10}
Slowly-filtrable one	sludge from the pretreatment of electroplating wastewater	15.8 μm	At 1 bar: 8.4×10^{12} At 3 bar: 1.4×10^{13}

The determination of R_m is considered more precisely in Chapter 4.

3.2.2 Compactibility

The average specific resistance of the filter cake, α_{av}, describes the ability of the cake to resist the filtrate or the wash water flow in the cake at a certain pressure. For incompactible, or also called incompressible, cakes α_{av} is constant and independent of filtration pressure under certain operating conditions. However, most cakes are compactible, or compressible, to some degree, and hence their average filtration resistance increases when higher filtration pressures are employed. On a compactible filter cake, the increase in filtration pressure primarily affects the cake layer near the medium and leaves the remainder essentially unchanged. This compaction of the lower layer affects the deliquoring of the whole cake. Because of such cases, in the filtration calculations α_{av} is assumed to be the average value at a certain pressure (Shirato *et al*., 1986b).

In general, compactibility of the filter cake is the key mechanism in the deliquorability analysis. The cakes are classified according to their compactibility coefficient, n, as follows (Tiller & Yeh, 1987, Leu *et al*., 1993, Sörensen *et al*., 1996):

1. Incompactible materials, $n=0$
2. Slightly compactible materials, $0 < n < 0.5$
3. Moderately compactible materials, $0.5 < n < 1$
4. Highly compactible materials, $n > 1$
5. Extremely compactible materials, $n >> 1$, (Sörensen *et al*., 1996)

In order to evaluate the separation characteristics of a cake, the effect of applied stress on the specific resistance must be known. When the average specific cake resistances are measured at different pressures, the compactibility coefficient for the material is obtained from the following equation (Tiller & Yeh, 1987, Leu *et al*., 1993, Svarovsky, 2000):

$$\alpha_{av} = \alpha_0 (1-n)\left(\frac{\Delta p}{\Delta p_0}\right)^n \tag{3.5}$$

where α_0 is the local specific cake resistance at unit applied pressure (when the pressure scaling pressure is Δp_0), n compactibility coefficient, and Δp_0 scaling pressure for local specific cake resistance. The equation is valid when the pressure drop across the cake is 1…7 bar and the material is slightly compactible, n<0.5 (Leu *et al*., 1993). When the values of α_{av} are plotted against the filtration pressure on a logarithmic scale, the compactibility coefficient, n, can be measured from the slope of this curve and the local specific cake resistance, α_0, from the intercept between the curve and y-axis. This power-law type constitutive equation is closely discussed by Lee & Wang, (2000).

Another way is to use a single test run for obtaining such test data. During the run the pressure is stepped up from one operating pressure to another, and then held constant until the next increase, without interrupting the flow. At each pressure step measurement of the cumulative volume of the filtrate is started from zero again. Δp is considered to be the dependent variable (Rushton *et al*., 1996)

$$\Delta p \frac{t}{V} = \left(\frac{\mu \alpha_{av} c}{2A^2}\right) V + \frac{\mu R_m}{A} \tag{3.6}$$

Then $\Delta p(t/V)$ is plotted against V and the compressibility is checked from the slopes.

3.3 SUMMARY

This chapter reviewed the general pressure filtration theory, (Eq. 3.1 - 3.3), which was ascertained to be the basis of the starch pressure filtration process. The filtration stage was considered as the basis for the success of the whole starch dewatering process,

because it determines the structure of the starch filter cake. The structure, in turn, determines the success of the washing and compression dewatering stages.

In this chapter, emphasis was given to the measurement of the average filtration properties, although the measurement of the local filtration properties and the particle packing theory was also expressed. The most important parameter in starch filtration was shown to be the average specific filtration resistance of the cake (Eq. 3.3), which controls the success of the following stages in the filtration cycle. Additionally, a short overview on cake compaction theory and the classification of materials according to their compactibility was given.

4 FILTER MEDIA

The filter medium is the keystone of any filtration system. Its structure and resistance affects the formation and the structure of the filter cake, and therefore the progress of the whole filtration process. The choice of the medium is critical to the quality of the filtrate and the filter cake, as well as to the capacity of the filter.

4.1 STRUCTURE

The filter medium is the keystone of any filtration system. It is a porous, heterogeneous structure, where pores have a non-uniform size and are usually of irregular geometry. Its structure affects the way that the particles deposit and pack on the medium surface in cake filtration (Rushton *et al*., 1996, Rainer & Höflinger, 2002). The two basic filter medium types used in pressure filtration are woven and non-woven cloths. Woven cloths are divided into mono- and multifilament cloths depending on the types of yarns used in the weaving. Also the weave pattern and the thickness of the cloth may alter. In non-woven cloths the fibres are randomly oriented, and hence their permeability and the filtration characteristics are dependent only on the felt porosity and fibre diameter. Additionally, finishing of the cloth, for example calandering, affects considerably its open area, and hence its permeability (Järvinen, 2005).

4.2 RESISTANCE

Usually the total resistance to flow in filtration has been broken into cake resistance and medium resistance, as shown in Chapter 3.2, Equations (3.1)-(3.3). Only the total resistance is measured and assumed that the medium resistance remains constant and the average cake resistance is a function of the solid load.

The filter medium resistance, R_m, can be measured from the plot of $(t\text{-}t_s)/(V\text{-}V_s)$ versus $(V+V_s)$ according to Equation (3.3). Thus R_m is measured by extrapolating from a straight line, and obtaining the second term of Equation (3.3.), which represents the

intercept between the line and the y-axis. However, it is a common problem in the pressure range from 0.3 bar to 7 bar, that the plot gives negative values for R_m. This indicates that there is an additional driving force for the filtration other than filtration pressure. This problem was faced also by Satoh (1957), who filtered starches. He resolved the problem by using only the first few filtration data points in the extrapolation and hence obtained satisfactory values for R_m. However, a more reliable method is to calculate R_m by applying Darcy's law to the initial stage of the filtration or to the flow of liquid through the clean cloth. Then the R_m is calculated (Rushton *et al.*, 1996, Salmela & Oja, 2004)

$$R_m = \frac{t\,A(\Delta p - \Delta p_m)}{\mu\, V} \tag{4.1}$$

where Δp_m is the pressure below the filter medium.

The assumption of the constancy of the filter medium resistance holds true, if the medium is chosen to resist blinding and that is proved with over thousand filtration tests. If the cloth is selected properly, the filtrate flow curves are identical from cycle to cycle over many months of operation, in another words, t/V vs. V plots do not change. (Mayer, 2000)

However, Leu & Tiller (1983) and Chase *et al*. (1994) have proved that as a matter of fact the medium resistance increases gradually with time and the mass of dry cake per unit area due to migration of fine particles into the interstices of a medium. The higher the applied pressure, the more easily the penetration occurs (Endo & Alonso, 2001). Larger particles can also block off the entrance to the pores and thus divert the fluid flow to other pores, or the stress of the cake may compress the fibres in the medium.

In any case of high levels of specific cake resistance ($\alpha_{av} > 1\times10^{12}$ m/kg) changes in the medium resistance, R_m, have little influence on the overall productivity. A partially clogged medium may still function quite satisfactorily in a system controlled by α_{av}. Rushton *et al*. (1996) have represented that this concept of negligible medium resistance leads to simple process equations, and it is justified when

$$\frac{\alpha_{av}\, w}{2AR_m} > 20 \qquad (4.2)$$

where w is the total mass of solids deposited. Another criterion for the concept of negligible medium resistance is that the clean filter medium resistance, R_m, should be less that 10% of the specific resistance of the filter cake, α_{av} (Rushton *et al*., 1996).

4.3 SELECTION

For the filter to give an optimum performance the medium used must be the best one available for the purpose. The change of the filter medium can perhaps significantly alter the economics of the filtration process. The selection of the filter medium must take into account the permeability of the clean and of the used medium, and its particle retention capability.

Practically no quantitative data exist which would facilitate the selection of the most suitable cloth for the separation of a particular solid-liquid mixture (Rushton & Griffiths, 1971/2004).The suitability of a medium must be decided by experiments or by previous practical experience. Rushton & Griffiths (1971/2004) have pointed out that it would be convenient if many cloth types could be characterised by an easily measured parameter, and further, if the latter could be linked to relevant measurable particle properties. They reported about many studies concerning the efforts to measure the overall permeability of the clean and used monofilament or multifilament cloths. However, the problem of the prediction of the overall permeability using easily measured cloth properties such as cloth, yarn and fibre densities has not yet been completely resolved.

Satoh (1957) used the ratio between the filter medium resistance and the filter cake resistance, ψ, for the selection of the filter medium for starch filtration applications. The ratio was calculated as follows:

$$\psi = \frac{R_m}{\alpha_{av}(1-\varepsilon_{av})\rho_s} \qquad (4.3)$$

where ε_{av} is the average porosity of the cake and ρ_s the density of dry solids. This means that the resistance of the medium, R_m, is proportional to the average specific cake resistance, α_{av}, even though the cake is compressible and even if the cake is somewhat different in its particle size. In the case of a suitably selected filter medium, the value of ψ was less than one millimetre for starch filtration applications by Satoh (1957). In addition to this ratio, Satoh (1957) used the microscopic investigation of the cloth for the selection of a suitable filter medium.

4.4 SUMMARY

This chapter gave a short overview of the choice of the filter medium for starch filtration applications. The concept of the filter medium resistance and its evaluation was introduced, and the criteria for neglecting the filter medium resistance in filtration calculations were discussed (Eq's 4.2 and 4.3). Two experimental articles concerning the choice of the filter medium for the filtration of starch slurries were reviewed and a dependency between the starch particle size and the filter medium resistance in filtration applications was introduced.

5 CAKE WASHING STAGE

Traditionally starch is washed in a conventional centrifuge or in a vacuum filter. In a centrifuge the dilution washing principle is followed: The wash water is continuously brought over the cake, whereby the mother liquid in the cake is diluted until the desired result is produced. Also in a vacuum filter the water consumption is high, because the slurry has to be diluted before it can be filtered and washed.

In 1997, the Larox Group introduced a displacement washing method, in which after the filtration period or after the filtration and dewatering periods a certain amount of wash liquid is brought on the top of a filter cake in the filter press. Then it is pressed through the cake, physically replacing the mother liquid with the wash liquid, which further reduces the quantity of the solute retained within the interstices of the cake. The intention is to drive the mother liquid from the pores of the cake by plug flow displacing it with the wash liquid. This is only possible in those pores open to flow and holding 'free' liquid. In the cakes, which are formed from small and porous particles, the mother liquid is held between the particles and within them. They can be removed only by mass transfer processes, like diffusion and mixing, which often become the rate-determining stages in washing. For fine and porous particles also the average specific cake resistance is usually high. This is the challenge in cake washing and good design is necessary to optimise the process or even to make it work.

The advantages of cake washing in a filter press are high washing efficiency and low wash liquid requirements. Less filtrate has to be recycled and less wastewater treated. Also the fact that washing may be carried out in situ on an existing filter is a benefit. On the other hand, the disadvantages are possible cake cracking and possible holes in the filter medium. These holes lead to craters forming in the cake during cake formation and then the passage of the wash liquid through any such craters.

There does not exist any experimental data concerning starch cake washing, except the author's own (Paavola, 1998, Paavola & Oja, 1999, Salmela & Oja, 1999a, 2000, and 2002, 2005b, and 2006).

5.1 PRINCIPLES IN CAKE WASHING ANALYSIS

In the ideal case for displacement washing only one void volume of wash liquid is required for 100% recovery of the residual filtrate, because true plug flow through the cake is obtained. In the real case, the pores are not uniform, the structure of the cake is heterogeneous and longitudinal mixing with diffusion takes place. In practice, only 30 to 86% of the retained filtrate is removed by displacement washing (Choudhury & Dahlstrom, 1957, Wakeman, 1980). A common way of describing the cake washing performance is to draw a washing curve (Figure 5.1), which is a plot of the wash result versus the washing time, t_w, the wash ratio, W_R, or the void volumes of wash liquid used, W. Lately, Mayer (2004) has emphasised the importance of relating the wash result to W_R. He has referred, among others, to the work of the author (Paavola & Oja, 1999) because the plot of the wash result versus the washing time can be misleading and the results from the different tests cannot always be compared. Lately, it has been shown by the author that the different series from the measurement of the starch cake washings are easily compared when the washing results are plotted against the wash ratio (Salmela & Oja, 2006). The effects of washing conditions and the height of the cake are excluded by using the wash ratio instead of the washing time, although the order and the usage of the process stages can be seen.

5.1.1 Washing Parameters

The wash result may be described by four alternative washing parameters, which are commonly used in monitoring and describing the washing performance. The wash results can be plotted as:

- *The fraction of solute removed from the cake*, F_s, (Wakeman, 1980, Wakeman & Tarleton, 1990 and 1999)

$$F_s = \int_0^{W_R} \frac{c_e - c_w}{c_0 - c_w} dW_R \tag{5.1}$$

where c_e, c_w and c_0 are the solute concentration in the wash effluent, in the wash water feed, and, in the retained filtrate, respectively. Usually the concentration in the wash water feed is zero.

- *The fraction of solute retained in the cake*, R_s, which reveals the purity of the cake

$$R_s = 1 - F_s = 1 - \int_0^{W_R} \frac{c_e - c_w}{c_0 - c_w} dW_R \tag{5.2}$$

The value of F_s should approach one and the value of R_s zero asymptotically.

- The dimensionless instantaneous concentration of solute in the wash effluent, F.

- The dimensionless concentration of the solute in the wash effluent collected in the washings receiver vessel.

The wash ratio, W_R. In a washing curve, the wash result is plotted against the wash ratio, which describes the ratio between the volume of wash liquid and the mass of dry solids produced. It is calculated (Wakeman, 1975, Wakeman, 1998) as follows:

$$W_R = \frac{V_w}{V_{f0}} = \frac{q_w t_w}{S\varepsilon L} \tag{5.3}$$

where V_w is the volume of the wash liquid used, V_{f0} the volume of the residual filtrate retained in the cake at the beginning of the washing, q_w the average velocity of the wash water flow, t_w the washing time, S is the cake saturation, i.e. proportion to the void volume filled by liquid, ε the porosity of the cake, and L the cake thickness.

The void volume of wash liquid used, W, is sometimes used instead of the wash ratio. It is calculated (Wakeman, 1975, Wakeman, 1998) as follows:

$$W = \frac{V_w}{V_{void}} = \frac{q_w t_w}{\varepsilon L} \tag{5.4}$$

where V_{void} is the void volume of the cake. If the cake is fully saturated when the washing starts, the wash ratio is equivalent to the void volume of the wash liquid (W=W_R).

The use of the washing parameters is widely tested. Such parameters are found not to be always so useful in industry, where actual concentrations and volumes of the wash liquid used are of greater importance (Wakeman & Attwood, 1988). It is commonly known, that it is difficult to obtain the value of the experimental wash ratio with a high degree of accuracy, and it is almost impossible to obtain an accurate value from an operating filter (Wakeman & Tarleton, 1999). The importance of analysis of the cake solute content is emphasised: 'Whenever washing data are being obtained from plant or laboratory experiments it is important to measure also the solute concentration in the cake, even though the solute concentration in washing may have been measured carefully' (Wakeman & Tarleton, 1999).

5.1.2 Washing Curve

When at least one of the washing results is analysed, the cake washing performance can be described by a washing curve. One typical washing curve is shown in Figure 5.1.

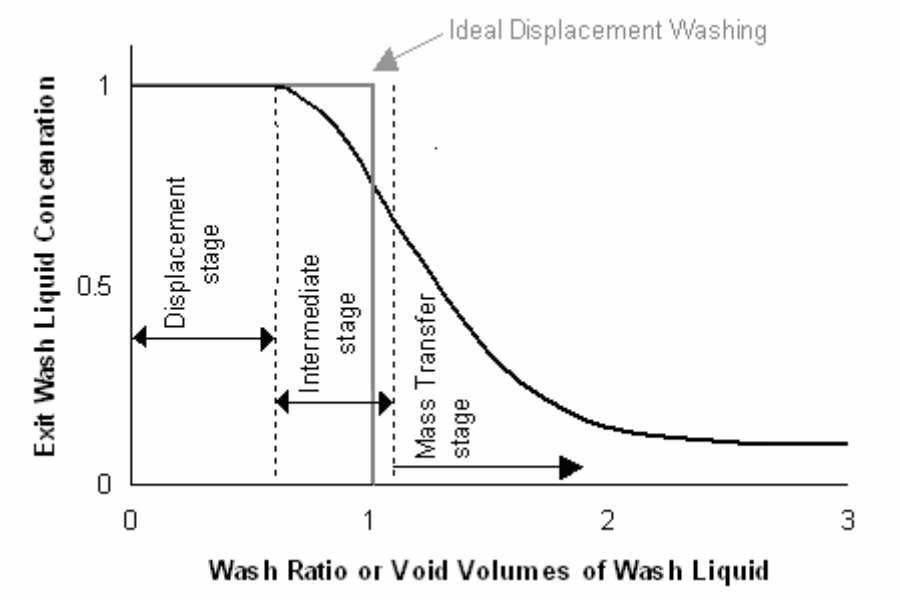

Figure 5.1 A typical experimental washing curve and an ideal displacement washing curve for a cake, which is fully saturated when the washing starts.

The washing curve can be divided into three regions (Wakeman, 1975):

1. *The hydraulic displacement stage*

   The exit wash liquid concentration remains constant and the wash liquid displaces the retained fluid. The efficiency of washing depends on the rate and mode of permeation of the wash liquid into pores of the filter cake, which in turns depends on the structure and geometry of the voids within the cake.

2. *The intermediate stage*

   An ideal mixing region where the exiting wash liquid concentration decays exponentially. Some pores are subjected to the displacement and others to the mass transfer mechanisms.

3. *The mass transfer stage*

   The region at low concentrations, where diffusion prevails and which is described by another exponential decay. The extent to which the mass transfer affects the washing process is reflected with 'a tail' on the washing curve.

The relative magnitude of each region depends on many factors. In general, regions one and two are more pronounced in highly saturated cakes, which is usually the case for compressible cakes. If the cake is dewatered by gas displacement prior to washing, it becomes unsaturated and its washing curve has a different shape than the washing curve for a saturated cake (Wakeman & Tarleton, 1990). In the dewatered cake, the mass transfer processes in any region of the cake voids are ineffective until the wash liquid has re-saturated that part of the cake. The mass transfer begins sooner in dewatered cakes than it does in fully saturated cakes, because there is little or no hydraulic displacement of the residual filtrate.

Also 'the tailing' of the washing curve is very common. Frequently the reason for that is the fine particulate cake. This 'tail' becomes important if a high degree of solute removal is required. Then the desired level of removal may never be reached or may only be reached after extremely long washing periods. (Wakeman & Rushton, 1976)

If the fraction of solute retained in the cake, R_s, is shown in the y-axis instead of the dimensionless instantaneous concentration of the solute in the wash effluent, F, the

displacement stage is not shown in the washing curve. However, the curve, which shows R_s against the wash ratio, W_R has usually a higher practical use than the normal washing curve relating F and W_R. It relates directly to the quality of the cake and gives more accurate results (Rahier & Hermia, 1988). It also detects the possible cake non-uniformities, which are the consequences of a non-ideal cake formation (Mayer, 2004).

In general, the shape of the washing curve depends on:

- The amount of dewatering done on the cake before washing
- The flow rate of wash liquid through the cake
- The mother liquid and wash liquid properties, such as viscosity and density
- The solids concentration in the feed before filtration
- Solute-to-solvent diffusivity
- Porosity, structure, initial saturation and homogeneity of the cake, and uniformity of its thickness.

The washing curve can be either measured experimentally or predicted using theoretical models. When the experimental curve is obtained, it is necessary to keep the cake properties constant and the same, as expected to prevail on the large-scale filter. In other words, the cake formation in the tests has to follow closely that of the actual filter.

5.2 WASH WATER FLOW RATE

When analysing the flow through the cake, a filter cake is thought to be a porous medium composing of an assemblage of particles through which a fluid may flow. An analogue between fluid flow through the capillaries of a regular cross-section and through the pores in a porous medium are commonly accepted. In theory, the wash water flow rate remains constant, since the resistances of both cake and filter medium are the results of preceding operations. The measure of the wash water flow rate through the cake is important, because it defines the mechanism, which is dominating during the cake washing process.

The first relation describing the average velocity of the flow of liquid through the particulate bed, q_w, is known as Darcy's law. It has the form (Wakeman, 1975, Coulson *et al.*, 1991)

$$q_w = K\frac{\Delta p}{L} \tag{5.5}$$

where K is a constant depending on the physical properties of the bed and fluid, and L the thickness of the bed.

In 1916 Sperry measured the flow rate through the cake in a small filter press with horizontal filter medium. In his experiments, the cake was first deposited by filtration and then the wash water was allowed to flow through the cake. The pressure was kept constant during the filtration and the washing stage in each test, but altered between the test runs. The volumetric interstitial flow velocity of the wash water through a straight capillary in a cake, Q_w, was assumed to be laminar, and it was calculated from Poiseuille's equation (Sperry, 1916, Choudhury & Dahlstrom, 1957, Coulson *et al.*, 1991)

$$Q_w = \frac{\pi r^4}{8l}\frac{\Delta p}{\mu_w} \tag{5.6}$$

where r is the inside radius of the pore, Δp the pressure difference over the system, l the length of the pore and μ_w the viscosity of wash liquid. This equation (5.6) can be used for filter cakes, as the thickness of the cake corresponds to the length of the pore l.

Since Darcy's law uses only the single parameter K for describing the characteristics of a filter cake, the Kozeny's equation, which is based on the Poiseulle's equation, is developed in order to relate these parameters to its geometric considerations. It has the form (Coulson *et al.*, 1991, Wakeman & Tarleton, 1999)

$$q_w = \frac{\varepsilon^3}{K'\mu_w S_0^2(1-\varepsilon)^2}\frac{\Delta p}{L} \tag{5.7}$$

where ε is the porosity of the bed, K' is Kozeny's constant, and S_0 the specific surface of the particles. It is commonly accepted that K' has a value of five.

Kozeny's equation can be also used for determining the specific surface of powder samples from permeability data, and in correlation with resistance data for fluid flow through porous media. However, for particles that deviate strongly from a spherical shape, for wide particle size distributions and for most consolidated media, the equation is often not valid. The reasons for that are the assumption of the circularity of the capillaries, the existence of flow channels of different diameters, the varying cross-sections of capillaries along their length, and the neglect of the energy losses by viscous dissipation (Wakeman & Tarleton, 1999). Also some other reasons may affect the flow rate. It is for example reported by Marecek & Novotny (1980), that if the viscosities of the filtrate and wash water are markedly different, the flow rate of the wash water will be rather unstable during the displacement stage of the washing.

5.3 THE EFFECT OF OPERATING VARIABLES ON WASHING

5.3.1 Cake Filling

In a horizontal filter press, it is necessary to maintain a fraction of the filter chamber volume free of cake after the filtration stage. If the chamber is full of cake, channelling of the wash water occurs, and hence washing is not uniform and, its efficiency is poor. According to Rahier & Hermia (1988) the best filling degree for the chamber is up to 85-90%.

Sometimes there will be some remaining slurry in the filter chamber when washing starts. Then the wash liquid will be mixed to some extent with the remaining slurry (especially if the single inlet mode is used), which will penetrate into the cake, where it will be richer in solute than in the case of the pure solvent wash. This obviously reduces washing efficiency. In the mean time, the cake thickness will increase because of the additional solid deposition. This situation can be described with two distinct cake filtrations (Rahier & Hermia, 1988).

5.3.2 Cake Porosity

The porosity of the filter cake affects the liquid volume in the cake at the start of the washing as well as the volumetric flow rate of the wash water through the cake. When the porosity is low, the wash water flow is slow but the washing effectiveness is very high, because there is time for mass transfer processes to occur. (Wakeman & Tarleton, 1999)

5.3.3 Wash Liquid Feed

According to Rhodes (1934) an increase in the flow rate of wash liquid will decrease the time of contact with the cake. At the same time the adhering films of liquid will be swept from the solid particles more effectively. The effects of these two factors will tend to neutralise each other.

Rahier & Hermia (1988) have found that in many cases it is advisable to stop the wash feed for a while at the end of the cycle. Then the wash liquid will have time to absorb a fraction of solute by molecular diffusion. This procedure may be repeated many times to enhance washing efficiency. This stop-start washing can reduce the amount of wash liquid used in the situations where cake cracking cannot be prevented, where the solid particles absorb significant quantities of the solute, or where mal-distribution of the wash liquid may be unavoidable, such as in some filter presses.

5.3.4 Cake Thickness and Wash Liquid Distribution

Thicker cakes can be washed more effectively, but thinner cakes are required for high cake product quality and capacity. A compromise has to be struck in practice to find a clear priority. If the cake is less than 30 mm, the thickness has a major influence on washing effectiveness. For cakes less than 20 mm, both the thickness and the pressure difference are important. (Wakeman, 1998, Svarovsky, 2000)

Washing efficiency depends also on the uniform distribution of the wash liquid into the cake. If the filter cake is thin, the wash liquid tends to channel and slight uniformities in the cake thickness have a great effect on the distribution of wash liquid over the cake surface. Channelling can be avoided if the cake thickness is 200 times larger than the mean particle diameter, but good efficiencies are also obtained though this criterion is not observed. The experimental results are less producible as the ratio of the cake thickness to particle size is decreased. (Rahier & Hermia, 1988)

If the cake has a heterogeneous structure, caused by sedimentation of particles, this distribution of particle sizes affects the accessibility of some pores. Then channelling is probable. This can be avoided by a combination of higher slurry feed rate and good inlet port design and position. Another method to reduce cake heterogeneity is to perform a gentle compression prior to washing.

5.3.5 Viscosities of Liquids

The viscosities of the liquids in both cake formation and the washing stage have a considerable effect on the final concentration of the solute. The efficiency of the washing decreases as the ratio between the viscosity of the mother liquid and the wash liquid increases. If these viscosities are equal, the washing curve fits the dispersion model (Carleton & Taylour, 1991). According to Wakeman (1975) it is preferable to choose a wash liquid with high viscosity and low density relative to the respective properties of the residual filtrate.

Also the effect of temperature has to be taken into account. It is necessary to maintain the optimum temperature of both the suspension and wash water as accurate as possible in order to keep the viscosities constant (Marecek & Novotny, 1980). If the wash liquid is heated, it reduces in viscosity and hence results in higher local velocities in the pores of the cake. This may not assist cake washing, but the increased rates of mass transfer will. (Wakeman & Tarleton, 1999)

5.3.6 Cake Dewatering before Washing

Compression Dewatering. The cake can be dewatered by compression prior to washing. Compression reduces markedly the amount of solute in the cake, but the cake saturation remains constant. A shorter time and less wash liquid are required to achieve a given amount of solute removal from compressed cakes, but a considerably greater pressure is necessary to drive wash liquid through the pores. If the same applied pressure is used for compressed and non-compressed cakes, the wash liquid flow through the compressed cake is considerably smaller.

Displacement Dewatering. If the residual filtrate inside the cake is displaced with gas (air), problems associated with the flow of two phases through a porous media will result. The method is effective only in cakes, which are formed from relatively large particles and have large internal pores (Wakeman & Rushton, 1976).

Rahier & Hermia (1988) have proved that displacement dewatering prior to washing can be advantageous, but the saturation should be reduced to below 50% to gain the advantage of a lower wash liquid consumption. Carleton & Taylour (1991) have found that displacement dewatering leads to very poor washing, because during dewatering the incoming fluid is air and this has a lower viscosity than the mother liquid. Then the efficiency of washing decreases as the initial saturation of the cake decreases.

In practice, cakes made of fine solids often crack on dewatering by displacement because of the air introduced into the cake causes high capillary stresses within the cake. In this case, the only way to avoid cake cracking is to start the wash with a fully saturated cake. This reduces washing efficiency a little, or the amount of wash liquid needed is a bit greater for the same efficiency, but the cracking is avoided. (Svarovsky, 2000).

5.4 MODELLING OF CAKE WASHING

Fasoli (1970) and Fasoli & Melli (1971) have categorised the cake washing mechanisms according to the wash water flow velocity and indicated that several of the mechanisms may act simultaneously or at any time a single mechanism may take a dominant role. During the early stages of washing the mass transfer from the uniform film of filtrate along the flow channels in the cake is assumed to occurs, while during the later stages the diffusional mechanisms prevail (Wakeman, 1975). The categorisation of the mechanisms is shown in Table 5.1 and in Figure 5.2 according to Fasoli (1970) and Fasoli & Melli (1971).

Table 5.1 Categorisation of washing mechanisms according to wash liquid velocity. The categorisation is only applicable to washing of saturated cakes.

Wash Liquid Velocity	Dominant Washing Mechanism
0 - 8 μm/s	Axial diffusion (uniform filtrate film)
8 μm/s - 0.2 mm/s	Dispersion (uniform filtrate film)
0.2 mm/s - 1 mm/s	Side-channel mass transfer (uniform filtrate film)
1 mm/s - 3 mm/s	Mixing, (exponential decay)
3 mm/s -	Side-channel diffusion

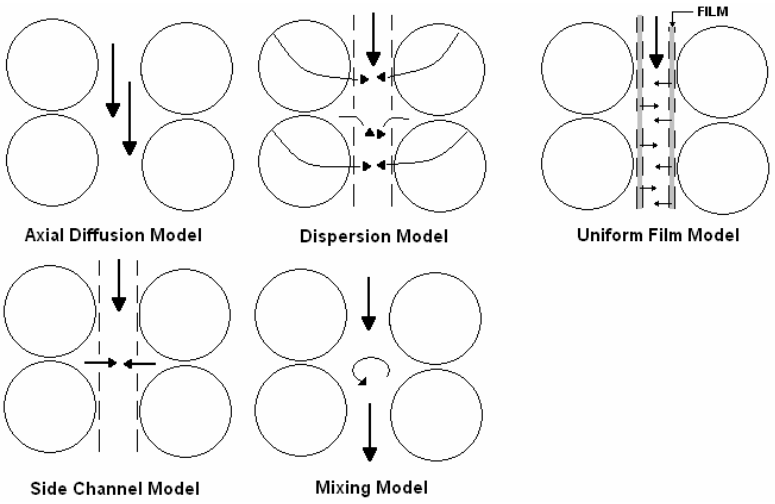

Figure 5.2 The principles of the washing mechanisms.

5.4.1 Dispersion model

Since the 1980's, the whole washing curve is commonly regarded as one: so-called dispersion model. It is found to be capable of replacing all previous washing models (Fasoli & Melli, 1971, Wakeman, 1980, Rahier & Hermia, 1988, Wakeman & Attwood, 1988, Hermia & Rahier, 1990, Wakeman & Tarleton, 1990, Wakeman & Attwood, 1990, Carleton & Taylour, 1991, Rushton *et al.*, 1996, Wakeman & Tarleton, 1999, Öhman & Theliander, 2002). The dispersion model is straightforward in its application and does not require laborious determination of arbitrary parameters before it can be applied. It is also versatile, easy to use and basic experimental test data only is required.

The dispersion model appears to fit many experimental data for the washing of saturated cakes. Even a simplified version of the model is applicable to washing fully saturated cakes containing non-porous solids at relatively high washing rates. Fasoli & Melli (1971) have claimed that the dispersion is the predominating mechanisms when wash liquid velocity is below 0.2 mm/s.

The model is based on the combined effects of molecular diffusion and mixing in the flow pores. These effects lead to dispersion of fluid, which is flowing through the cake. This, in turn, causes considerable deviations from plug flow. The basic assumptions underlying the application of the dispersion model are (Rahier & Hermia, 1988, Wakeman & Attwood, 1990):

- The structure of the cake is homogenous
- The cake porosity is uniform through the thickness of the cake (average cake porosity)
- The cake does not compress further during the passage of wash liquid through its interstices
- The dissolved substances are uniformly distributed throughout the volume of the cake
- There are no stagnant zones in the cake
- Solute adsorption on the solid phase does not appear.

Dispersion Model Equation. Wakeman (1980) presented the equation for the dispersion model in the form from which the concentration of dissolved material in the wash effluent can be calculated

$$\frac{c_e - c_w}{c_0 - c_w} = 1 - \tfrac{1}{2}\left[erfc\left(\frac{1-W_R}{2\sqrt{W_R}}\sqrt{D_n}\right) + \exp D_n\, erfc\left(\frac{1+W_R}{2\sqrt{W_R}}\sqrt{D_n}\right)\right] \tag{5.8}$$

where D_n is the dispersion parameter. It is assumed in the model that in the beginning of washing the exit wash liquid concentration equals the solute concentration in the mother liquid (c_e=c_0), and after a long period of washing, the exit wash liquid concentration equals the solute concentration in the wash liquid (c_e=c_w). The equation (5.8) is only valid for saturated cakes, but in practice it also gives a good estimate when applied to dewatered cakes during the later stages of washing, that is, for wash ratios greater than one. The model is tested with a variety of solids, like calcite, flocculated calcite, talc, magnesium carbonate, china clay, sodiumbicarbonate, fiberglass, ballotini, couple of filter aids, and their mixtures by Wakeman & Attwood (1988 and 1990).

The dispersion parameter, D_n, characterises the shape of the washing curve and describes the effectiveness of washing: the higher the number, the more effective the washing. It can be considered in terms of the Reynolds number for wash liquid flow through the cake, and the Schmidt number describing the ratio of the molecular diffusivity of the momentum to the molecular diffusivity of the mass. Hence (Wakeman, 1980)

$$D_n = \frac{q_w L}{D_L} = \mathrm{Re}\, Sc\left(\frac{L}{d_p}\right)\left(\frac{D}{D_L}\right) = \left(\frac{\rho_w q_w d_p}{\mu_w}\right)\left(\frac{\mu_w}{\rho_w D}\right)\left(\frac{L}{d_p}\right)\left(\frac{D}{D_L}\right) = \frac{q_w d_p}{D}\left(\frac{L}{d_p}\right)\left(\frac{D}{D_L}\right) \tag{5.9}$$

where D_L is the axial dispersion coefficient for the solute/solvent system, Re the Reynolds number, Sc the Schmidt number, d_p the particle diameter, D the binary molecular diffusion coefficient for solute, which is of the order of 10^{-9} m^2/s, and ρ_w the density of wash water.

The dispersion coefficient. In order to use the dispersion model it is necessary to predict the dispersion coefficient. Usually, as a starting point, the binary molecular diffusion coefficient for the solute in a pure solvent, D, is taken from the literature. It is worth of noticing, that the presence of dissolved species can have a significant effect on the pure liquid diffusion coefficient, especially if the solutes show strong thermodynamic interactions or when their concentrations are high. However, the value used is of lesser importance when estimating the washing performance on the industrial equipment. When the value of D is known, the value of the axial dispersion coefficient, D_L, can be calculated. The ratio of D/D_L is dependent on tortuosity of the cake. An increase in tortuosity generally corresponds to a decrease in the dispersion coefficient. Tortuosity factors of $\sqrt{2}$ have been found generally suitable for ReSc<1, hence

$$\frac{D_L}{D} = \frac{1}{\sqrt{2}} = 0.707 \tag{5.10}$$

For larger values of ReSc, when the particles are relatively large and regular and the cake thickness is greater than 10 cm bed, the dispersion coefficient is calculated

$$\frac{D_L}{D} = 0.707 + 1.75\,\mathrm{Re}\,Sc \qquad L>10 \text{ cm} \tag{5.11}$$

For smaller, irregular particles with a size distribution and a variety of different shapes, the current dispersion coefficient underestimates the diffusion and does not fit the 'tail' of the washing curve. Hence, for relatively thin cakes (< 10 cm) of fine particles (Wakeman & Attwood, 1988)

$$\frac{D_L}{D} = 0.707 + 55.5(\mathrm{Re}\,Sc)^{0.96} \quad L<10 \text{ cm} \tag{5.12}$$

More effective washing is achieved with small values of the axial dispersion coefficient, D_L, or thicker filter cakes. Small axial dispersion coefficients are obtained with cakes composed of small particles and washed with low wash velocities. At any applied pressure, lower wash velocities are naturally associated with finer particle cakes, because the specific resistance of such cakes is high. Hence, a more effective washing should be found with thick filter cakes of fine particles. (Wakeman & Tarleton, 1999)

Simplified dispersion model. In many practical cases a perfect displacement is assumed. Then the second term in Equation (5.8) becomes negligible and the simplified version of the dispersion model can be used for fully saturated cakes, which are washed at relatively high rates (Rahier & Hermia, 1988, Hermia & Rahier, 1990, Carleton & Taylour, 1991, Svarovsky, 2000).

The dispersion model chart. According to Wakeman (1980), Wakeman & Attwood (1988) and Wakeman & Tarleton (1999) the general solution to the Equation (5.8) can be shown in the form of charts, like Figure 5.3. These charts show the variation of solute concentration (as a dimensionless concentration of solute in washings, as a fraction of the solute removed from the cake, or as a fraction of the solute retained in the cake) with the usage of wash liquid. The charts make it relatively easy to estimate washings using the dispersion model, but great care must be taken in its application. Cake cracking, channelling and initial mal-distribution of the wash liquid can produce a major error.

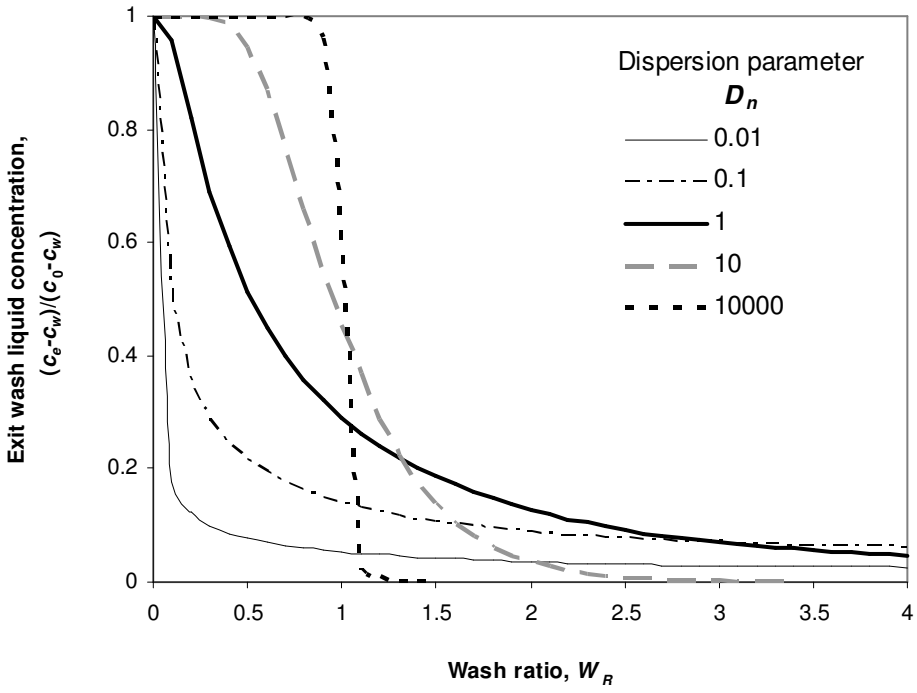

Figure 5.3 General solution to the dispersion model equation (5.8), when the cake is fully saturated prior to washing.

The shape of the curves is similar to those found in practice except that the practical curves do not fall so rapidly in the end. The reason is that the dispersion model does not take into account the 'tail' of the washing curve. It is not possible to achieve a pure step change in fluid consistency for the wash liquid feed even under well-controlled laboratory conditions. In industrial applications, disturbance of the cake surface and back diffusion of solute into the wash liquid on the cake surface will prevent the production of an ideal boundary condition. (Wakeman & Attwood, 1988)

5.4.2 Exponential Decay Model

Rhodes (1934) proposed an exponential decay equation to describe the way in which the concentration of solute material in exit wash liquid changes with time during the intermediate washing stage (Figure 5.1). The filtrate and the wash liquid were assumed to be mixed for the whole volume of the pores in the cake. During the initial period the wash water was assumed to displace directly a considerable amount of the mother liquor and the exit wash liquid concentration stayed about constant. In the beginning of the second period the exit wash liquid concentration started to decrease, and most of the remaining soluble material was carried out by diffusion. This second stage is described with the model, which assumes that the wash liquid and the residual filtrate are mixed in the entire pore volume of the cake (Rhodes, 1934, Marecek & Novotny, 1980). Hence, the concentration of solute in the exit wash liquid, c_e, is directly proportional to the concentration of the solute in the cake and that changes in the two are equal and opposite. The equation has the form (Rhodes, 1934, Wakeman, 1980)

$$c_e = c' \exp\left(-\frac{w_c Q_w t_w'}{AL} \right) \tag{5.13}$$

where c_e is the exit wash liquid concentration, c' the concentration of the wash water at the beginning of the second washing period, w_c the washing parameter, and t_w' the time from the beginning of the second washing period. If w_c is determined experimentally, the solute concentration at any instant of washing can be calculated. Also the effects of changes in the rate of wash water and in the cake thickness during the progress of

washing can be calculated. Theoretically w_c may vary with the rate of flow and with the cake thickness, but usually it is assumed to be constant. It is determined solely by the texture of the cake and the nature of the solvent and the solute. Rhodes (1934) has shown that a large variation in the rate of flow has almost no detectable influence on the value of w_c.

This model is only accurate for short washing times since it does not take into account diffusional processes within the capillaries and the various structures of the solid phase particles (Rushton *et al.*, 1996). However, for example Marecek & Novotny (1980) have applied it to longer washings (W_R up to 6) by dividing the washing into different stages and by establishing different values for the w_c-constant for different parts of the washing curve.

5.4.3 Other Washing Models

Also other washing models have been published, but very commonly the dispersion model is found to be capable of displacing these other models (Fasoli & Melli, 1971, Wakeman, 1980, Rahier & Hermia, 1988, Wakeman & Attwood, 1988, Hermia & Rahier, 1990, Wakeman & Tarleton, 1990, Wakeman & Attwood, 1990, Carleton & Taylour, 1991, Rushton *et al.*, 1996, Wakeman & Tarleton, 1999, Öhman & Theliander, 2002). However, some of them may be relevant to particular types of washing. The exponential decay model and the mixing model are simple and easy to use, but they may not be capable of describing the whole washing curve accurately enough. The uniform film model, the side-channel model and the diffusion model are accurate but difficult to apply, because they contain unknown parameters, which have to be obtained from experimental measurements before the models can be applied in practice.

Uniform film model (Wakeman, 1975). At low wash liquid velocities pure *axial diffusion* occurs. The pores in the filter cake form dead spaces and pockets, which are distributed along the lengths of the pores. The spaces are called side channels, and there is no bulk flow in or out of these channels. The pores have a stagnant film of filtrate remaining on their surface and the wash liquid only extracts the remaining solute from

this film by molecular diffusion. Plug flow in the channels, no sorption by the particles, and no axial dispersion are assumed. The mass transfer across the stagnant film boundary occurs at a rate proportional to the solute concentration difference. At slightly higher wash liquid velocities the *dispersion* of solute into the wash liquid also takes place. According to Wakeman (1975), the uniform film model can be used to describe the washing curve, when the ratio between the exit wash liquid concentration and the initial concentration of filtrate (c_e/c_0) is from 0.1 to one.

The side channel model (Wakeman, 1975) assumes that residual filtrate is contained in the blind side channels in the cake, when the wash liquid flows along straight channels without any axial mixing. The transfer of soluble material from the side channels starts when the wash liquid reaches the point of emergence of the side channel into the main channel. The transfer of solute is by molecular diffusion only, and the transport through the cake by plug flow. The blind-side-channels are assumed to be wedge-shaped. The model does not take into account the porosity distribution along the depth of the cake, the porosity of the particles forming the cake, the possible change in the direction of the flow, the change in the cake structure due to wash water pressure higher than the filtration pressure, the possible mixing occurring at the liquid interface, or the possible flocculation or agglomeration of the particles in the cake. Wakeman (1975) has also shown that the solute concentration in wash effluent is lower in the case of cakes of angular particles, and that this side channel model can be used to describe the washing curve, when the ratio between the exit wash liquid concentration and the initial concentration of filtrate (c_e/c_0) is less than 0.1.

Mixing model (Wakeman, 1975). When the velocity further increases, the more mass transfer of solute from stagnant residual liquid held around the particle contact points into the flowing wash liquid occurs. In the region of mixing, the retained filtrate and the wash liquid get mixed. Choudhury & Dahlstrom (1957) have extended the exponential decay model equation (Eq. 5.13) to facilitate the calculation of the percentage of solute remaining in the cake after washing in which the mixing of the wash liquid and the filtrate is the controlling mechanism

$$R_s = (1-E)^{W_R} \tag{5.14}$$

where E is the percentage of the solute removed from the cake with a wash ratio of unity (washing efficiency), and W_R is the wash ratio actually used. Choudhury & Dahlstrom (1957) tested Equation (5.14) by washing Na_2O from 60 w-% aluminium trihydrate. They found the agreement between experimental results for a wash ratio less than 2.1 and experimental values for efficiency (E) from 35 to 86%. Equation (5.14) is useful for rough estimates of wash liquid requirements for relatively fast washing rates, where no more than W_R=2.1 is needed. Only one experimental value of R_s at W_R=1 is needed. For W_R>2.1 more sophisticated models that accounts for the effects of diffusion have to be used.

5.5 SUMMARY

The intention of this chapter was to provide several simple and reliable methods for monitoring the cake washing process, both experimentally and theoretically. The chapter introduced the basic cake washing theories and the methods for monitoring and analysing the washing stage in a filter press. The different ways for estimating the performance of the cake washing, like washing parameters (Eqs. 5.1-5.4) and washing curves (Fig. 5.1) were reviewed. Also the factors affecting the shape of the washing curve, and the operating variables affecting the washing performance, were introduced.

Whichever washing mechanism is applied, the modelling of the cake washing is based on the wash water flow through the cake. Hence, the equations for calculating the wash water flow rates were introduced (Eqs. 5.5-5.7). Since the 1980's, the dispersion model (Eq. 5.8) has been commonly regarded as the main washing model. It and the simple exponential decay model were introduced in detail. Also other models (uniform film model, side channel model, and mixing model) were shortly introduced.

6 CAKE DEWATERING STAGE

In cake dewatering, the filtrate trapped within the voids of filter cake, is displaced by applying de-saturating forces to the cake. Such forces can be mechanical or hydrodynamical relying on different cake properties for their relative success. In mechanical dewatering the cake is compressed in order to reduce its void volume and at the same time its moisture content, while it remains fully saturated. In hydrodynamical dewatering air, which is sucked or blown through the cake, displaces the retained filtrate in the cake. This leads to the simultaneous flow of two fluids through the cake and, hence, the saturation of the cake decreases.

Both dewatering methods can be applied to cakes with a high void volume. For finer particles the void passage is smaller and the pressure due to surface tension is significantly high. Hence, the displacement becomes increasingly difficult as the particle size decreases.

6.1 CAKE SATURATION

After the filtration or washing period the pores of the filter cake are filled with filtrate or wash liquid. When the fluid flow to the cake is stopped, the flow from the cake continues until a residual equilibrium saturation level is reached. It represents the retention of a quantity of fluid due to the influence of capillary forces. At that moment, the cake is said to be fully saturated, and its value of saturation, S, is unity. When the cake is dewatered by air displacement its fractional volume of voids occupied by liquid decreases. It is desirable to be able to predict the level of saturation in a filter cake, and the time required to reach that level. Saturation, S, is defined as (Rushton *et al.*, 1996, Wakeman & Tarleton, 1999)

$$S = \frac{V_l}{V_{void}} \tag{6.1}$$

where V_l is the volume of liquid in the pores of the cake, and V_{void} the void volume of the cake, which is calculated

$$V_{void} = \varepsilon_{av} AL \tag{6.2}$$

where ε_{av} is the average porosity of the cake, A the filtration area, and L the height of the filter cake, i.e. AL is the bulk volume of the cake. The average porosity of the cake, ε_{av}, is calculated as follows:

$$\varepsilon_{av} = 1 - \frac{m_s}{\rho_s AL} = \frac{\rho_s (1 - m_{av})}{\rho_s (1 - m_{av}) - \rho} \tag{6.3}$$

where m_s is the mass of dry solids in the cake, ρ_s the density of dry solids, m_{av} the mass of wet cake per unit mass of dry cake, and ρ the density of the filtrate.

6.2 COMPRESSION DEWATERING

The separation of a liquid from a solid/liquid mixture by compression is often referred to as expression. It often follows the dewatering by filtration and it is that part of the dewatering, in which the compression causes a sudden increase in hydraulic pressure throughout the slurry. The hydraulic pressure remains equal to the applied pressure until the particles become networked or form a cake. Then the further deliquoring is achieved by consolidation during which the hydraulic pressure decreases with time. This chapter concerns with the expression of a filter cake under a constant applied pressure, which is often the case in a filter press.

Since mechanical dewatering is usually much cheaper than any thermal method, expression is increasingly important in many chemical processes. In the case of starch processing, the application of thermal processes is even more restricted because of the gelatinisation property of starches and the formation of harmful acrylamides in heating (Alavi, 2003).

6.2.1 Principles

Filter cake compression dewatering was studied in 1906 by Hankel & Offenbach. Walker (1907) has cited them as follows: ‘When separating solids from liquids by

means of the filter press it is frequently found impossible to exert sufficient pressure upon the press-cake through the feed-channel, to free the cake from liquid. This difficulty can be met if one filtering face of each chamber is fitted with an elastic, waterproof membrane instead of the usual filtering medium. When the chamber has been filled under the pressure exerted by the pump, air or water is forced behind this membrane, and the pressure thus exerted transmitted to the cake. In this way the water content of the cake can be decreased from 85% of water as obtained by the ordinary filter press, to less than 50%. This increased efficiency is obtained, however, at the expense of one-half of the working surface of the press.'

Also nowadays, in most designs compression is achieved by inflating a diaphragm, which presses the loose freshly filtered cake towards the filter medium. These filters are called variable chamber presses or diaphragm filter presses. According to Shirato *et al.,* (1986b) the solid-liquid separation using a filter press fitted with a membrane-compression mechanism can be divided into three stages:

1. a primary stage at the filtration pressure (low pressure filtration),
2. a secondary stage, in which the residual slurry is filtered at the expression pressure, or if the chamber is clogged up with filter cake before the pressure changes, the cake is compressed by the filtration pressure,
3. a filter cake consolidation stage (high pressure filtration/consolidation), which is divided into primary and secondary consolidation.

Filtration-consolidation. If the solids volume fraction in the original slurry is low, the expression is made up from two basic processes: filtration and consolidation. In filtration, the pattern of filtrate flow changes after the filter chambers of the press are completely filled with slurry. The applied mechanical compression causes an increase in hydraulic pressure uniformly throughout the slurry and the resulting hydraulic pressure is equal to the applied mechanical pressure. The expression proceeds following the principles of filtration.

Primary consolidation. When the critical solid content is exceeded, the particles become networked or a cake is formed. At the end of the cake formation stage in any type of cake filter, the solids pressure at the cake surface varies from zero at the cake surface, to the full operating pressure at the cloth. This results in a corresponding

variation in the cake porosity, given in Figure 6.1 by Carleton & Salway (2004). Further dewatering is achieved by consolidation, during which the hydraulic pressure in the pores of the cake decreases with time. At the same time, the local porosity of the cake decreases and the applied pressure is transferred from the pore liquid to the compressed solid structure. If the slurry has a high initial solid content, the entire expression process may be achieved through consolidation. At the end of the compression stage in a diaphragm press, the pressure throughout the cake is equal to the applied operating pressure. The porosity is constant, and equal to the value corresponding to this pressure (Figure 6.1). (Wakeman & Tarleton, 1999, Carleton & Salway, 2004).

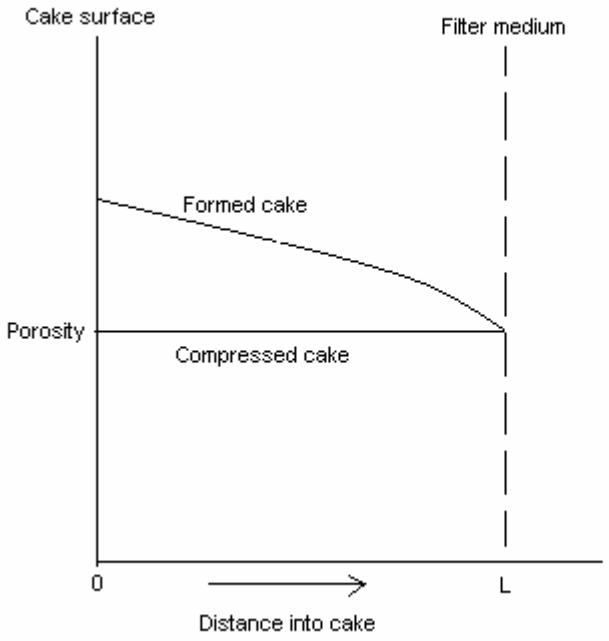

Figure 6.1 Porosity profile of the formed cake and compressed cake in a diaphragm filter press.

Secondary consolidation. At the end of the consolidation stage the hydraulic pressure is nearly equal to zero throughout most of the filter cake. Firstly, the larger voids of the cake are filled by particles moving into closer packed positions. Secondly, the voids, which are much smaller than the particles are filled by fragments of the original particles (Wakeman, 1975). In practical filter press operation, however, the movement of cake solids in the filter chambers are considerably retarded by the friction between

the cake solids and the filter medium. Thus, such a marked dewatering due to secondary consolidation is not expected in practice. (Shirato *et al*., 1986b).

Practical operations. According to Tiller & Yeh (1990), it is usually advantageous to change over to expression while there is still some slurry left in the filter chamber, so as to increase the average rate of filtration. If there is some slurry remaining in the filter chamber at the end of the low-pressure filtration, then in the expression, the residual slurry is being filtered at the higher pressure. At the same time the structure of the filter cake, which has been deposited during the low-pressure filtration, is changed under the effects of the higher pressure. After that the actual consolidation begins.

6.2.2 Experimental Evaluation

Experimental evaluation of the effects of compression on the filter cake can be carried out in a laboratory piston press filter (Wakeman & Tarleton, 1999, Oja & Martin, 1999 and 2000). Data are available from the piston press

- as the volume of filtrate collected and/or
- as the thickness of the solid/liquid mixture

expressed as a function of pressing time. When both data are available, they can be converted from one to the other and the preservation of the material balance can be checked. The thickness of the solid/liquid mixture in the cylinder at any time is calculated as follows:

$$L_{mix} = \frac{V_0 - V}{A} \tag{6.4}$$

where V_0 is the volume of slurry in the piston press at the start of the test and L_{mix} is the thickness of the solid/liquid mixture when a volume V of filtrate has been removed.

The transition point. Location of the point, t_{tr}, at which the filtration ends and the expression begins, can be difficult to detect even with a transparent cell. For many industrial situations, the exact location is unimportant, and only the average solidosity is needed (Tiller & Yeh, 1990). The equations describing filtration are applicable to the

initial stage of liquid removal. The transition from filtration to consolidation occurs when the hydraulic pressure begins to decrease and the solids content exceeds a critical value, which is unique for each mixture. At that transition moment the solid/liquid mixture has a thickness, L_{tr}, which can be calculated from (Shirato *et al.*, 1986a, 1986b, and 1987b, Oja & Martin, 1999, Wakeman & Tarleton, 1999)

$$L_{tr} = \left(\frac{m_{tr} - 1}{\rho} + \frac{1}{\rho_s} \right) \rho_s \omega_0 \tag{6.5}$$

where m_{tr} is the ratio of the mass of wet cake to the mass of dry cake at the end of the filtration stage and ω_0 is the total volume of solids per unit filtration area submitted to the compression. The value of m_{tr} cannot be measured experimentally during the normal expression test. Is has to be estimated from the known characteristics of constant pressure filtration. ω_0 is constant during a test and can be calculated as follows:

$$\omega_0 = \left(C_{av} L_0 \right)_{t=0} \tag{6.6}$$

where C_{av} is the solids volume fraction in the original slurry and L_0 the original thickness of the slurry. The solids volume fraction in the original slurry, C_{av}, can be calculated, when the mass fraction of solids in the initial slurry, s, is known

$$C_{av} = \frac{s\rho}{(1-s)\rho_s + s\rho} = 1 - \varepsilon_{av} \tag{6.7}$$

The transition between the filtration and consolidation mechanisms can be identified with many experimental methods:

1. The end point of filtration can be measured from the filtration equation by plotting *t/V* versus *V* curve. The filtration ends when the curve begins to rise strongly. In this method, the transition point is not always very clear. (Shirato *et al.*, 1987a)
2. Another way is to plot *dt/dV* against *V*. The end point is more easily identified in this plot. The filtration ends when a sudden and sharp increase in the slope of the graph appears. (Sedin *et al.*, 1997, Wakeman & Tarleton, 1999)
3. Oja & Martin (1999 and 2000) have shown that there is one easy way to determine the end point of filtration and that is to use the work cylinder pressure data given by the piston press test filter. The filtration ends, when the work cylinder pressure begins to rise.

4. The fourth method is to plot $-\Delta L/\Delta(t^{0.5})$ against the dewatering time, t, on a logarithmic scale. The plot is a straight line parallel to x-axis during constant pressure filtration. The transition point (t_{tr}, L_{tr}) is the point where the plot starts to decrease rapidly. From thereon the mixture is consolidated and the value of $-\Delta L/\Delta(t^{0.5})$ decreases. At the end of the consolidation it reaches zero, but this would take a long time. This method holds true when the original mixture is a slurry. (Shirato *et al*., 1986a, 1986b, 1987a and 1987b, Wakeman & Tarleton, 1999)

Only few studies concerning the determination of the transition from filtration to consolidation are published. For example Sedin (2003a) compared method one (t/V vs. V) with method two (dt/dV vs. V). In method one, it was assumed that the average specific filtration resistance has been stabilised immediately. In method two the transition point was measured as the time when the measured values deviated more than three standard deviations from the values calculated from the filtration equation. Sedin (2003a) proved that method two overestimated the time due to the fact that dt/dV is function of V and t that increases monotonously, according to the classical filtration equation. Method two described the data well when the coefficient of regression was near to unity for the experiments. Thus the standard deviation of the model was small, and therefore the error in the estimation of the transition point was also small.

Sedin & Theliander (2004) estimated the value of the transition point by methods two (dt/dV vs. V) and three (pressure data), and through the material balance of the solid material in the filter cell. In the material balance, the height of the filter cake was thought to have a maximum at the transition point and it was evaluated by the pressure transducers inside the filter chamber. The methods were compared by filtering kaolin slurry. The agreement between the methods was good: the estimations of the transition points gave approximately the same value. The difference was only a few percent. Methods two and three were found to be safer to use than the method, in which the height of the filter cake was calculated. Method three, using only the pressure data, was the most straightforward method as it did not require any calculations.

The consolidation ratio. The progress of the filter cake consolidation is easily followed using an empirical relationship for the consolidation ratio U_c (for example: Shirato *et al*. 1986b, and 1987b, Hermia & Rahier, 1990, Wakeman & Tarleton, 1999)

$$U_c = \frac{L_{tr} - L}{L_{tr} - L_\infty} \tag{6.8}$$

where L_∞ represent the final cake thickness.

6.2.3 Factors Affecting Expression

Suspension. The properties of the suspension determine the extent to which it can be expressed. Particle size, and hence the porosity of the filter cake, has a major effect on expression. Expression becomes slower as particle size is reduced, because the magnitude of intra-particle forces increases and the permeability of the mixture decreases. The compression dewatering is preferred prior to the displacement dewatering, when the particle size is less than 20 μm (Wakeman & Rushton, 1976).

pH. The effect of pH on the expression varies according to the surface properties of the particles. The surface charge is illustrated as the zeta-potential of the solid material. At low zeta-potential the particles tend to aggregate due to the low repulsive forces between them. This results in an open cake structure. The low filtration pressures are adequate and the expression can be used to further reduce the cake moisture content. When the zeta-potential is high, the repulsive forces between particles are higher and the cake structure is therefore open. The expression may be necessary to further dewater the cake. (Wakeman & Tarleton, 1999)

Sedin *et al*. (2002) studied the influence of pH on the dewatering of magnetite in a piston press filter. They found that the zeta-potential of particles did not influence the dewatering behaviour in the early stages of filtration, but it influenced the intermediate and final stages. Regardless of applied pressure, the average specific filtration resistance was found to have lower values at the iso-electric point compared to a zeta-potential of -40 mV. Häkkinen & Oja (2002) studied the filterability of colloidal TiO_2-particles which were coated with Al_2O_3. They found that the filterability can be improved by operating near the iso-electric point of the suspension.

Filtration pressure. The effect of filtration pressure on the average cake porosity is greatest at low pressures, between 0 and 3 bar (Wakeman, 1975).

Cake compactibility. More compactible materials are often also less permeable. Therefore, the cakes, which consolidate under hydraulic pressure and are difficult to filter, are the best candidates for mechanical compression. Compression also improves cake release, closes any cracks and tightens the cake, thus making any subsequent cake washings more effective (Svarovsky, 2000).

Skin effect. In filtration of extreme compressible slurries the formation of a skin above the filter medium may appear. This skin becomes limiting to liquid release from the cake during expression. If this happens, almost infinite time is needed to significantly increase the solid content during expression. On the other hand, this skin can be exploited, if the compression is in the reverse direction to filtration. Then the skin works as a mechanical piston, which compresses the cake, (Tiller & Horng, 1983, Shirato *et al*., 1987a, Sörensen *et al*., 1996). Rushton *et al*. (1996) have mentioned that in starch filtration a skin effect may appear. The formation of skin layer was detected also in the experimental part of this work, if the filtration pressure was too high. The cakes filtered at high pressure became very difficult to wash or to dewater by displacement. The wash water flow through these cakes was very slow, as well as the flow of filtrate during the compression stage.

Cake Washing. Also the following stages in the filtration cycle must be taken into account in the performance of the compression stage. If the cake is washed after compression, the wash liquid flow rate through the cake should be high enough to ensure an effective washing performance. If compression is too high it can produce a large increase in the specific cake resistance, and hence reduce the effectiveness of the cake washing. The wash liquid flow rate may become very low even when high pressure differences are employed (Wakeman & Rushton, 1976). The effect of compression before the washing step was discussed in Chapter 5.3.6.

6.2.4 Modelling of Consolidation

The models for the prediction of cake consolidation behaviour in pressure filtration may be classified (Sedin 2003b) as follows:

1. according to whether they incorporate secondary consolidation or not, i.e. Terzaghi-Voigt model vs. Terzaghi's model
2. according to the number of the parameters used in the models
3. based on non-linear or linear coefficients in the model, i.e. primary compactibility and the average specific cake resistance

In modelling, the consolidation is divided into primary and secondary consolidation. The primary consolidation begins, when the hydraulic pressure within the cake begins to decrease with time, and ends when the hydraulic pressure throughout the cake is zero. Then the secondary consolidation can further decrease the cake thickness and porosity. The primary consolidation is modelled by a Terzaghi element and the secondary consolidation by a Voigt element, (Sedin, 2003b). These Terzaghi and Terzaghi-Voigt models are based on Darcy's equation combined with the continuum equation, and most conveniently written in terms of a moving coordinate ω that represents the volume of solids per unit area between the plane and the filter medium. The models contain an assumption regarding the pressure-porosity profile at the transition between the cake build-up and the expression phase. They consist of a spring with its compression proportional to the force acting on the spring. The force represents pressure, and the compression of the spring represents the local void ratio in the expression model (Sedin, 2003b).

6.2.4.1 Terzaghi Model

Expression kinetics can be described by the Terzaghi model, which takes into account only the primary consolidation. In this model the local void ratio of compressed cake during the consolidation depends only on the local solids compressive pressure. The equation of the Terzaghi model has the form (Shirato *et al*., 1986a, and 1986b, Hermia & Rahier, 1990, Sedin *et al*. 1997 and 2002, Sedin 2003a and 2003b)

$$U_c = 1 - \exp\left(-\frac{\pi^2}{4} T_c\right) \tag{6.9}$$

where T_c is a dimensionless consolidation time, defined as

$$T_c = \frac{i^2 C_e t_c}{\omega_0^{\,2}} \tag{6.10}$$

where i is the number of drainage surfaces involved in the dewatering process, C_e the modified consolidation coefficient, and t_c the compression time. ω_0 represents the thickness of a cake, L, whose porosity is zero, and it is calculated as (Hermia & Rahier, 1990)

$$\omega_0 = L(1-\varepsilon) \tag{6.11}$$

6.2.4.2 Terzaghi-Voigt Model

The Terzaghi-Voigt model takes into account also the secondary consolidation, the so-called creep-effect. In this model the variation of the porosity in the filter cake is caused by the change in the local compressive pressure and the simultaneous effect of the creep of the solids. Hence the porosity is a function of the local compressive pressure and of compression time. For the constant pressure expression of filter cakes, the Terzaghi-Voigt model has two forms (Shirato *et al*., 1986a, 1986b and 1986c, Sedin *et al*., 1997, Wakeman & Tarleton, 1999, Sedin, 2003a and 2003b)

$$U_c = (1-B)\left[1 - \exp\left(-\frac{\pi^2}{4} T_c\right)\right] + B\left[1 - \exp(-\eta\, t_c)\right] \tag{6.12}$$

and

$$U_c = 1 - B\exp(-\eta\, t_c) \tag{6.13}$$

where B is the ratio between the liquid volume squeezed by the secondary consolidation and the total liquid volume squeezed from the mixture during the entire expression, and η is an empirical constant that characterises the creep of the material. These equations are called the Terzaghi-Voigh model with three parameters and the Terzaghi-Voigh model with two parameters, respectively. They are simplifications of the complete

Terzaghi-Voigt model, but they can be used with confidence, if the dewatering rate due to the secondary consolidation is much smaller than the rate due to the primary consolidation (Shirato et *al*., 1986c, Sedin *et al*. 1997). Hence, the three time constants are defined as

$$\theta_1 << \theta_3 \tag{6.14}$$

$$\theta_2 << \theta_3 \tag{6.15}$$

The first time constant, θ_1, represent the resistance due to liquid flow in Terzaghi's element. It is calculated as follows:

$$\theta_1 = \frac{4\omega_0^2}{\pi^2 C_e} \tag{6.16}$$

The second time constant, θ_2, is due to liquid flow resistance of the Voigt element

$$\theta_2 = \frac{4\omega_0^2 B}{\pi^2 C_e (1-B)} \tag{6.17}$$

and the third time constant, θ_3, represents the creep deformation of the solid material (Shirato *et al*., 1986c)

$$\theta_3 = \frac{1}{\eta} \tag{6.18}$$

Both parameters, B and η, in the model equations can be determined from the latter stages of a plot of ln(1-U_c) versus t_c (Shirato *et al*., 1986a and 1986b). After an initial stage of expression, ln(1-U_c) changes linearly with the expression time. The more compactible the cake is, the wider the linear regime will be (Lee & Wang, 2000). The proper methods for the determination of B and η for the consolidation of starches are introduced in Salmela & Oja (2005a).

6.2.4.3 Shirato Model

Terzaghi and Terzaghi-Voigt models are not useful tools in industry, since they include three empirical constants C_e, B and η, which have to be determined by two graphical

plots. Shirato *et al*. (1986b) represented the semi-empirical equation for the consolidation ratio as follows:

$$U_c = \frac{\sqrt{\frac{4T_c}{\pi}}}{\left[1+\left(\frac{4T_c}{\pi}\right)^{\upsilon}\right]^{1/2\upsilon}} \tag{6.19}$$

where υ is the consolidation behaviour index which takes the secondary consolidation effects into account. This model may be represented in the form of a chart (Shirato *et al.*, 1986a), as shown in Figure 6.2.

Consolidation ratio, U_c, is a straight line in the early stages of consolidation and C_e can be obtained from the slope of this line. According to Shirato *et al*. (1986b) the model, fits well for semi-solids, where U_c versus $T_c{}^{0.5}$ curve is a straight line in the early stage of expression. The fit in the early stages is not so good for the materials with a slurry origin, because the analytical solution of Terzaghi's and the Terzaghi-Voigt models and the experimental data for the constant-pressure expression of filter cakes represent an inverted S-shape curve. According to Shirato *et al*. (1986b) the difference in shape between the two expression curves may be due to the difference in the initial void distribution in the original materials to be expressed. As the expression proceeds, the difference between the two expression curves narrows and is not appreciable at the latter stage of the expression which is of practical importance in industry.

Shirato *et al.* (1986a) have proved that the maximum percentage error in the consolidation ratio, U_c, calculated by Equation (6.19) with υ=2.85 compared to the theoretical Terzaghi equation is only 0.60%. With smaller values of υ Equation (6.19) may give even more satisfactory values and the so-called secondary consolidation may be taken into account.

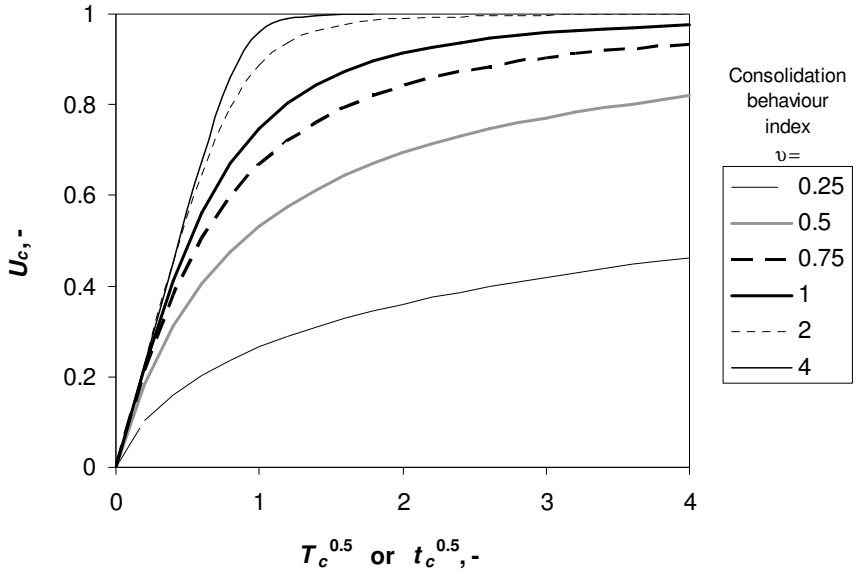

Figure 6.2 A plot of the consolidation ratio equation (6.19) showing the variation of the consolidation ratio U_c with the different values of the consolidation behaviour index υ.

6.3 DISPLACEMENT DEWATERING

In displacement dewatering, air-drying, the residual filtrate remaining in the cake is displaced with gas, usually air. The gas is forced to flow into the filter chamber and through the cake, and hence the problems associated with the flow of two phases through porous medium result. Each fluid has to be considered to establish its own tortuous pathways through the cake voids and each phase is though to also have their own associated pressure, viscosity and permeability. However, it is generally accepted that Darcy's law can be extended to describe the flow of each fluid flowing simultaneously (Wakeman & Tarleton, 1999).

6.3.1 Principles

Soon after airflow is started fingers of air begin to extend into the cake in the direction of liquid displacement. The liquid film separates these air fingers from the surface of the solid particles. When the air pressure is further increased, some of the air fingers become continuous fissures of air passing throughout the depth of the cake. When

finger breakthrough occurs, the effective drainage pressure is reduced, since the dewatering is less efficient. Finally, when the airflow has preceded long enough, the equilibrium between air pressure, surface and interfacial tensions, pore radii, and the pore liquid pressure is achieved. Not all of the fingers break through the cake, particularly if the cake contains very fine pores. (Wakeman, 1975)

The cake dewatering by air displacement can be explained by the capillary pressure diagram, which is shown in Figure 6.3. A certain pressure, called the threshold pressure, must be exceeded in order that air may enter the filter cake. So long as the pressure difference exceeds the threshold pressure some cake saturation reduction will result, until the irreducible saturation level, S_∞, is reached. It is the minimum moisture content, which can be achieved by air displacement at any pressure. When it is reached, the filtrate flow ceases and only air is flowing through the cake voids.

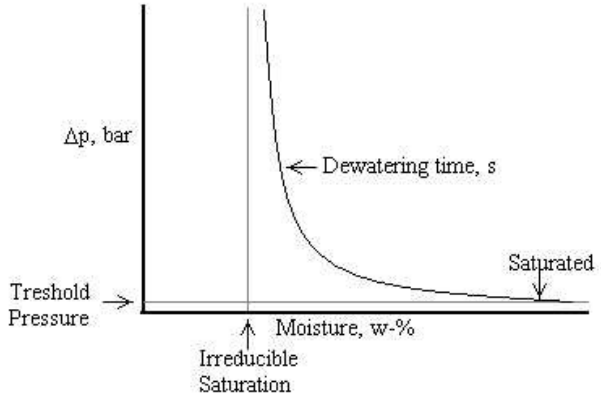

Figure 6.3 A typical capillary pressure diagram in filter cake displacement dewatering.

The capillary pressure diagram applies usually only to a certain cake thickness, but it can also be used for the cakes thinner than that thickness. The diagram is applicable to cakes with a relatively open structure, and it can be used in analysing the pressure filtration, where dewatering is accomplished by applying gas at high pressures to the cake surface. However, a combination of small particles, compression and compactibility of the cake leads usually to cakes, which can be extremely difficult to dewater by air displacement (Wakeman & Rushton, 1976).

The threshold pressure, p_t, can be calculated (Wakeman & Tarleton, 1990)

$$p_t = \frac{4.6(1-\varepsilon_{av})\sigma}{\varepsilon_{av} d_p} \tag{6.20}$$

where σ is the surface tension of the liquid to be removed from the pores of the cake, and d_p the mean size of the particles in the cake. The value of d_p most commonly used in deliquoring calculations is the surface-volume mean diameter related to the volume specific surface or the size related to the surface-volume mean diameter (Wakeman & Tarleton, 1999)

The reduced saturation, S_R, may be determined (Wakeman & Tarleton, 1999)

$$S_R = \frac{S - S_\infty}{1 - S_\infty} \tag{6.21}$$

where (S-S_∞) represents the part of the retained liquid, which can potentially be removed at any time during dewatering, and (1-S_∞) represents the total amount of potentially mobile liquid in the cake at the start of dewatering. The reduction of reduced saturation, S_R, can be calculated as a function of the dewatering time, and as a function of an increase in the air flow rate.

6.3.2 Factors Affecting Displacement Dewatering

The most important factors, which affect the displacement dewatering, are shown in Table 6.1 according to Wakeman (1975). Later, Wakeman & Tarleton (1999) have shown that the relative effects of these factors can be demonstrated by defining a 'base case'. The change in any of the factors can then be compared with this case.

The capillary retention forces in the pores of the filter cake depend primarily on the size and size range of particles forming the cake, and in the way how the particles are deposited during the formation of the cake. As the mean size of the particles decreases and the particle size range increases, a higher threshold pressure is needed in order to

displace the retained liquid. Commonly, the air displacement is effective only in cakes, which are formed of relatively large particles and hence have large internal pores (Wakeman & Rushton, 1976).

Table 6.1 Factors affecting the displacement dewatering.

Cake Properties	Fluid Properties	Interfacial Properties	Other Factors
Particle size	Density	Interfacial tension (liquid-liquid)	Temperature
Particle shape	Viscosity	Surface tension (gas-liquid)	Pressure gradient
Mode of particle packing			Rate of displacement
Dimensions of the cake			

The time needed to achieve a certain level of solid content can be shortened by increasing the pressure drop across the cake. As the pressure drop is increased, the liquid is forced from successively smaller pores, and hence the total fraction of voids containing stagnant fluid is reduced (Wakeman, 1975). On the other hand, if the dewatering time is kept constant, moisture content is reduced more at higher pressures, with a parallel increase in the cake production capacity (Svarovsky, 2000).

6.4 SUMMARY

This chapter reviewed two different filter cake dewatering theories: compression dewatering and displacement dewatering. The theories differed from each other because of the driving force of dewatering and the filter cake saturation during the dewatering process (Eq. 6.1). Compression dewatering was studied more extensively than the displacement dewatering, because the properties of the starch particles were found to be more appropriate to compression dewatering and it is very commonly applied in filter presses, while there exist no results concerning the compression of starch filter cakes.

First, the principles of compression dewatering were introduced. It was divided into primary and secondary consolidation, and the concept of the consolidation ratio was introduced (Eq. 6.8). Also the factors affecting the compression dewatering were shortly

presented. The emphasis was given to the measurement of the transition point, where the dominant mechanism was changed from filtration to consolidation, as well as to the modelling of the filter cake compression. The equations given for the modelling the compression dewatering stage were the Terzaghi model (Eq. 6.9), Terzaghi-Voigt models (Eq.'s 6.12 and 6.13), and the Shirato model (Eq. 6.19).

Finally, this chapter also gave a short overview of cake dewatering by air displacement. The capillary pressure diagram for the monitoring of the displacement dewatering was introduced (Fig. 6.3.), as well as the factors affecting the displacement dewatering.

7 EXPERIMENTAL PROCEDURE

This chapter introduces processing of the starch powders and starch slurries used in the experimental part of this work, as well as the identification of the starches used in the experiments. Additionally, the test filters and the experimental arrangements and procedure during the filtration tests and the comparability of the data between the two test filters are introduced.

7.1 TEST SLURRIES

Commercial native wheat, barley and potato starches were chosen to be the main starches studied in this work, because they represent the starch grades most commonly used in Finland, and they were readily available. Additionally, native maize starch was studied in order to show how it differs from the other studied starches.

The native starches, which were used in the experimental part of this study, were delivered to laboratory in the dry form, containing 10-20 w-% of absorbed water. The content of the absorbed water in each production lot was measured before preparation of the slurries by taking a sample of the starch powder, and weighing, drying and re-weighing it. The native starches were produced by Ciba Specialty Chemicals Oy (Anjalankoski, Finland), except that the native maize starch was produced by Tate & Lyle PLC (Koog aan de Zaan, Netherlands). The modified starches were delivered to the laboratory in the slurry form. They were taken from the starch production process of Ciba Specialty Chemicals Oy (Anjalankoski, Finland) prior to the final dewatering step. They were used as received. The specification of the different production lots of each starch is shown in Table 7.1, as well as their use in the tests, which are introduced in this thesis.

The test slurries from native starches were prepared by mixing the starch powder, which contained the absorbed water, with deionised water. The slurries were homogenised for several hours by mixing before the filtration experiments. The feed solid concentration of each test slurry was measured by taking a sample of the slurry, and weighing, drying and re-weighing it. The average solid concentration of the studied starch slurries was

40.2 ± 0.5 w-% expressed as kilograms of dry starch per kilogram of slurry. The standard error of the concentration was estimated with a 95% confidence interval.

Sodium chloride, which is a natural impurity in starch slurry, was used as a tracer in the washing experiments (Rhodes, 1934, Wakeman & Attwood, 1988, Sedin & Theliander, 2004, Salmela & Oja, 1999a, 2000, 2005b, 2006). The chloride content of each native starch slurry was adjusted by the addition of commercial sea salt from Parttia Oy (Kotka, Finland). From the modified starches, the cationic wheat and potato starches were chosen for the tests because they were readily available and recommended by the manufacturer. They are commonly used in paper manufacturing. The chloride content of the modified starch slurries were not affected, because they were high enough for washing experiments. The exact chloride content of each test slurry was measured with a Sherwood Chloride Analyzer 926 that uses the titration method based on the traditional silvernitrate-reagent titration, which relies on the formation of a very insoluble salt, silver chloride. The chloride content was measured by taking a slurry sample of 2 dl. Then the solids were allowed to settle, and a 5 ml sample was taken from the supernatant liquid of the sample. This sample was diluted with 20 ml of de-ionised water, because the measuring range of the analyser was from 0 to 999 mg/dm^3. A sample of 500 µm was taken for chloride analysis. The average chloride content of the native starch slurries was 19 893 ± 523 ppm, when the standard error of the concentration was estimated with a 95% confidence interval. The corresponding value for the modified starches was 5714 ± 315 ppm. The total standard deviation of this measurement technique was estimated with Pierre Gy's sampling theory. It was 1.97%, which indicates that this measurement technique is correct (Pitard, 1989, Gy, 1998, Salmela & Oja, 2006).

7.2 FILTRATION EXPERIMENTS

The filtration experiments introduced in this thesis are performed with two different test filters:

- Piston press test filter, with a filtration area of 78.5 cm^2
- Laboratory-scale filter press Larox PF 0.1 H2, with a filtration area of 0.1 m^2

7.2.1 Piston Press Test Filter

The piston press test filter is equipped with a slurry feeding system, a computer-controlled piston press and a data collection system. The pressure of the pneumatic work cylinder regulates the position of the piston and the pressure applied by the piston. Two smaller work cylinders lift and lower the inner wall of the chamber. The piping below the filter medium is filled with water and the filter cloth is placed on a filter medium support. Then the operating parameters are selected. The data collection system is initiated and the inner wall of the filter chamber and the piston are lifted up in order to open the feeding ports that are located just above the filter medium. Next, the slurry flows automatically into the chamber and lifts the piston up to the pre-set position. After filling, the feeding ports are closed by pushing the inner wall of the chamber down. After this, the filtration is started automatically by the pre-set pressure profile. The pressure transmitter in the filtration chamber only works in contact with liquid. When the filtration is over, the control system automatically starts the expression at the pre-set pressure. After expression, the check valve is opened, the filling water drained from the piping and the data collection system is stopped. The piston is lifted up and the filter cake removed, weighed, dried, and re-weighed to measure its moisture content. The flow sheet of the piston press test filter is shown in Figure 7.1.

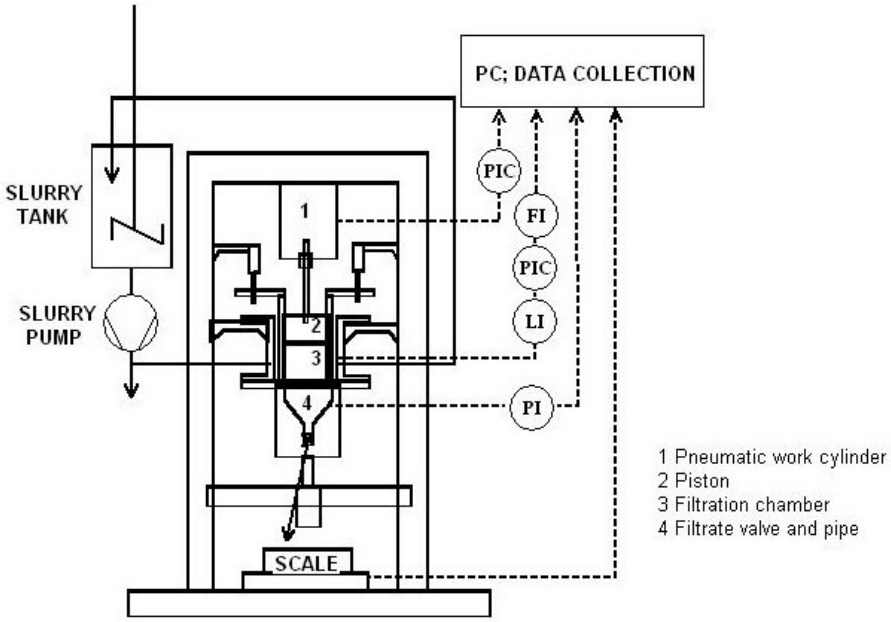

Figure 7.1 Flowsheet of the piston press test filter (Oja, 1996).

The following experiments were performed with this filter:

- **The average specific filtration resistances** of the starch filter cakes were measured by performing a series of measurements, in which certain starch slurry was filtered at a constant pressure. The duration of the filtration stage depended on the starch grade and on the filtration pressure. Between the tests the filtration pressure was increased gradually from one to seven (or eight) bar. After each filtration, the cake was automatically compressed at a constant pressure of 16 bar until the flow of filtrate ceased. (Salmela & Oja, 2000/ Table 4, and Figure 1, Salmela & Oja, 2006 / Table 1)

- **The compactibilities** of starches were measured by plotting the average specific filtration resistances measured with piston press test filter against the filtration pressures according to Chapter 3.2.2. The compactibility was obtained from the slope of this curve. (Salmela & Oja, 2000/ Table 4, and Figure 1, Salmela & Oja, 2006 / Table 1)

- **Compression dewatering** experiments with different starches were performed with this filter in order to test how to measure the transition from filtration to consolidation and to follow the proceeding of the consolidation. In this filter, the transition occurred automatically. The compression was continued until the data had stayed constant for a while. (Salmela & Oja, 2005a / 1-7, and 9, Tables 2 and 3, Salmela & Oja, 2005b / Figure 3)

7.2.2 Small-Scale Filter Press

The small-scale filter press has a data collection system but the operation is manually controlled. At the start of the filtration the slurry feed line to the filter chamber is opened. It takes some time to reach a constant pressure value which is predetermined by adjusting the air-operated double-diaphragm feed pump pressure. Pumping is continued for one to three minutes depending on the starch type, in order to obtain a final filter cake height of 30-40 mm. The duration of the filtration period is kept at a level that is approximately equal to that in the automatically controlled Piston Press filter. Then, the feed line is closed and the next stage is started. The pre-wash compression and/or

displacement, if included, are/is short. Their duration is about 10-20 s. The washing is performed by opening the wash valve, which brings the wash water from the pressurised tank into the filter chamber. The duration of the washing depends on the required product quality. The diaphragm compression is initiated by pumping pressurised water over the rubbery diaphragm which is located at the upper interior surface of the chamber. It compresses the cake until the filtrate flow from the filter chamber ceases. Additionally or instead of compression dewatering, the cake can be air-dried by feeding pressurised air into the filter chamber. The flow sheet of the laboratory scale filter press Larox PF 0.1 H2 is shown in Figure 7.2.

After the filtration test, the cake was removed, and its chloride content was measured by taking a sample of about 5 g from the cake, weighing, reslurring it with 50 ml of water, centrifuging and analysing the chloride content of the supernatant with a Sherwood Chloride Analyzer 926. The chlorides remained in the cake dissolved in the water during reslurring. The uncertainty of this sampling technique was estimated by the fundamental sampling error model (Pitard, 1989 and Gy, 1998). The obtained total standard deviation of the chloride content of the cakes was 2.5%, which indicates that the sampling prodecure is accurate. The cake was also weighed, and a sample was taken to measure the solid content of the cake.

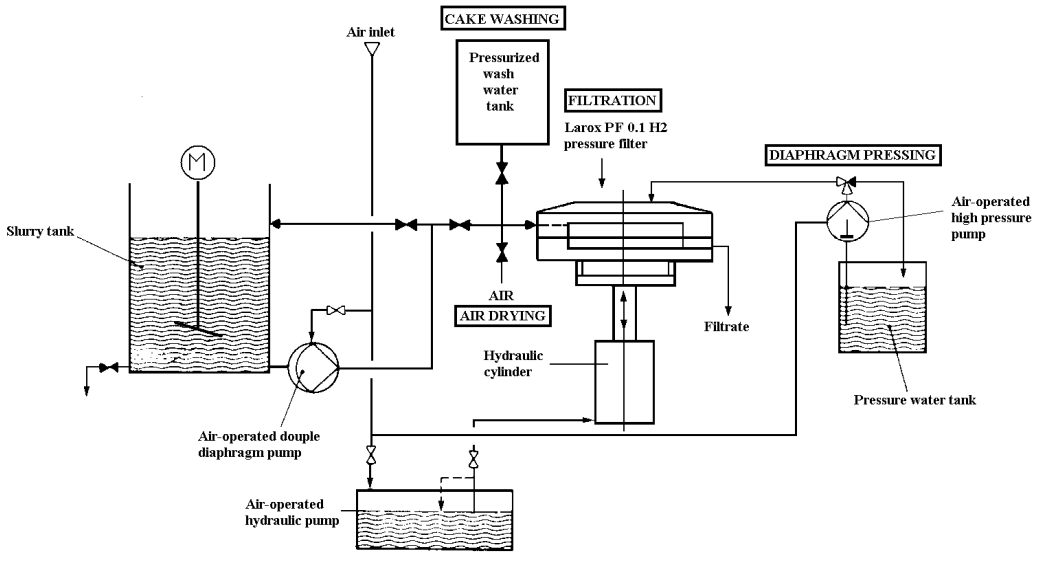

Figure 7.2 Flowsheet of the test filter Larox PF 0.1 H2.

The following experiments were performed with this filter:

- **Filter media testing.** Different filter media were tested with this filter by filtering pure water through each medium in turn, and by measuring the water flow rates during the tests. Then the corresponding tests were performed with starch slurry. (Salmela & Oja, 2004)

- **Cake washing tests** were performed following the procedure described above. The temperature of the wash water and the slurry was about 20°C. A series of measurements were performed so that in the first test the cake was not washed, in the second test it was washed until the conductivity of the out-flowing filtrate had fallen to a low, constant level, and in the following tests, the duration of washing was shorter, and increased gradually from test to test. (Salmela & Oja, 1999a, / Figures 2-5, Table 1, Salmela & Oja, 2000 / Figure 2, Salmela & Oja, 2006 / Figures 4-7, Table 2)

- **Compression dewatering** experiments with different starches were performed with this filter in order to test the comparability between the results given by the piston press and the results given by this filter. The filtration stage was performed following the filtration stage in the automatic piston press filter, and the compression with the diaphragm was performed at 16 bar until the data remained constant for a while. (Salmela & Oja, 2005a / Figure 8, Tables 2, 3)

- **Different combinations of filtration stages** were tested with this filter. The results obtained from each stage were analysed individually, and the different combinations were compared by trying to maximise the capacity of the filter and the purity of the cake. (Salmela & Oja, 2005b)

7.2.3 Comparability of the Data

Ideally there should be agreement between the values of the average specific cake resistances, α_{av}, for the different filters. However, experiments have shown that there exists differences caused by the unequal thicknesses of the filter cakes and the type of

cake formation. The cake thicknesses are different, because the height of the filter chamber in the piston press is 70 mm, whereas in the filter press it is 45 or 60 mm. In the piston press, the cake is formed by sucking the slurry very quickly into the filter chamber, whereas in the filter press the slurry is pumped and the pressure inside the chamber is maintained with a double-diaphragm pump. Commonly, the values of α_{av} given by the filter press are higher than the values given by the piston press.

The main benefit in using the piston press test filter is its automatism. When the slurry is filtered at a certain pressure, it is easy to predict from former experiments, how the same slurry behaves during filtration in the filter press. A suitable duration for the filtration stage is easily measured with the piston press, and hence, the filtration stage is easily optimised in the filter press.

7.3 IDENTIFICATION OF STARCHES USED IN THE EXPERIMENTAL PART

Table 7.1 shows the different production lots of the starches used in this study. It also specifies which starch is used in a certain experiment, and how it is marked in the publication in question. The production lots used in the tests are specified and characterised later within the Results-part of this thesis.

Table 7.1 Different production lots for each starch, and the specification of their use in the tests introduced in this thesis.

Publication	Starch lot	Figure	Test filter	Table	Mark
Salmela & Oja,					
2000	Native Wheat RC 98	1	Piston Press	3, 4	Wheat-D, A
		2a, 2b	Filter Press		
	Native Wheat RC 1/99	1	Piston Press		Wheat-U, B
	Native Maize, Amylum	1	Piston Press	3, 4	Corn
	Native Barley RC 6/99		Piston Press	3, 4	
	Native Potato RC 6/99	1	Piston Press	3, 4	Potato
2004	Native Wheat RC 12/99	1		1	
	Cationic wheat RC 215LV	5 – 7	Filter Press		
1999a	Native Wheat RC 98	2 – 5	Filter Press	1	native wheat
2006	Native Wheat RC 98	4, 5	Filter Press	2	Wheat 1,2
	Native Wheat RC 12/99	1, 2, 4, 5	Filter Press	2	Wheat 3-6
	Native Barley RC 6/99	1, 2, 6	Filter Press	2	Barley 1
	Native Barley RC 12/99	1, 2, 6	Filter Press	2	Barley 2-4
	Native Potato RC 12/99	1, 2, 7	Filter Press	2	Potato 1-5
	Native Maize, Amylum	1, 2	Filter Press		
	-same starches in Table		Piston Press	1	
2005a	Native Wheat RC 1/99	1 – 5, 6a, 9	Piston Press		native wheat
		8	Both filters		
	Native Potato RC 12/99	6b	Piston Press		native potato
	Native Barley RC 6/99	6c, 9	Piston Press		native barley
	Native Maize, Amylum	6c, 9	Piston Press		Native maize
	Cationic wheat RC 215LV	6a, 7	Piston Press		cationic wheat
	Cationic potato RC 150LP	6b	Piston Press		cationic potato
	-same starches in Tables		Both filters	2, 3	
2005b	Native Wheat RC 98	2	Filter Press		Wheat 1,2
	Native Wheat RC 12/99	1	Piston Press		Nat.wheat
	Cationic wheat RC 215LV	1	Piston Press		Mod.wheat
	NativeBarley RC 6/99	1	Piston Press		Nat.barley
	NativePotato RC 12/99	1	Piston Press		Nat.potato
	Cationic potato RC 150LP	1	Piston Press		Mod.potato
	Native Maize, Amylum	1, 3	Piston Press		Nat.maize

RESULTS

8 STARCHES

Based on the discussion in Chapter 2, the starch grades have different structures and characteristics depending on their origin. In the experimental part of this thesis, it is assumed that the studied starches represent typical commonly manufactured starches, and their characteristics follow those given in Chapter 2. Additionally, the most important characteristics from the perspective of filtration are measured experimentally. This chapter also comprises unpublished results that complement the filtration studies.

8.1 STARCH GRANULE STRUCTURE

The structure of the different types of starch granules was described in Chapter 2.3. In this work, potato represents the tuber starches, whereas wheat, maize, and barley starches represent the cereal starches. The amylose/amylopectin-ratio of the studied starches is assumed to be at the typical ratio shown in Table 2.1 in Chapter 2.3.1. The assumed amylose contents are also shown in Table 8.1. Amylose is assumed to form a clear, viscous gel in water, whereas amylopectin is assumed to absorb water and to swell in excess water, as shown in Chapter 2.3.1.

The internal structure of the starch granules and their crystallinity are assumed to follow those shown in Chapters 2.3.2-2.3.3. The crystallinity is assumed to be in the range of 15-40%, which is the typical range of the starch granule crystallinity. The cereal starches, wheat, barley and maize, are assumed to have an A-type diffraction pattern, whereas the tuber starch, potato, is assumed to have a B-type pattern.

The swelling of starch granules in water was discussed in Chapter 2.3.4. All the starch granules are assumed to be totally swollen in this work, because they have been in the slurry form at least 24 hours before they are filtered or analysed. The amount of water bound by starch is measured by drying the starch samples in the oven over 48 hours and by expressing the solid content as the percentage of the total weight to the dry starch.

The particle size distributions are measured from the swollen samples in the presence of de-ionised water. Hence, the sizes given in this thesis are always the sizes of the swollen starch granules. The possible swelling of the starch granules during the filtration cycle caused by the pure wash water is ascertained not to happen by measuring the size of the granules in the slurry before the experiment, and the size of the granules in the cake after the experiment. The particle size distribution did not change during the filtration process, and hence the consequence of the possible swelling of starch granules during the experiment is ignored in this work. Also the gelatinisation of the starch granules, which was described in Chapter 2.3.4, is ignored, because all the experiments were performed at temperatures between 20 and 25°C, which is clearly below the gelatinisation temperatures of the starches.

The impurities in starch granules are described in Chapter 2.3.5. The contents of impurities in the starches used in the experimental part of this work are assumed to follow these normal trends, and they were not analysed. The only impurity measured is the chloride content, which is used as an indicator of the filter cake purity.

8.2 PARTICLE SIZE AND PARTICLE SIZE DISTRIBUTION

Within this thesis work, the particle sizes of the different starch types were measured in order to understand the filtration behaviour of the starches, and to find the differences in the filtration behaviour between the different starch types. The volume-based particle size distribution of the starches were measured with a Coulter® LS130 particle size analyser and the number-based particle size distributions by a Pamas-SVSS analyser. Additionally, the manual image-analysis was performed with an optical microscope Olympus BHS connected with an AnalySIS® Soft Imaging System via a Hamamatsu C4200 videocamera and C2400 camera unit.

The Coulter® LS130 is a light scattering particle size analyser, which uses the diffraction of the laser light by particles as the main source of information about the particle size. The swollen starch sample was dispersed in de-ionised water and three replicate measurements were performed for every starch grade. The optical model used

in the measurements was created with the Coulter® LS130 –software, version 2.11. The model was based on the Laser Diffraction Proficiency Testing Scheme (LDPTS) measurements in which potato starch was one of the test samples of Round 13 samples. The model uses a value of 1.53 for the refractive index of the starch and a value of 1.327 for that of water (French, 1984). The value of the imaginary refractive index is 0.2. The Z-score of the potato starch mean particle size measurement of round 13 samples was 0.23, which means that the value of the device used in this thesis was closer than two standard deviations to the average value of the international ground group of Coulter® LS130 users' values. Since it was clearly less than two, the model is validated by the international ground group of Coulter® LS130 users. Also the Fraunhofer optical model with PIDS was tested, and ascertained to be suitable for starches. The values obtained for the average mean diameters of different production lots of starches are shown in Table 8.1. Since the difference in the mean particle sizes between the different production lots of each starch is small, the values for the average mean diameters can be assumed to be quite constant for every starch grade. Hence, the values for the average mean diameters of different native starch grades with the standard deviations are shown in Table 8.2 (Salmela & Oja, 1999a/Table 1, 2000/Table 3, and 2004/Table 1). The volume-based particle size distributions of the studied starches are shown also in Figure 8.1 (Salmela & Oja, 2006/Figure 1).

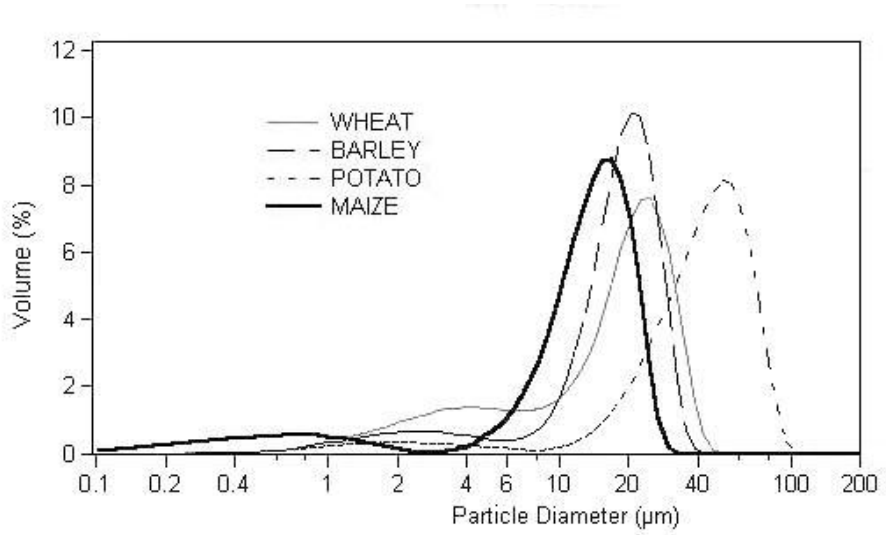

Figure 8.1 The volume-based particle size distribution of different native starches measured with a Coulter® LS130 particle size analyser.

The Pamas SVSS analyses of single particles. The sample is moved through the sensor by a stepper motor controlled syringe. The syringe draws the sample through the HCB-sensor at the optimum flow rate. The surface area of every particle is measured optically from the surface area of the shadow of the particle by the sensor. The size of the particle is the diameter of a circle, whose area corresponds to the area of the particle shadow. The particle concentration is observed as 32 operator selectable size tresholds as a population-distribution.

The swollen starch sample was dispersed in de-ionised water and three replicate measurements were performed for every starch grade. The number-based particle size distributions measured with Pamas SVSS are shown in Figure 8.2 (Salmela & Oja, 2004/Figure 1), and the values obtained for the average mean diameters in Tables 8.1 and 8.2.

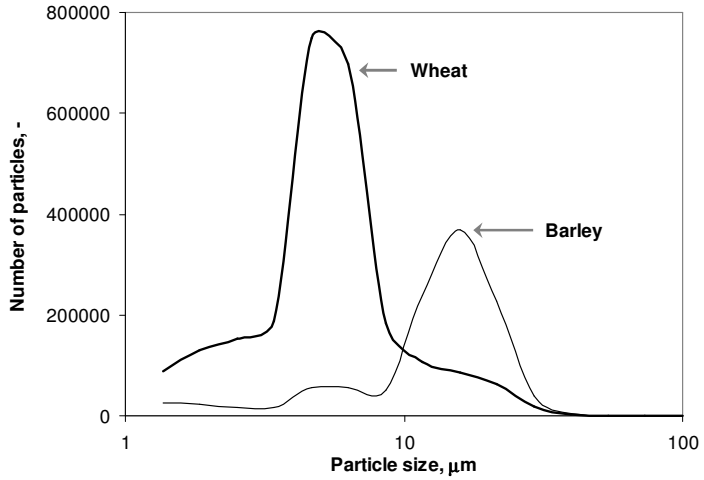

Figure 8.2 Number-based particle size distributions of native wheat (lot 12/99) and barley (lot 12/99) starch. Both distributions are measured with a Pamas-SVSS particle size analyser.

The AnalySIS® Soft Imaging System was used for analysing the pictures of 300…400 particles per starch grade. The pictures were taken from the optical microscope Olympus BHS which was connected with the AnalySIS® via a Hamamatsu C4200 videocamera and C2400 camera unit. Only the whole single granules were considered in the results and the others were rejected. The diameters for starch particles were derived by measuring the size dependent property of the particle and relating it to the linear

dimension. Tables 8.1 and 8.2 show the values of the average mean diameters and the equivalent circle diameters given by the AnalySIS®-image analyser.

Table 8.1 The form of the purchased starch, the assumed amylose content, and the mean particle sizes of the different production lots of starches were measured with three different particle size analysers and two different optical models.

STARCH LOT	ORIGINAL FORM OF STARCH	AMYLOSE CONTENT, %	MEAN PARTICLE SIZE, µm			
			Coulter LS®130 Pstarch-model	Coulter LS®130 Fraunhofer-model	Pamas-SVSS	AnalySIS® Soft Imaging System
Native Wheat RC 98	Powder	26	17.01	16.87		
Native Wheat RC 1/99	Powder	26	18.04	17.86		
Native Wheat RC 12/99	Powder	26	17.72	17.54	5.44	14.7
Cationic Wheat 215LV	Slurry	26	13.54			10.7
Native Barley RC 6/99	Powder	22	16.58	16.60		
Native Barley RC 12/99	Powder	22	17.73	17.74	12.38	20.6
Native Potato RC 6/99	Powder	20	41.48	41.46		
Native Potato RC 12/99	Powder	20	42.56	42.51		24.3
Cationic Potato 150LP	Slurry	20	33.82			
Maize, Amylum Meritena 100	powder	28	13.14	13.15		13.9

Table 8.2 Typical average mean diameters of different native starch grades measured with three different particle size analysers. The values given by the Coulter® LS 130 are the averages of the different production lots and their standard deviations.

	Barley	Maize	Potato	Wheat
Average Mean Diameter, µm				
- Coulter® LS 130	17.2 ± 0.7	13.2 ± 0.1	42.6 ± 0.6	17.3 ± 0.6
- Pamas SVSS	12.4			5.4
- AnalySIS®	20.6	13.9	24.3	15.3
Equivalent Mean Circle Diameter, µm				
- AnalySIS®	19.4	13.2	23.1	14.7

According to Figures 8.1 and 8.2, and Tables 8.1 and 8.2, it is evident that the four native starch grades have a different particle size and particle size distribution and, therefore, have to be treated as different materials. The wheat starch contains a remarkable fraction of small particles, which is clearly seen in the form of the primary peak in Figure 8.2, and hence its particle size distribution is strongly bi-modal. This is also supported for example by Jane *et al*., (1994), Stapley *et al*., (1999), Manelius & Bertoft (2002), and Tester *et al*., (2004). On the other hand, the native barley starch used in this study does not have a bi-modal particle size distribution, which is shown to be

typical for barley starch by Snyder (1984), Jane *et al*., (1994), and Tester *et al*., (2004). A small fraction of smaller barley particles is seen in Figures 8.1 and 8.2, but it is not as remarkable as in the case of wheat starch.

The particle size distribution is directly related to the rate of filter cake formation. The fraction of fine particles in the slurry leads to a dramatic decrease in the cake porosity (Mota *et al*., 1999). This is supported by the filtration experiments with the piston press filter. In the Piston press test filter, the filtration ends automatically, when there is no more free water in the filter chamber and the cake is formed. Hence, the time needed for starch cake formation at a certain pressure can be easily compared. In the experiments, the cake formation was slowest for wheat starch, intermediate for maize and barley starch, and very short for potato starch. For example, wheat starch needed 6 minutes, barley starch 50 seconds, maize starch 30 seconds, and potato starch about 3 seconds to form a cake, when the filtration pressure was 3 bar (Salmela & Oja, 1999b). This indicates clearly the effect of small wheat particles on the cake formation. The smaller particles tend to plug the bigger pores, and hence decrease the porosity of the cake. The low porosity then decreases the wash water flow through the cake. On the other hand, the potato starch is easy to filter, because it has a greater mean particle size than the other starches have, and hence, the porosity of the potato starch cake is higher, and the cake is easier to wash.

As a conclusion, by knowing the particle size distribution of the filtered starch, the filtration behaviour can be anticipated from former experiments. The particle sizes obtained are not necessarily absolute values, but they allow easy comparison of the different starch grades.

8.3 PARTICLE SHAPE

The particle shape affects the filter cake formation and its filtration resistance. The shapes can be studied with an optical microscope or with a scanning electron microscope (SEM).

When the SEM is used, the starch samples are prepared by permitting the granules to fall on double-sided tape, which is attached to the specimen stub. The samples are coated with a thin layer of metal under high vacuum to prevent the build-up of a primary electron negative charge on an otherwise nonconductive starch (Fitt & Snyder, 1984). The SEM of the starch granules provides information on the smoothness of the surface and the determines if any of the granules are broken. In this study, the shapes of the particles were analysed by a scanning electron microscope JEOL JSM-5800. The pictures taken are shown in Figure 8.3.

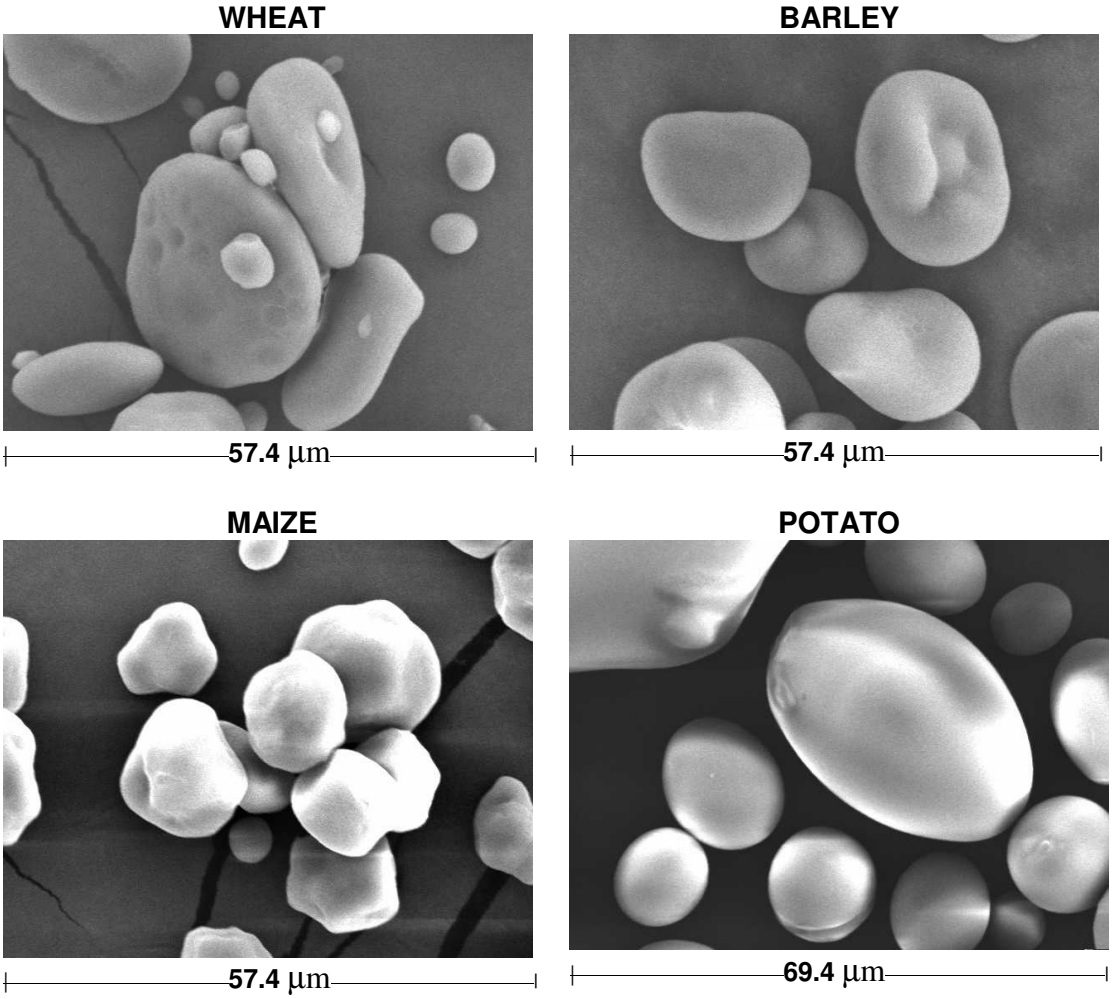

Figure 8.3 The shapes of the starch particles analysed with a JEOL JSM-5800 Scanning Electron Microscope (Salmela & Oja, 2006/Figure 2).

The light microscope provides information about the shape and size of the particles, details on the internal structure of the starch particles, and the growth rings of large hydrated granules. The optical microscope used in this study was an Olympus BHS

connected to an AnalySIS® Soft Imaging System via a Hamamatsu C4200 videocamera and C2400 camera unit. The pictures are shown in Figure 8.4.

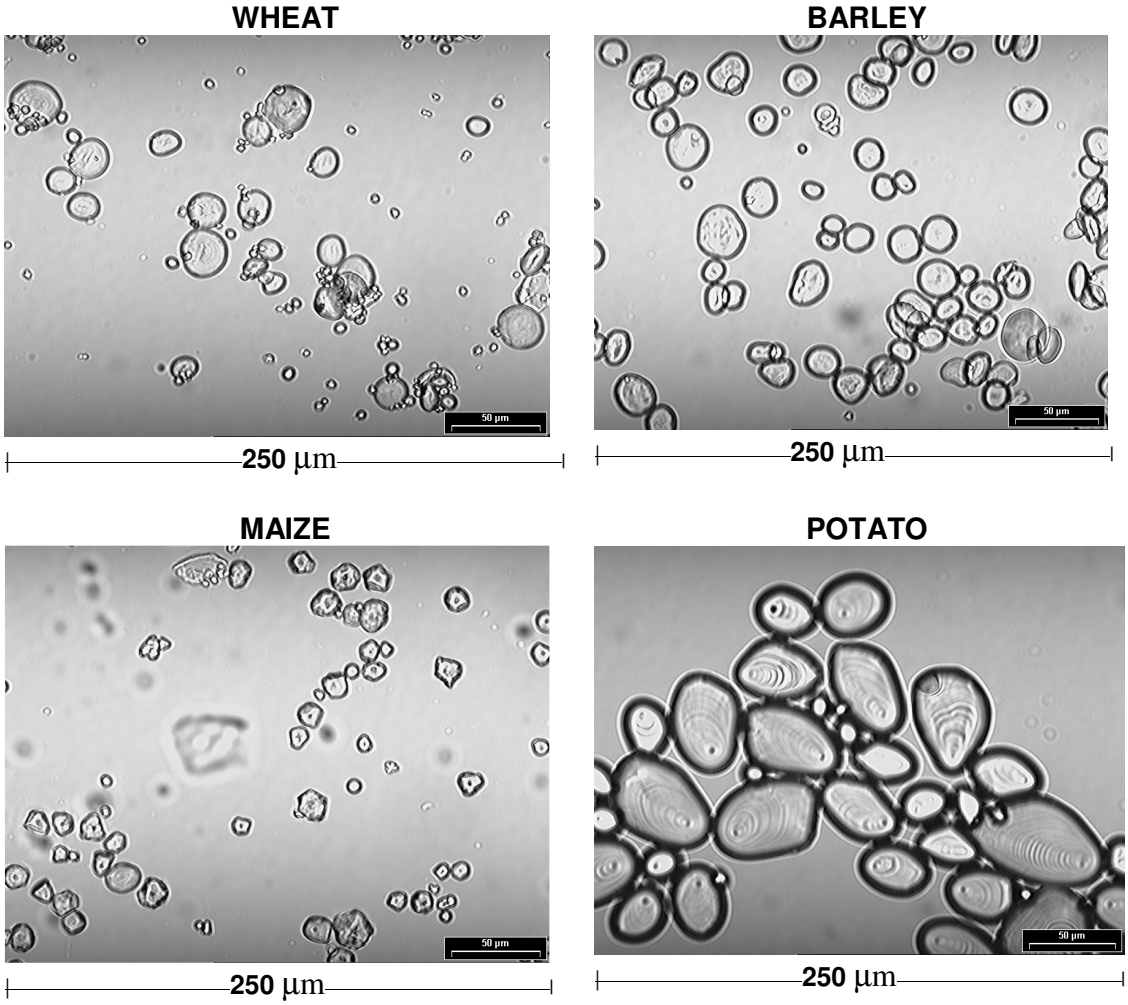

Figure 8.4 The shapes of the starch particles analysed with an optical microscope Olympus BHS connected to an AnalySIS® Soft Imaging System via a Hamamatsu C4200 videocamera and C2400 camera unit.

The shapes of the particles are compared also with the shape factors. The shape factor, Φ_1, provides information about the slimness of the particles. The AnalySIS® -shape factor does not describe any specific shape, but it helps in comparing the different starch grades. The third shape factor, Φ_2, compares the stability of the particle position during the particle size measurement. The shape factors measured for different native starch grades are shown in Table 8.3. There were very little variations between the shape factors of different production lots, and hence the shape factors are generalised to represent the native starch grades independent of the production lots.

Table 8.3 Shape factors for native starches.

	Barley	Maize	Potato	Wheat
Shape Factor Φ_1	0.82	0.82	0.76	0.80
AnalySIS® Shape Factor	0.94	0.89	0.92	0.88
Shape Factor Φ_2	0.75			0.38

Figures 8.3 and 8.4 show, that the visual shapes of the starch particles used in this study, follow the general classification of the starch particle shapes, which was given in Chapter 2.4.2 and Table 2.2 (Jane *et al*., 1994). The tuber starch, potato starch, has large and smooth particles, whereas the cereal starch particles are thicker and round in shape. Maize starch granules are irregular polygons with sharp edges. Also the impression of the typical spherical protein microbodies, which have impeded the local growth of the wheat starch granules, can be seen.

In Figures 8.3 and 8.4, wheat and barley starch granules are visually quite similar to each other in size and in shape, except that the fraction of small wheat starch granules is clearly seen, and the small wheat starch particles seem to be adhered onto the surface of the larger particles. On the other hand, several barley starch granules have one larger hollow on their surface. Potato starch granules seem to be bigger, round or oblong in shape, and they have a very smooth surface. Maize starch granules are the smallest and the most angular in shape of the four starches. However, when the values of the shape factor, Φ_1, in Table 8.3 are considered, the barley and maize starches have the most round shape, but wheat starch is very close to them, whereas potato starch has a slightly smaller value of 'roundness'. The AnalySIS®-shape factor does not give remarkable differences between the shapes of the particles. The values of the shape factor, Φ_2, in Table 8.3 show that the position of the granule has an effect on its measured shape, and it has to be taken into account when choosing the measuring technique.

Additionally, it was also ascertained, or whether the filtration affects the shape of the starch particles. Some samples from the slurries were studied with SEM before the filtration experiments, and then the samples from the filtered cakes were studied similarly. No noticeable differences were observed between the shapes of the particles before the experiment and after the experiment.

As a conclusion, all the studied starch grades have quite a round shape and the differences between their shape factors are so small, that they can be considered as spheres during filtration cycle analysis. Hence, the shapes of the particles do not have to be taken into account in the filtration equations.

8.4 DENSITIES OF STARCHES

The density of the starch particles affects their settling rate in water. Hence, the density has to be taken into account in the planning of the filtration. The density of starch powders used in this study was measured by weighing a certain amount of starch, and measuring the liquid volume which was displaced by the starch. The weighed starch contained 10-20% of naturally absorbed water. Starch was allowed to swell before the volume was measured. The density of the starch slurries to be filtered was measured by weighing 500 ml of the slurry, for which the solid content was known. The measured densities of the native starch powders and slurries used in this study are shown in Table 8.4.

Table 8.4 Densities of different native hydrous starch powders and starch slurries.

	Barley	Maize	Potato	Wheat
Density of starch powder, kg/m^3	1434	1418	1443	1498
Density of 40 w-% starch slurry, kg/m^3	1172	1172	1156	1176

As a conclusion, because the differences between the densities of the different starch grades are very small, they play no role in the filtration.

8.5 SETTLING OF STARCH

The settling velocities of the 40 w-% native starch slurries were measured in this thesis in order to ensure that the effect of sedimentation can be neglected in the analysis of the data. The settling of the 40 w-% native starch slurries was allowed to proceed in a measuring glass over a couple of days until the final height of the sediment layer was achieved. The settling curves are shown in Figure 8.5. The settling rate of the same

slurries to which were added 2% of NaCl per kilogram of dry starch were also studied, because this NaCl-content is typical during the processing of starch slurries.

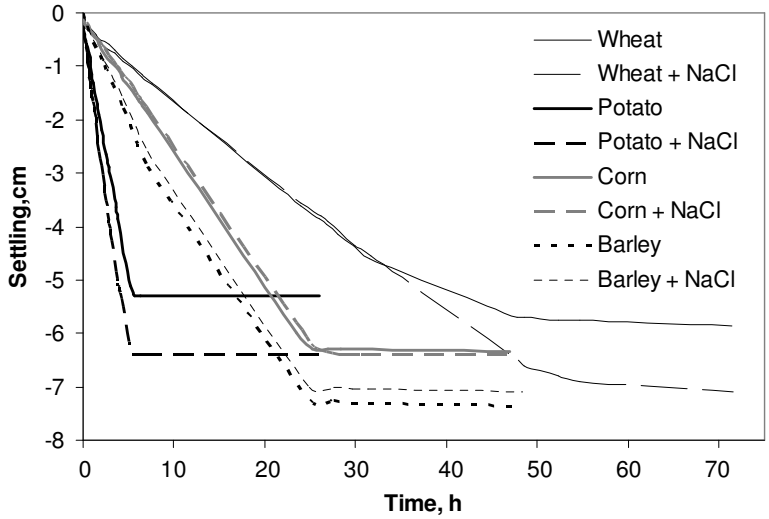

Figure 8.5 Settling curves of 40 w-% native wheat, barley, potato and maize starch slurry, and settling curves of the same slurries with 2% sodium chloride as an impurity.

Figure 8.5 shows that the sedimentation rate of the 40 w-% starch slurry is very slow in the filtration applications. Usually the filtration cycle lasts a couple of minutes, and during this time sedimentation is very minimal. The sedimentation velocity of starches seems not to be directly proportional to the mean particle size. The settling velocity of potato, barley and maize starch seems to be proportional to the mean particle size, but wheat starch has a considerably slower settling velocity. The reason for this might be to do with the bi-modal particle size distribution and the great amount of small particles. The addition of sodium chloride, which is one of the main impurities in starch processing, did not affect appreciably on the settling velocity, but starch forms a denser sediment layer when the slurry contains sodium chloride. Obviously, the salt content in the slurry influences the final cake structure, and hence the possible change in the salt content of the original slurry has to be taken into account in filtration applications.

As a conclusion, these very slow settling rates prove that the sedimentation can be neglected for concentrated starch slurries in comparison to filtration, and hence, the general filtration equations can be used. Additionally, the salt content of the original slurry affects the final cake structure.

8.6 FLOW BEHAVIOUR OF STARCH SLURRIES

The rheology of the starch slurries was tested at the probable conditions that will arise in the plant in order to minimise the slurry handling problems in filtration. Figure 8.6 shows the viscosities of the starch slurries of varying solid content versus the shear rates, as well as the corresponding power law –model curves calculated from Equation (2.6). The viscosities were measured with a Brookfield LVTD -viscometer which relies on rotational motion to achieve a simple shearing flow.

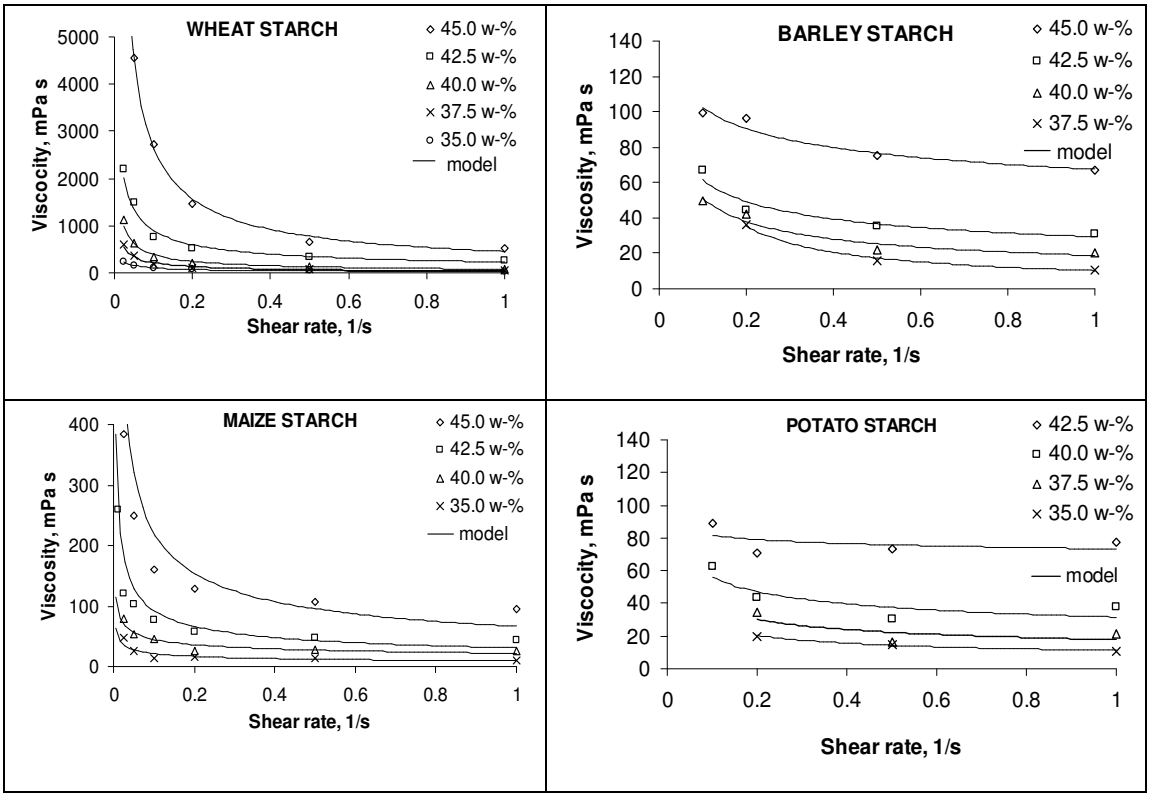

Figure 8.6 Viscosities of starch slurries of varying solid contents with different shear rates, and the power law-type model for describing the experimental results. Viscosities were measured with Brookfield LVTD-viscometer.

According to Figure 8.6 all studied starches show non-Newtonian, shear-thinning, pseudoplastic behaviour at their typical filtration concentrations. The viscosity-curve is well approximated by the Ostwald-de Waele power law model, which is used extensively to describe the non-Newtonian flow properties of a liquid in theoretical analyses as well as in practical engineering applications (Barnes *et al.*, 1989).

When the flow curve of a certain slurry is measured, and the flow behaviour index n_v calculated, then the flow of that slurry during pumping can be anticipated when planning the filtration operations. If the pumping pressure changes, the shear rate varies which in turn affects the viscosity of the slurry (Weltmann, 1960), or on the contrary, if the concentration of the slurry changes, the viscosity changes also, which in turn has to be taken into account in the pumping.

As a conclusion, the viscosity of the slurry decreases if the concentration of the slurry decreases and vice versa. This means that the pumping of the slurry has to be regulated depending on the concentration of the slurry, if the cake formation needs to stay unchanged.

8.7 SUMMARY

Generally, within this thesis, the internal structure, amylose/amylopectin-ratio, crystallinity, and gelatinisation of starches were assumed to follow the known characteristics of starches reviewed in Chapter 2. Also the content of the impurities were assumed to be in the 'normal range', whereas NaCl was used as an indicator of the cake purity. The particles were assumed to be totally swelled in all the analysis and experiments, and hence the given particle sizes were the sizes of the swollen starch particles.

The measured particle size distributions of different starches were shown in Figures 8.1 and 8.2 and the mean particle sizes in Tables 8.1 and 8.2. It was ascertained, that when the particle size distribution of the starch to be filtered is known, its filtration behaviour can be anticipated from the former experiments of starch with a similar particle size distribution. The shapes of the starch particles were visualised in Figures 8.3 and 8.4. Also the shape factors of different starch grades were measured in order to get quantitative information about the shapes (Table 8.3). It was proved that the shape of the starch particles does not have a noticeable effect on the filtration behaviour and the differences between starch types are small. Studied starches were considered as spheres.

The differences between the densities of the different starch grades were shown to be very small (Table 8.4). For filtration the density differences played no role. The settling rates of 40 w-% starch slurries were very small (Figure 8.5), which proves that the sedimentation can be neglected in the filtration of concentrated starch slurries, and hence, general filtration equations can be used. Of course, if the slurry to be filtered is remarkably less concentrated, the sedimentation eventually begins to affect the filtration.

Finally, the viscosity of starch slurries at their typical filtration concentrations (35-45 w-%) were found to have non-Newtonian, shear-thinning, pseudoplastic behaviour and to follow the power law –model curves (Figure 8.6). If the concentration of the slurry to be filtered decreases, its viscosity decreases too, which in turn causes the pumping power of the slurry to be decreased.

From now on, within this thesis, it is assumed that for every native and modified starch grade the mean particle size, particle size distribution, and other characteristics are almost constant for that certain starch type, and the variations between the different production lots are minimal. Hence, from now on within this thesis, the starch grades are mainly named after their grade without mentioning the production lots.

9 FILTRATION STAGE (Appendix I: Salmela & Oja, 2000)

The different starch types are characterised by means of their specific filter cake resistance and its dependency on the filtration pressure. This chapter gives the filtration characteristics of the starch slurries used in the experimental part of this work.

9.1 AVERAGE SPECIFIC CAKE RESISTANCE AND COMPACTIBILITY

In starch filtration tests with the piston press test filter and with the small-scale filter press, the average specific cake resistances are calculated from Equation (3.3) by measuring the slope of the test data plot $(t\text{-}t_s)/(V\text{-}V_s)$ versus $(V+V_s)$ and by calculating the average specific cake resistance from it. Then the average specific cake resistances measured at different filtration pressures with the piston press test filter are plotted against the filtration pressure on a logarithmic scale, as shown in Figure 9.1 (Salmela & Oja, 2000/Figure 1, and 2005b/Figure 1). The compactibility of each starch is measured from the slopes of these curves and the local specific cake resistances from the intercepts between the curves and y-axis, when the pressure scaling pressure is one bar. The values of the compactibilities and the local specific cake resistances are shown in Table 9.1 (Salmela & Oja, 2000/Table 4, 2005a/Table 2, and 2006/Table 1). The R^2-value in Table 9.1 reveals how closely the estimated values for the trendline correspond to the actual data.

Figure 9.1 shows that the specific resistance of a filter cake depends on the origin of the starch particles. There may be even a hundredfold difference between the different starch grades. Native wheat and barley starch, as well as both of the modified starches, seem to be slightly compactible, when the n-values in Table 9.1 are compared to the limiting values given in Chapter 3.2.2 (Salmela & Oja, 2000/Table 4). Wheat starch is the most compactible of all starches, and it has the greatest value of α_0. The fraction of small wheat starch particles, which was shown in Chapter 8.2, is possibly blocking the cake voids, and hence the volume of the voids and flow channels are smaller in wheat starch cakes than in the cakes formed from other starches (Salmela & Oja, 2006). The specific resistances of native maize starch cakes are quite independent of the filtration pressure between the pressure of 2 bar and 6 bar, as well as the specific resistances of

the native potato starch cakes between a pressure of 1 and 4 bar (Salmela & Oja, 2000, 2006).

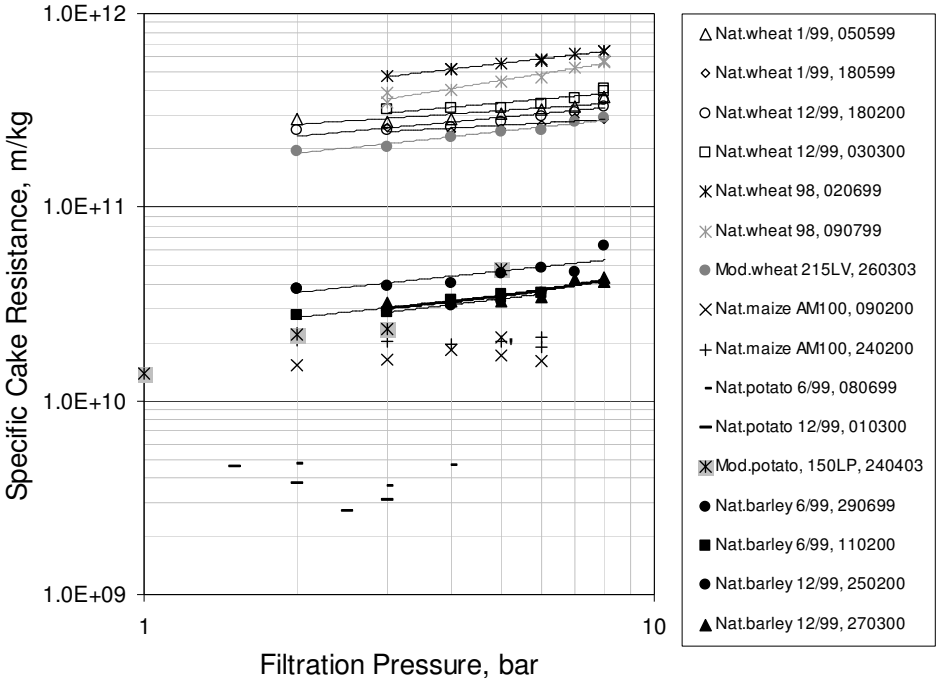

Figure 9.1 Average specific resistances of different starch filter cakes in pressure filtration with a piston press test filter. Parallel production lots and test series are shown. The compactibility of each starch is measured from the slopes of these curves and the local specific cake resistances from the intercepts between the curves and the y-axis.

Takai *et al.* (1987) measured the compactibilities of starches in the pressure range from 0.188 bar to 1.15 bar. Their results showed that potato, sweet potato and maize starches form incompressible filter cakes and wheat starch cake was compressible (n = 0.36) in that pressure range. Correspondingly, Kozicki & Kuang (1994) showed that the diameters of the tapioca starch granules decreased with increasing compressive pressure across the filter cake suggesting that tapioca starch particles were compressible over the range of filtration pressure from 3.5 bar to 6.9 bar.

Table 9.1 Compactibility coefficients of different starch grades, local specific cake resistances of the starches at Δp_0 =1 bar, and the coefficients of determination. The experiments are performed with a piston press test filter, with parallel production lots and test series.

Starch type, production lot, Slurry code	Compactibility n, -	Local specific cake resistance at 1 bar $\alpha_0 \times 10^{12}$, m/kg	Coefficient of determination R^2, -
Native wheat 1/99, 050599	0.18	2.88	0.711
Native wheat 1/99, 180599	0.15	2.45	0.694
Native wheat 12/99, 180200	0.25	2.59	0.826
Native wheat 12/99, 180200	0.24	3.10	0.780
Native wheat 98, 020699	0.31	4.86	0.994
Native wheat 98, 090799	0.43	3.96	0.957
Cationic wheat 215LV, 260303	0.28	2.17	0.964
Native barley 6/99, 290699	0.28	0.414	0.738
Native barley 6/99, 110200	0.28	0.309	0.935
Native barley 12/99, 250200	0.31	0.296	0.957
Native barley 12/99, 270300	0.32	0.311	0.771
Native maize AM100, 090200	0.05	0.174	0.777
Native maize AM100, 240200	0	0.204	0.914
Native potato 6/99, 080699	0	0.043	0.838
Native potato 12/99, 010300	0	0.035	0.705
Cationic potato, 150LP, 240403	0.72	0.463	0.919

The maize and potato starches could not be filtered at higher pressures, because their filtration rate is high even at low pressures (Salmela & Oja, 2000). The time needed to form a starch cake in a piston press test filter seems to be directly proportional to the specific cake resistance, and the cakes with lower specific cake resistances can be filtered at lower pressures (Salmela & Oja, 2000). If the filtration pressure is too high, the permeability of the cake will be too low, and hence, the cakes cannot be washed. On the other hand, the filtration pressure has to be high enough in order to form a cake, which is thick enough to keep the capacity of the filter high. The higher the α_{av} or α_0, the longer it takes to wash the cake. The wash water flow rate in the cake with higher specific resistance is lower and, hence it takes a longer time for the wash water to flow through the cake (Salmela & Oja, 2006).

Also small differences between the production lots and of the slurries of the same starch grade are observed, as can be seen from Table 9.1. The reason is that the measurement of the compactibilities from the slopes of the trendlines is quite sensitive. If some of the

data points deviate slightly from the trendline, it changes the slope of the curve and decreases the coefficient of determination. Usually these uncertain points are obtained with the lowest pressures, although the deviations are not very remarkable. However, it is easily seen from Figure 9.1, that each starch grade has its own typical range of average specific cake resistances, and the compactibility coefficients of different production lots and slurry lots are quite close to each other. This means that the origin of starch has to be taken into account when planning the filtration process. Different starch grades need their own typical pressures and durations for the stages during the pressure filtration cycle due to their typical specific filtration resistance and compactibility. The variation between the slurries and the production lots of each starch is small, and hence each slurry does not have to be tested independently. When the origin, concentration and particle size distribution of the slurry are known, its average specific filtration resistance and compactibility, and hence the filtration conditions needed, can be anticipated from former experiments with that starch grade (Salmela & Oja, 2000).

Also the modification of starches affects their filtration properties. When wheat or potato starches are cationised, they seem to become more compactible and the filtration resistance of the potato starch is increased (Salmela & Oja, 2006). Hence, the each modified starch has to also be treated like a different starch grade.

9.2 SUMMARY

This chapter showed that the average specific cake resistance and the compactibility depended strongly on the origin of the starch particles (Fig. 9.1 and Table 9.1). The average specific resistance of maize and potato starches was not affected by the filtration pressure at pressures from one to seven bar, whereas wheat and barley starches showed a slightly compactible behaviour at these pressures. This indicated that Eq. (3.5) is suitable for starch filtration applications.

The results introduced in this chapter indicated, that the filtration stage for every starch grade had to be optimised by searching for a suitable combination of filtration pressure, duration of filtration, cake height, and the average specific resistance of the cake, and at

the same time by keeping the capacity of the filter as high as possible. When the origin, the concentration, and the particle size distribution of the slurry to be filtered are known, its probable behaviour during the filtration cycle can be checked from the former filtration runs of slurry of similar type. Additionally, it is important to define the requirements for the quality of the end product and for the capacity of the filter, and hence decide whether to wash, compress and/or air-dry the cake. The higher the typical average specific resistance of the starch to be filtered, the longer the filtration and washing times needed. Also slightly higher pressures have to be used in order to keep the flow rate and capacity high enough.

10 MEDIA FOR STARCH FILTRATION APPLICATIONS

(Appendix II: Salmela & Oja, 2004)

There are only a few articles dealing with filter media for starches, but a clear dependency between particle size and the filter medium resistance in starch filtration is evident. Satoh (1957) filtered 17.8 w-% and 26.7 w-% sweet potato starch and potato starch slurries. The mean diameter of the projected area of his sweet potato starch was 8.4 µm, whereas that of the potato starch was 25.6 µm (Satoh, 1957). The resistance of the same medium was more than six times greater in the filtration of smaller particulate sweet potato starch than in the filtration of greater particulate potato starch. In spite of the difference in the particle size data, the ψ's, shown in Equation (4.3), coincide with each other. Additionally, for sweet potato starch the ratio between the resistances of the caked cloth and the clean cloth was between 370 and 440 and for potato starch between 57 and 75 (Satoh, 1957). The concept of the caked cloth means that the filter medium was stuck together with the so called first layer of the cake. From these values it can be concluded that there was penetration of the smaller sweet potato starch particles into the medium.

Takai *et al*. (1987) found that the filter medium resistance remained constant during six filtration runs for a certain starch subjected to filtration pressures from 0.118 bar to 1.15 bar, but the value depended on the starch type. Wheat starch, which had a strongly bimodal particle size distribution, had more than two times greater filter medium resistance than the maize or potato starch. The probable reason for this dependence between the particle size and the filter medium resistance was due to the penetration of small particles into the filter medium (Takai *et al*., 1987).

In Salmela & Oja (2004) three different filter cloths were tested in order to find the differences between these cloths in the wheat starch application. The aim was to find an easy way for the comparison of different media and to show the effect of the medium on the filtration performance. Cationic wheat starch slurry (215LV) was chosen as a test slurry, because of the problems existing in the industrial-scale pressure filtration of wheat starch slurry. The cloths were manufactured by Tamfelt Corp., and chosen according to their recommendation. The tested cloths are all used in starch industry

applications. Cloths A and B were multifilament filter cloths, but their fabric and weave types were different. Cloth C was a multi-layer cloth, in which the weave type was not seen. The pictures of the clean cloths and the pictures of the same cloths after 15 filtration tests are shown in Figure 10.1 (Salmela & Oja, 2004/Figures 2 and 3).

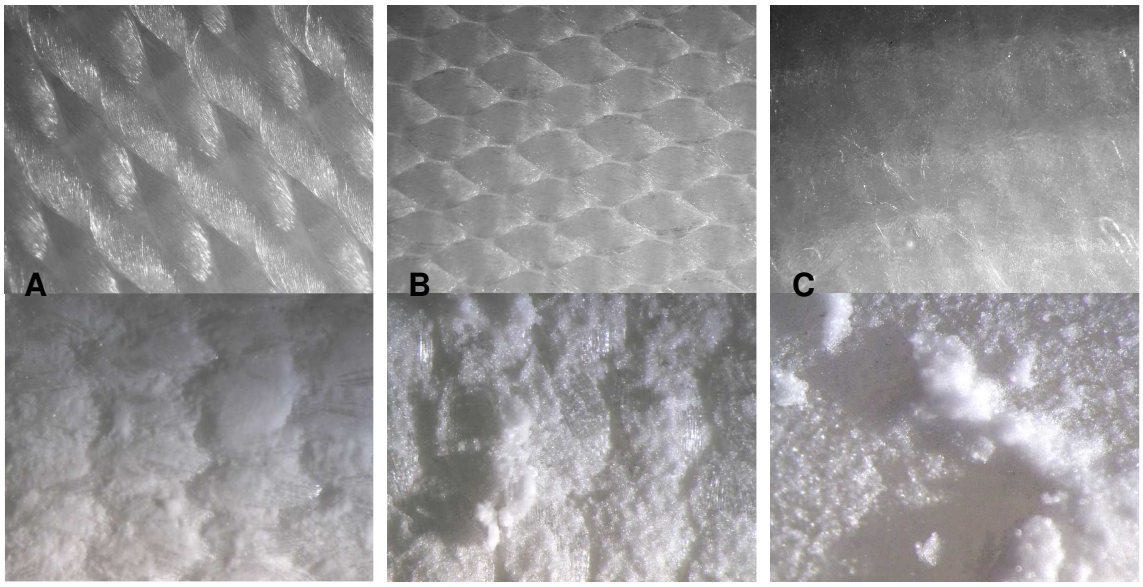

Figure 10.1 Filter cloths tested in wheat starch filtration systems. Upper pictures are taken from the clean cloths and the lower from the used filter cloths, in which the main cake is stripped off, but the cloth is not washed. Magnification of the pictures is ×80. (Salmela & Oja, 2004/ Figures 2 and 3).

The resistances of the filter cloths were measured before the filtration tests, and after 6, 12 and 15 filtration tests (Salmela & Oja, 2004/Figure 4). They were tested in the same small-scale filter press as the actual filtrations, but by filtering clean water through the cloth, which was normally mounted in the test filter, laboratory scale Larox 0.1 H2 – filter. After each filtration run, the turbidity of the filtrate was measured, and the cloth was washed from both sides with pressurised water. The resistances of cloths A and B, and the turbidities of the filtrates increased when the amount of filtration runs increased (Salmela & Oja, 2004/Figures 4 and 6). This indicated the migration of small particles into the medium. However, the criterion of negligible filter medium resistance, shown in Chapter 4.2 was fulfilled. The resistance of cloth C decreased as the amount of filtration runs increased possibly due to further wetting of the medium, and despite the fact that the media was wetted before the experiments.

The performance of the filter cloths during the normal filtration test is compared as follows (Salmela & Oja, 2004): the process conditions are kept constant in order to form similar cakes in each parallel test. The only difference between the filtration runs was the cloth, and hence the changed flow of filtrate through the system of the cloth and the cake. Then the out-coming filtrate flows in the tests are compared, as shown in Figure 10.2. The differences in the flow curves indicate the differences in the weaves and the materials of the cloths. The sharp rise in the curve during the dewatering is a consequence of the small amount of air, which was left inside the filter chamber during the filling (Salmela & Oja, 2004).

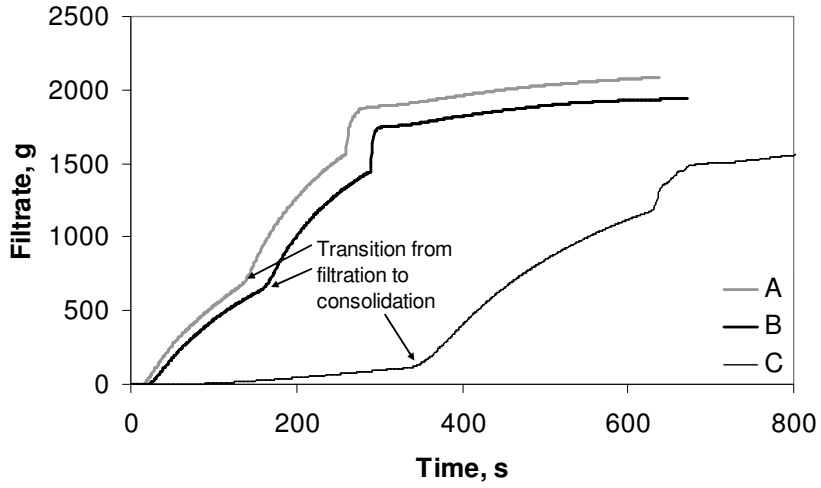

Figure 10.2 Filtrate flows through three different filter cloths in cationic wheat starch (215 LV) pressure filtration applications. The curves are from the normal filtration runs, in which the process conditions are similar, and the only variable is the cloth type (Salmela & Oja, 2004/Figure 5).

By comparing the curves of the filtrate flow through the cakes and the cloths from the normal filtration runs, it is possible to compare the performances of the filter cloths in the actual filtration process. The cloth A, which has the lowest resistance measured by filtering pure water, has the highest filtrate flow rate in the actual filtration run. Correspondingly, with cloth B, which has a slightly greater resistance, the filtrate flow is slightly smaller in the actual filtration test. Whereas with cloth C, which has a much greater filter medium resistance, the filtrate flow is considerably lower. Thus, the filter medium resistance is inversely proportional to the filtrate flow in the normal filtration run. (Salmela & Oja, 2004)

10.1 SUMMARY

This chapter showed how a suitable filter medium for a certain starch filtration application can be searched easily and without any extra devices. The testing was based on the comparison of filtrate flow rates through the cake and the cloth in the normal filtration runs (Figure 10.2). Additionally, the microscopic investigation of the cloths was introduced (Figure 10.1).

Three different types of filter media were tested. The results showed that resistance of the media A and B increased when the number of filtration runs increased. This indicated that the small particles penetrate into the cloth and cause some blinding. The medium C was very tight, and its resistance decreased when the number of filtration runs increased. It was too tight for the 40 w-% cationic wheat starch slurry.

The choice of the filter medium for the starch filtration application was not easy or straightforward. Generally, it is important to know the concentration of the slurry to be filtered, and then which starch or starches are to be filtered. Additionally, the choice depends on whether it is preferable to maximise the capacity of the filter, or to get as clear a filtrate as possible (Salmela & Oja, 2004, Figures 6 and 7).

11 CAKE WASHING STAGE (Appendix III and IV: Salmela & Oja, 1999a and 2006)

Filter cake washing can be monitored experimentally by measuring the amount of impurities in the filtrate or in the cake. The monitoring method and the other operating variables affect the shape of the resulting washing curve. Another way to describe the washing performance is to use theoretical washing models. This chapter gives the experimental starch cake washing curves and their modelling.

11.1 MONITORING OF STARCH CAKE WASHING

In pressure filtration of starches the cake washing stage was monitored by measuring the amount of impurities retained in the cake, R_s, (Eq. 5.2) and the conductivity of filtrate, F, showing the instantaneous concentration of solute in the wash effluent. The samples from starch cakes were taken from the bottom part of the cake, from the top of the cake, and through the whole cake depth representing the average value, in order to follow the movement of the wash water in the cake. The chloride concentrations measured from the samples are plotted against the washing time in Figure 11.1 in order to obtain the washing curves. The corresponding figures in Salmela & Oja, 1999a, (Figures 2 and 3) show the amount of chloride retained in the cake, whereas in Figure 11.1 the fraction of chloride retained in the cake is shown.

When the two monitoring methods shown in Figure 11.1 are compared by showing the measured chloride contents as a function of time, it is evident, that the samples taken from the cakes indicate the chloride content of the final product, whereas the conductivity of the filtrate is a good indicator of the proceeding of the washing process. The value of the on-line conductivity begins to decrease, when the wash water penetrates through the cake. At this point the wash water has displaced most of the mother liquor, which remained in the cake from the beginning of cake washing stage. Thus, in practise, the washing can be monitored by measuring the conductivity of the filtrate as a function of time (Salmela & Oja, 1999a/Figure 5). The sudden decrease in the value of the conductivity indicates that the cake is mainly washed and the washing stage can be finished.

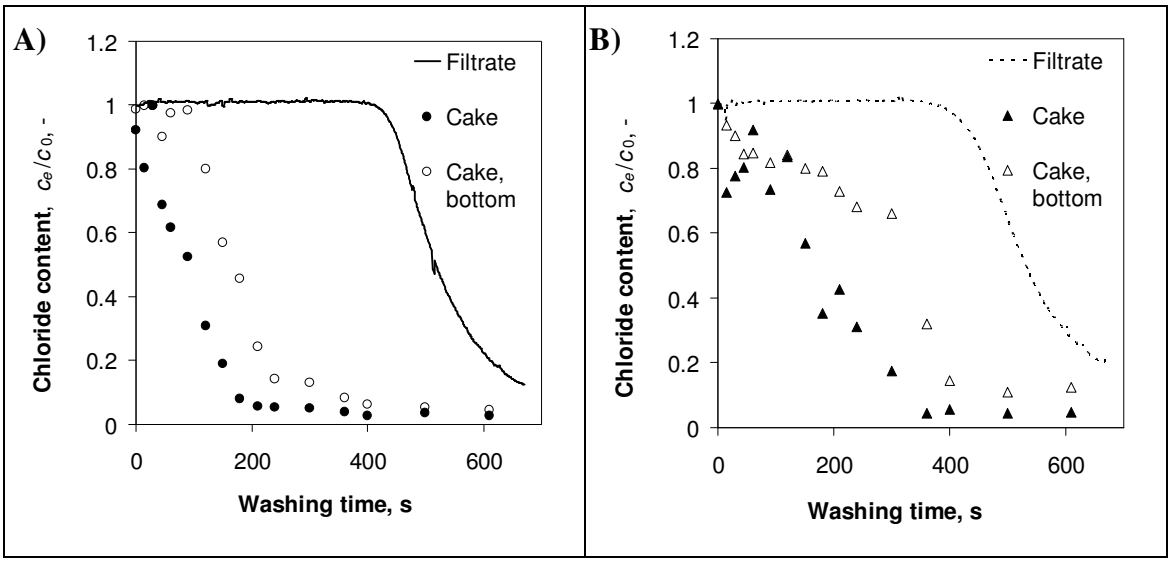

Figure 11.1 The native wheat starch (Native wheat RC 98) washing curves, showing the instantaneous concentration of solute in the wash effluent, F, and the fraction of solute retained in the cake, R_s. F is analysed by measuring the conductivity of the out-flowing filtrate as a function of the washing time, and R_s by taking samples from the cakes (representing the average solute content) and from the bottom layer of the same cakes, which are washed with different amounts of wash water. **A)** Cakes are washed after the filtration stage, and **B)** cakes are compressed (60 s at 16 bar) between the filtration and washing stage.

Of course, this in an ideal situation, and there is always a risk of bypassing of the wash liquid due to shrinking or cracking of the cake. However, these problems are rare in starch filtration experiments, and if they occur, they are easily noticed due to the sudden decrease and fluctuations in the conductivity of the filtrate.

If the samples taken from the different heights of the cakes are studied, it is evident that the impurities are very quickly washed away from the top part of the cake, whereas the bottom part may have a quite high concentration of impurities even at the end of the washing stage. The concentration of the sample representing the average value in the whole cake is directly related to the duration of the washing stage (Salmela & Oja, 1999a/Figures 2 and 3). This sampling shows that the progress of the flow of wash water is similar to plug flow. Hence, the sample representing the average chloride content in the cake gives the most realistic value for the cake chloride concentration.

When R_s is shown as a function of the washing time, the washing curves from the series of measurements, performed at a different filtration pressure and having different cake

heights, differ. If the difference needs to be ignored in order to compare the series of measurements, R_s can be shown as a function of the wash ratio, W_R, as shown in Figure 11.2. (Salmela & Oja, 2006 / Figure 4b). However, while the filtration conditions are ignored, the compression and/or displacement after washing can be seen from the curves. The washings in which the cake is compressed after washing (wheat1 and wheat2) have the steepest washing curves. It is evident that compression with a higher pressure enhances the washing process, because the wash water flows more effectively through the cake under the influence of the diaphragm pressing.

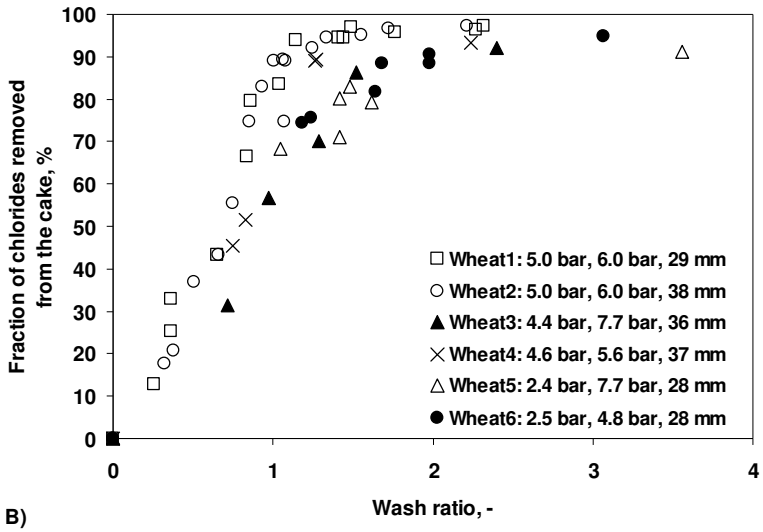

Figure 11.2 Typical experimental washing curves for saturated wheat starch cakes, when the washing results are shown as a function of the wash ratio. The name of the series is followed by: the filtration pressure, the washing pressure, and the cake height. (Salmela & Oja, 2006 / Figure 4b)

11.2 THE EFFECT OF OPERATING VARIABLES ON WASHING

11.2.1 Cake Dewatering before Washing

Figures 11.1 and 11.2 show also how the pre-wash compression dewatering affects on the shape of the washing curve. The shape depends on the amount of dewatering carried out on the cake before washing, and on the initial structure of the cake. In the case, when the cakes are dewatered before washing (case B in Figure 11.1), the R_s curves

slope more gently than in the case, when the cakes are not compressed (case A). This is due to a slower wash water flow rate in the cakes in case B, in which the porosity and the initial saturation of the cakes is decreased and the structure of the cakes made more homogeneous by compressing the cake with a diaphragm before washing.

Figure 11.3 shows how the magnitude of the pre-wash compression affects on the starch cake washing. This figure shows the fraction of chloride retained in the cake, whereas in publications of the author (Paavola, 1998, Salmela & Oja, 1999a/Figures 2-4, and 2000/Figure 2), the washing curve shows the amount of chloride retained in the cake. It is evident, that the compression before cake washing reduces the wash water flow through the native wheat starch cake, if the wash water pressure is kept constant. The higher the compression pressure the lower is the wash water flow through the cake. In compressed cakes, a smaller amount of wash water is sufficient to ensure the same purity of the starch cake, because the porosity of the cake, and hence the amount of filtrate in the cake is smaller. On the other hand, a slower wash water flow through the cake reduces the capacity of the filter. The capacity of the filter for fixed solute removal is better in the filtrations without pre-wash compression. Generally, in filtration of starches, the pre-wash compression can be employed, but it must be short and the pressure should not be too high. In that way, the pre-wash compression homogenises the structure of the cake and squeezes the filtrate above the cake into the cake, but does not affect the specific resistance of the cake.

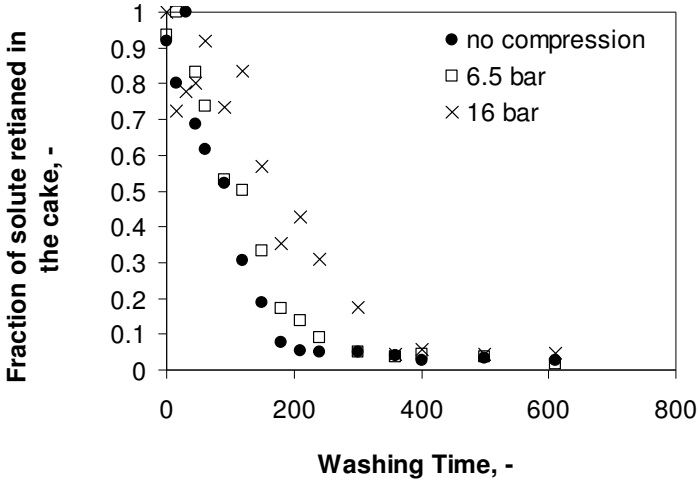

Figure 11.3 The effect of wash pre-wash compression on the washing of native wheat starch filter cakes (native wheat RC 98). The filtration pressure was 5 bar and two different compression pressures were tested.

11.2.2 Cake Thickness

The thickness of the forming starch filter cake depends on the height of the filter chamber, on the filtration pressure applied, and on the duration of the filtration stage. A lower filtration pressure causes the formation of lower cakes (Salmela & Oja, 2006, Figure 4A: Wheat5 and Wheat6), which in turn decreases the capacity of the filter. On the other hand, lower cakes with lower resistances are quicker to wash, which in turn minimises the effect of the low cake heights. Washing takes a longer time in the thicker cakes, because there is more mother liquid to be displaced. On the other hand, the capacity of the filter increases when the thicker filter cakes are formed, because one filter cycle can then produce more pure starch (Paavola, 1998, Salmela & Oja, 1999a / Figures 2 and 3). It is important to find an optimum cake formation for every starch type, which will maximise the capacity of the filter and guarantee the structure of the cake to be favourable for washing. This can be achieved by comparing the particle size distribution of the slurry to be filtered, the typical average specific cake resistance of that starch type and the washing data from former filtration experiments performed with this starch.

11.2.3 Wash Liquid Feed

The effect of wash liquid feed in starch cake washing is studied by the author (Paavola, 1998, Salmela & Oja 1999a and 2000). The tests are performed by keeping the test conditions unchanged, and only altering the wash water pressure. The results are shown in Figure 11.4. This Figure shows the fraction of chloride retained, whereas in Salmela & Oja, (1999a and 2000) the washing curves show the amount of chloride retained in the cake. According to Figure 11.4, the washing is more effective with a higher wash water pressure. The cake is washed slightly faster, which in turn enhances the capacity of the filter, because the cycle time is reduced due to the shorter washing stage. Normally the wash water pressure used in wheat starch filtrations is one bar greater than the filtration pressure. If the wash water pressure is 3 bar greater than the filtration pressure, the benefit of the increased wash water liquid feed is evident. A further increase in wash water pressure from 3 to 5 bar greater than the filtration pressure, is no longer more beneficial (Salmela & Oja, 1999a / Figure 4, 2000 / Figure 2b).

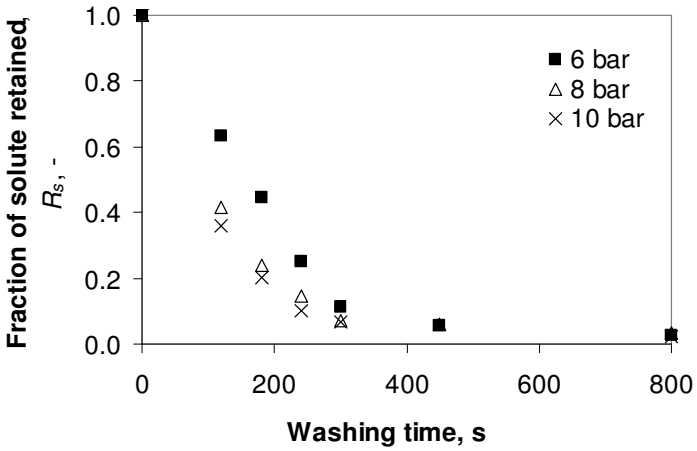

Figure 11.4 The effect of wash water pressure on the washing of wheat starch filter cakes (Native wheat RC 98). The filtration pressure was 5 bar.

The amount of wash water needed to reach a certain level of purity is slightly greater when a greater wash water pressure is used, although the amount of the mother liquid to be displaced is the same independent of the wash water pressure. This is possible due to the shorter time for the mass transfer processes in the cake with the higher wash water flow rates (Salmela & Oja, 2000).

The wash water flow rate depends also on the starch grade. The higher the average specific resistance of the starch cake, the higher the wash water pressure needed in order to wash the cake effectively. The washing of potato starch, which has the lowest specific cake resistance of all starches, is quickest, whereas the washing of wheat starch is slowest. So, the higher α_{av}, the longer the time needed to wash the cake or a higher wash water pressure has to be applied. This can be seen from Figures 5-7 in Salmela & Oja (2006) by comparing the shapes of the washing curves of different starches.

11.3 MODELLING OF STARCH CAKE WASHING

When the filter cake washing is modelled, the common way to represent the results is to show the amount of impurities retained in the cake as a function of wash ratio, W_R (q. 5.3). For the calculation of W_R the volume of residual filtrate in the cake at the start of the washing, V_{f0}, can be estimated from the mass balance of the filtrate at the beginning

of the washing stage, but this is a laborious task for a series of measurements, which contain hundreds of individual measurements (Salmela & Oja, 2006). Hence, it is not practical in the monitoring of an industrial cake washing process. In this study the amount of filtrate in the cake at the start of washing is assumed to be equal to the void volume of the cake after the test. This may sometimes be quite a rough estimation, because the cake is compressed or dried with air after the washing stage, but this study establishes that for the washing of starch filter cakes it is suitable, and facilitate the analysis of the cake washing data. The volume of the residual filtrate in the cake at the start of washing, $V_{f\,0}$, is calculated from the known solid content of the cake, s, the height of the cake, L, and the filtration area, A, which are measured after the whole filter cycle, as follows:

$$V_{f\,0} = (1 - s)AL \tag{11.1}$$

The starch cake washing results are modelled with the dispersion model and with the modified exponential decay model (Salmela & Oja, 2005b and 2006). The dispersion model, which commonly appears to fit many experimental data for the washing of saturated cakes (Wakeman & Attwood, 1988 and 1990) is used in its original form, Eq. (5.8). The exponential decay model has been applied to short washings (Rhodes, 1934), and to longer washings (Marecek & Novotny, 1980) by dividing the washing period into different stages and by establishing different values for the parameter w_c for different parts of the washing curve. However, in this thesis, the exponential decay model, Eq. (5.13), is applied to the starch washing results obtained by measuring the content of the solute remaining in the cake, R_s, (Salmela & Oja, 2006) in the form

$$R_s = \exp(-w_c W_R) \tag{11.2}$$

The experimental results and the curves given by the dispersion model and the exponential decay model are shown in Figure 11.5. The uncertainty of the modelling was estimated by resolving w_c from Equation (11.2) and differentiating Equation (11.2) regarding R_s and W_R, which was resolved from Equation (11.1). Hence, the parameters in W_R were the solid content, s, and the height of the cake, L, as well as the amount of wash water used V_w. The uncertainty of sampling was estimated with the fundamental

sampling error model (Pitard, 1989, Gy, 1998). The total standard deviation of the sampling of the cakes was 2.50% (Salmela & Oja, 2006), which means that the measured value of R_s can change ± 2.5%. The average cake height and the average cake solid content of each series of measurements was calculated as an average of the measured heights and solids contents of the cakes in that series of measurements. Then the standard deviation and the standard error with a confidence interval of 95% and with the assumption of normal distribution for these parameters in each series of measurement were calculated. They are shown in Table 11.1 as well as the average absolute error of the parameter w_c.

The corresponding experimental curves are obtained from every series of measurements of starch washing tests (Salmela & Oja, 2006). The shape of the experimental curve changes due to the starch type, as can be seen from Figure 11.5, and due to the filtration conditions. Hence, the value of the dispersion parameter, D_n, in the dispersion model and the washing parameter, w_c, in the exponential decay model changes too. Additionally, the washing of potato starch cannot be modelled with the dispersion model, because the curve obtained from the potato starch washing is outside the area of the dispersion model.

Both of the washing models fit well with the experimental results when about 75% of the chloride-impurity is removed from the washing of starches (Salmela & Oja, 2006 / Figures 5-7). It is typical for both of the models that they fail during the short starch cake washings, when the wash ratio is smaller than one. These short washings are usually ineffective, because they reduce the capacity of the filter without affecting considerably the quality of the starch product. Hence, the typical wash ratio used in starch cake washing is greater than one, and therefore the dispersion model and the exponential decay model can be used for describing the behaviour of starch cake washing. The only parameter, which is needed, is the dispersion parameter, D_n, in the dispersion model or the washing parameter, w_c, in the exponential decay model. The exponential decay model takes the tail of the experimental washing curve better into account than the dispersion model does as can be seen from Figure 11.5 or from Figure 5-7 in Salmela & Oja (2006). Therefore, it is recommended prior to the dispersion model. The typical values of the model parameters, their errors with 95% confidence

interval, and the typical experimental conditions used in starch washing tests are shown in Table 11.1.

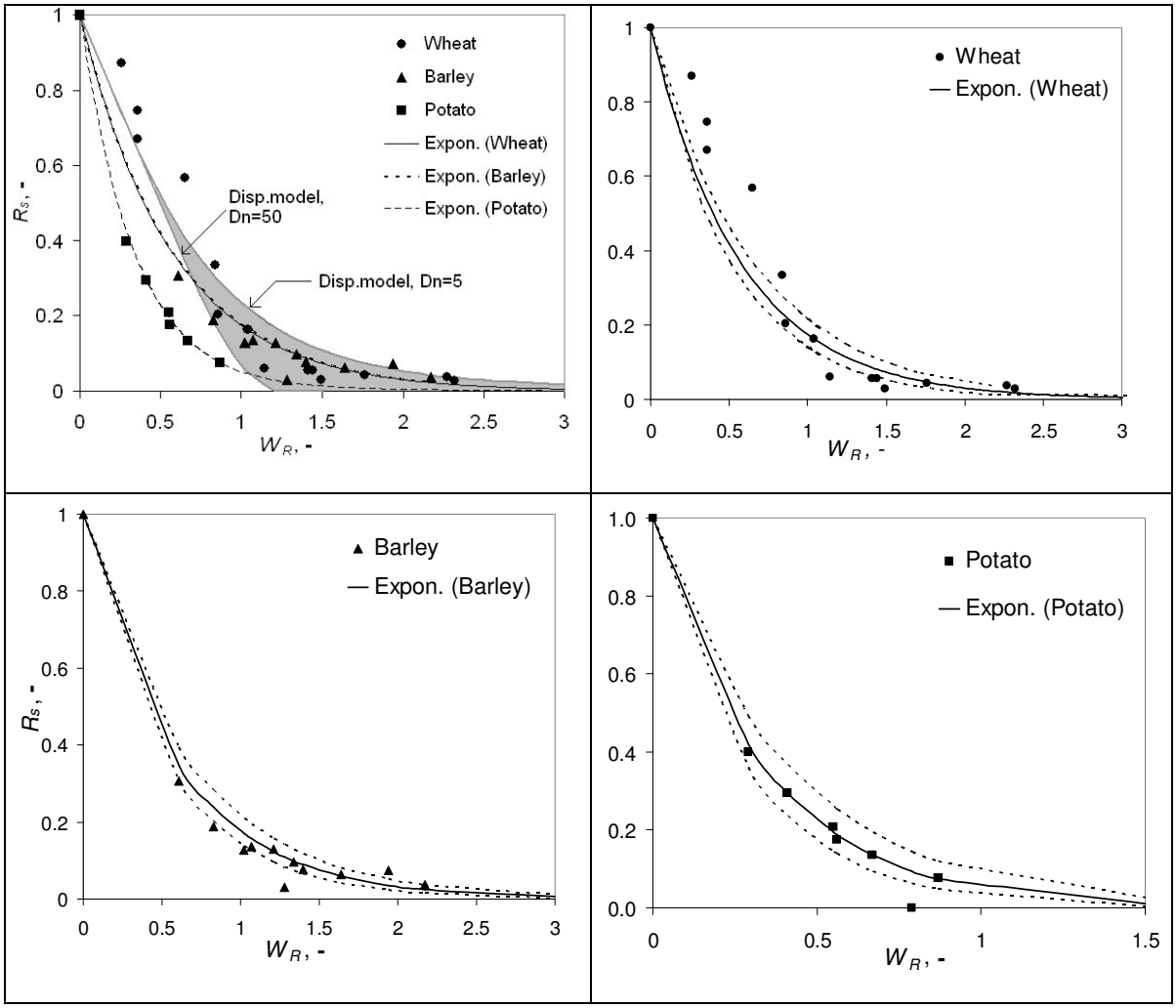

Figure 11.5 *First*: Washing results from native wheat, barley and potato starch washings, and the corresponding dispersion model curves with different D_n-values as well as the corresponding fitted exponential decay model curves. *The others*: Washing results from the tests with each starch grade, the corresponding exponential decay model curve, and the broken line curves showing the uncertainty of the exponential decay model with 95% confidence interval. (Salmela & Oja, 2005b / Figure 2, 2006 / Figure 5-7: wheat1, barley2, and potato4)

Table 11.1 The conditions during the filtration cycles in the cake washing tests, the average properties of the formed cakes with their standard errors with 95% confidence interval, and the values of the washing parameters with their standard errors with 95% confidence interval for different series of measurements of native starches (Salmela & Oja, 2006 / Table 2).

STARCH *	FILTRATION		WASH	PRESSING		AIR	CAKE		PARAMETERS	
	Δp, bar	t, s	Δp_w, bar	Δp_c, bar	t, s	t, s	L, mm	s, %	D_n, -	w_c, -
Wheat 1	5.0	120	6.0	6.5	60	110	29 ± 0.4	62.3 ± 0.33	5-10	1.75 ± 0.22
Wheat 2	5.0	180	6.0	6.5	60	240	38 ± 0.4	60.5 ± 0.12	5-10	1.80 ± 0.16
Wheat 3	4.4	120	7.7	-	-	180	36 ± 1.1	58.8 ± 1.73	5	1.05 ± 0.14
Wheat 4	4.6	120	5.6	-	-	180	37 ± 0.4	61.2 ± 0.32	1-5	1.35 ± 0.12
Wheat 5	2.4	120	7.7	-	-	180	28 ± 1.4	57.0 ± 1.96	1-5	0.84 ± 0. 16
Wheat 6	2.5	120	4.8	-	-	200	28 ± 1.1	60.6 ± 0.54	5	1.08 ± 0.14
Barley 1	0.87	90	2.6	-	-	90	24 ± 1.4	63.3 ± 0.43	5	1.31 ± 0.20
Barley 2	0.92	95	5.6	-	-	90	37 ± 1.2	62.2 ± 0.16	5-10	1.73 ± 0.21
Barley 3	0.92	75	2.3	-	-	120	28 ± 1.6	62.9 ± 0.29	5	1.22 ± 0.18
Barley 4	0.90	125	2.0	-	-	120	38 ± 2.2	61.8 ± 0.34	5-10	1.63 ± 0.21
Potato 2	1.05	90	4.1	15.2	60	-	50 ± 1.3	59.7 ± 2.53	-	4.33 ± 0.86
Potato 4	0.57	70	3.8	15.5	60	-	44 ± 3.4	52.8 ± 1.71	-	2.98 ± 0.53
Potato 5	0.66	100	4.0	15.8	90	-	66 ± 2.5	55.7 ± 0.59	-	2.87 ± 0.26

* Wheat1-2: Native Wheat RC 98, Wheat 3-6: Native wheat RC 12/99, Barley1: Native Barley RC 6/99, Barley 2-4: Native barley RC 12/99, Potato1-5: Native Potato RC 12/99

According to Table 11.1, each starch grade seems to have a typical D_n-value or range of values, which is quite independent of the filtration conditions. The w_c-parameter seems to be more sensitive to the experimental conditions, even though every starch grade has a typical range of w_c –values, and the values of the measures of dispersion are sufficiently small and even. Among the series of wheat starch measurements, the cakes which are compressed after washing have a greater w_c-value than the non-compressed cakes. Among the barley starch series, the cake height seems to affect the w_c-value so that the thicker cakes have higher w_c-values. Among the potato starch series, the filtration pressure is the affecting factor so that with a higher filtration pressure the w_c-value is higher. All in all, when a typical w_c- or D_n-value in the prevailing conditions is known, both of the models can be used for the estimation of starch cake washing performance.

If some of the filtration or washing conditions change, the direction of the change in w_c-value can be predicted on the basis of the former experimental data. The increase in the value of the parameter w_c in the exponential decay model indicates:

- a faster washing process
- a thicker cake
- a higher washing pressure in barley starch filtrations
- a lower washing pressure in wheat starch filtrations
- a higher filtration pressure and
- steeper washing curve.

11.4 SUMMARY

This chapter introduced the different ways for estimating the performance of starch cake washing (Figure2 11.1 and 11.2). It was shown that the most reliable method is to take a sample from the filter cake and to analyse its average impurity content. The other way, which can be used during the washing test, is to monitor the conductivity of the out-flowing filtrate: the displacement washing process is mainly finished, when the conductivity of filtrate begins to decrease.

The effect of the operating variables, like compression dewatering before washing (Figure 11.3), the thickness of the cake, and the wash liquid feed (Figure 11.4) was also examined experimentally. Short and gentle compression before washing was ascertained to be favourable, the thicker cakes to increase the capacity of the filter, and the slightly higher wash water pressure to intensify washing.

The dispersion model (Eq. 5.8), which is commonly regarded as the main washing model, and the modified exponential decay model (Eq. 11.2) were applied to the starch cake washings. They both fitted well during the later part of starch cake washing, and they were ascertained to be suitable for the easy modelling of the starch cake washing stage in pressure filtration. Finally, the changes in some parameters were examined for the prediction of washing results for conditions, which had not been tested.

12 CAKE DEWATERING STAGE (Appendix V: Salmela & Oja, 2005a)

There is almost no literature available concerning the dewatering of starches. The only one is the patent of Stocker (1996) which concerns a process for enhancing the dewatering of starch in a vacuum filter, in a plate and frame filter, and in a centrifuge. In their process, a composition comprising polybutene in combination with at least one surfactant having a melting point or pour point lower than 20°C was added to the starch slurry. The use of this composition among starch slurry increased the amount of filtrate and the drainage rate of the sample.

12.1 COMPRESSION DEWATERING

Before the compression dewatering stage in the starch filtration process can be analysed, the actual starch point of compression has to be known. Additionally, it is good to know the factors affecting expression. This chapter introduces the determination of the actual start points of compression for different starch types, the effects of the operating variables on expression, and the modelling of the starch expression stage.

12.1.1 The Start Point of Compression

The transition from filtration to consolidation appears if the filtration pressure differs from the compression pressure. Usually the filtration has ended, the cake formed and subjected to compression, but the filtration pressure is still dominant or, alternatively, the filtration is continued at a higher pressure, the compression pressure, until the cake is formed. In Salmela & Oja (2005a), four different methods for the estimation of the transition from filtration to consolidation were compared by filtering different starches (Salmela & Oja, 2005a/Figures 1-4, Table 3). The methods were also introduced in Chapter 6.2.2. All of the four methods were found to be suitable for starch applications, but method three was the most straightforward to use, if the work cylinder pressure data was available. Methods two (dt/dV against V) and four ($-\Delta L/\Delta(t^{0.5})$ versus t) gave the same value for the transition point. This value was a couple of seconds greater than the

value given by method three, but the influence of the difference on the compression calculations and modelling was minimal, as shown in Salmela & Oja (2005a / Figure 5). Method one (t/V versus V) gave slightly greater values for the transition point and the exact point was more difficult to find, because the change in the slope was not very sharp. Method four was found to be the second most straightforward method, and it should be used in starch filtration applications, if the work cylinder pressure data is not available. Practically, the selected method had a very minor effect on the value of the transition point, and therefore on the values of the experimental parameters in the dewatering models. However, the correct choice of the transition point was critical to the success of the modelling of starch compression dewatering (Salmela & Oja, 2005a).

12.1.2 Factors Affecting Expression

The consolidation stage in pressure filtration of starches is easily described with the consolidation curve showing the consolidation ratio, U_c, (Eq. 6.8) against the square root of the dewatering time (Salmela & Oja, 2005a / Figure 6). The shape of the consolidation curve depends mainly on the following factors:

Suspension. From Figure 12.1 (Salmela & Oja, 2005a / Figure 6) it is clearly seen, that the consolidation rate of wheat starch, which contains a remarkable fraction of small particles, is the slowest of all starches. The consolidation rate of potato starch is the highest, and comparing all the starches, consolidation becomes slower as particle size is reduced.

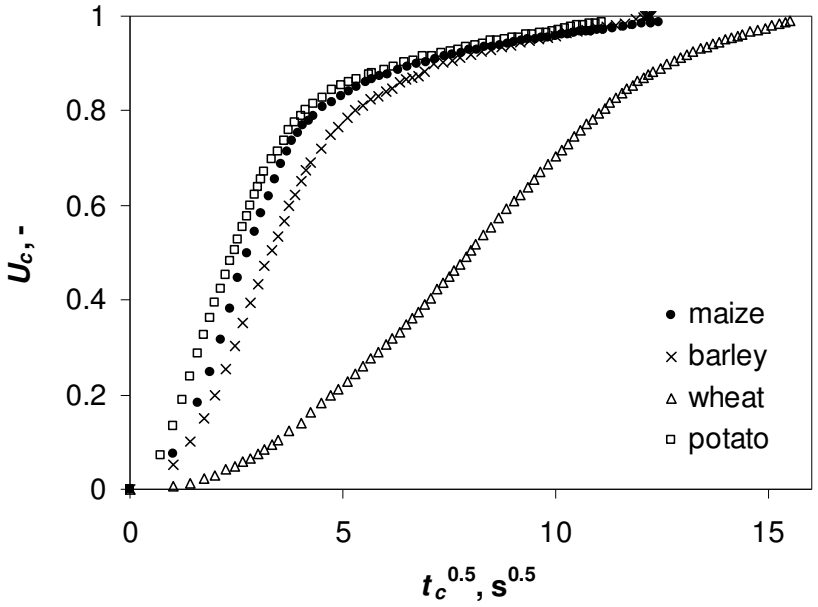

Figure 12.1 The consolidation of different native starches* in the piston press test filter. The starch slurries were filtered at 5 bar pressure and consolidated at 16 bar. The consolidation was started automatically, when cake formation occurred. (results from: Salmela & Oja, 2005a / Figure 6).

- Maize: Amylum Meritena 100, Barley: RC 6/99, Wheat: RC 1/99, Potato: 12/99

pH. According to Figure 8.5 it is obvious that the NaCl content of the slurry affect the separation behaviour of starches by influencing the stability of particles in the slurry. Typically the salt content in the slurry to be filtered is 2% chlorides per dry starch or less, and it remains quite unchanged from lot to lot. However, it is important to know if the salt content changes, because it affects on the stability of particles, and hence the filtration behaviour of the slurry.

Filtration pressure. The effect of filtration pressure on the average cake porosity is greatest at low pressures, between 0 and 3 bar (Wakeman, 1975), which are typical in starch filtration applications. The cake porosity, in turn, affects the initial structure of the cake in compression. In filtration of starches, the filtration pressure has the strongest effect on the structure of the wheat starch cake, because wheat starch contains a fraction of small (2…10 μm) particles, which fill some of the voids in the cake. The particle size distributions of other starches are more equal in size, and hence the porosity of the cake remains higher although the filtration pressure increases.

Cake Washing. If the cake is washed after the compression stage, some points have to be taken into account. In Chapter 11.2.1, Figure 11.3, the effect of pre-wash compression on cake washing was discussed (Paavola, 1998, Salmela & Oja, 1999a and 2000). The washing curves of three different test series were compared: 1) cakes, which are filtered and washed, 2) cakes, which are filtered, compressed at 6.5 bar and then washed, and 3) cakes, which are filtered, compressed at 16 bar and then washed. It is evident that the pre-wash compression increased the specific cake resistance and reduced the porosity of the filter cakes, and hence reduced the wash liquid flow rate through the cake. The higher the pre-wash compression pressure, the greater was the decrease in the filtrate/wash water flow rate. If there is a need to compress the cake before washing, it is desirable to compress it at a pressure, which is as close to the filtration pressure as possible.

12.1.3 Modelling of Consolidation

Different theories of expression, which are introduced in Chapter 6.2.4, were tested in order to find an easy and proper way to interpret the experimental data from starch dewatering and to select the correct model(s) for the dewatering of certain starches in a pressure filter (Salmela & Oja, 2005b and 2006). The models are the Terzaghi model (Eq. 6.9), the Terzaghi-Voigt models with two and three parameters (Eq.'s 6.12 and 6.13), which are simplifications of the complete Terzaghi-Voigt model, and the Shirato model (Eq. 6.19 and Fig. 6.2). The simplified Terzaghi-Voigt models can be used with confidence, if the dewatering rate due to the secondary consolidation is much smaller than the rate due to the primary consolidation (Shirato et *al*., 1986c, Sedin *et al*. 1997). Therefore, the three time constants are defined and ascertained to fulfil the requirements (Eq.'s 6.14-6.18).

In the Terzaghi model, the only parameter, the modified consolidation coefficient, C_e, is determined from the early stage of the experiment, obtained from the $\ln(1\text{-}U_c)$ versus t_c curve. For wheat starch, which needs the longest cycle time, the early stage is chosen to be 0→100 s, and for other starches 0→30 s. In the Terzaghi-Voigt model with two parameters, B and η are determined from the latter stage on the plot $\ln(1\text{-}U_c)$ versus t_c. B is measured from the intersection between the curve and the y-axis whereas η from the

slope of the curve. For wheat starch, the latter stage is chosen to be 100→150 s and for other starches 30→100 s. In the Terzaghi-Voigt model with three parameters, the values of the parameters C_e, B and η are determined using non-linear curve fitting based on the least squares method. The initial estimates for the parameters used in the curve fitting are those measured from the Terzaghi model and the Terzaghi-Voigt model with two parameters. In the Shirato model, the consolidation behaviour index is determined by plotting the experimental consolidation curve to Figure 6.2. (Salmela & Oja, 2005a)

The parameters of the expression models determined from the Terzaghi or simplified Terzaghi-Voigt models are found to differ from the parameters of the complete Terzaghi-Voigt model, when different materials are filtered (Sedin, 2003a). Sedin *et al*. (2002) observed that the consolidation coefficient, C_e, was also slightly pressure dependent in the dewatered magnetite slurry. The dependence was greater at the iso-electric point than it was at -40 mV zeta-potential.

The parameters for different starches are measured by Salmela & Oja (2005a / Table 5), and given in Table 12.1. These results show that also the parameters given by the simplified Terzaghi-Voigt models differ depending on the amount of the parameters in the model. The variances of the estimated parameters are found to be satisfactory in the study of Salmela & Oja (2005a), as well as in the study of Sedin (2003a). The measurement uncertainties based on the experiments of this study are measured as follows:

- Four parallel tests with native wheat starch (RC 1/99) were performed
- Each test data was analysed with four different methods for measuring the transition from filtration to consolidation
- Each test data was modelled with the Terzaghi model, the Terzaghi-Voigt model with two and three parameters, and the Shirato model, and the values of the model parameters were measured
- Each parameter in the models had 16 values (4 samples, 4 transition points)
- The standard deviation and variance for the parameters were calculated
- The standard error was calculated and the value for the estimation uncertainty with 95% confidence interval was given.

- The percentage error with 95% confidence interval from the average parameter value was assumed to be valid for all starch types, because the native wheat starch, which was used in the estimation, had the most uncertain behaviour during the estimation. The consolidation of other starch types was easier to describe with the models. The percentage errors are given in Table 12.2.

The parameter C_e is the easiest to estimate from the Terzaghi model, and it is quite insensitive to process changes. It also has the lowest standard deviation of the parameters. When C_e is estimated from the piston press test data with the Terzaghi model, its value can be used in estimating the consolidation in a filter press if the start point of consolidation is chosen according to the methods introduced in Chapters 6.2.2 and 12.1.1 (Salmela & Oja, 2005a / Figure 8). The parameters B and η are strongly correlated to each other and quite sensitive to the initial estimates for the values used in the curve fitting (Salmela & Oja 2005a /Table 4). η has the highest standard deviation of all of the parameters and it is very sensitive to the mathematics of the data analysis.

Table 12.1 Total volume of dry solids per unit filtration area, and expression parameters for different starches under a filtration pressure of 5, 3 or 1 bar and compression pressure of 16 bar. The values are measured with the piston press filter.

Starch*	Filtration Pressure	Dry Solids	Terzaghi	Terzaghi-Voigt 2 - parameters		Terzaghi- Voigt 3-parameters		
	Δp bar	ω_0 $m^3 m_2^-$	C_e $m^2 s^{-1}$	B -	η s^{-1}	C_e $m^2 s^{-1}$	B -	η s^{-1}
Native potato	1	0.0195	1.65×10^{-5}	0.260	0.0220	2.74×10^{-5}	0.257	0.0223
Cationic potato	5	0.0195	6.66×10^{-6}	0.522	0.0131	1.69×10^{-5}	0.528	0.0133
	3	0.0202	1.02×10^{-5}	0.252	0.0168	1.45×10^{-5}	0.283	0.0287
Native barley	5	0.0208	1.05×10^{-5}	0.341	0.0208	1.83×10^{-5}	0.342	0.0223
Native maize	5	0.0218	1.50×10^{-5}	0.224	0.0170	2.48×10^{-5}	0.213	0.0151
Native wheat	5	0.0216	2.19×10^{-6}	2.232*	0.0210	4.68×10^{-6}	1.876**	0.0158
Cationic wheat	5	0.0209	4.98×10^{-6}	0.345	0.0140	4.93×10^{-6}	0.370	0.0268
	3	0.0210	2.72×10^{-6}	0.656	0.0136	2.93×10^{-6}	0.662	0.0166

* Native potato RC 12/99, Cationic potato 150LP, Native barley RC 6/99, Native maize: Amylum Meritena 100, Native wheat RC 1/99, Cationic wheat 215LV.

** unnatural value (should be <1, since B is the ratio of the creep deformation to the total deformation)

Table 12.2 The percentage standard errors with 95% confidence interval for the expression parameters in modelling of starch cake consolidation. The values are obtained by filtering native wheat starch at 5 bar and compressing it at 16 bar. It is assumed that the error values hold for all the starches.

	Terzaghi	Terzaghi-Voigt 2 - parameters		Terzaghi- Voigt 3-parameters		
	C_e	B	η	C_e	B	η
The standard error with 95% confidence interval	2.5 %	2.5 %	1.9 %	56.4 %	3.0 %	12.8 %

The Terzaghi model gives a good description of starch cake consolidation during its early stages and can be used for the modelling of the initial expression phase (Figures 12.2 and 12.3). However, poor agreement is a common problem during the later stages, and the model fails to predict the expression behaviour of many materials in the final expression phase, because the model assumes that the consolidation coefficient, i.e. compactibility and specific filtration resistance, is independent of applied pressure (Sedin, 2003a). However, The Terzaghi model is found to be adequate in the modelling of starch compression dewatering during the first 65…100 seconds of expression (Salmela & Oja, 2005a / Figure 7). This is often adequate in the modelling of industrial filtration applications, because the duration of the compression has to be kept short in order to maximise the capacity of the filter. The benefit is that the Terzaghi model is easy to use, because its only parameter, C_e, is quite insensitive and the value taken from the piston press test data can be used in estimating for larger scale filter press data (Salmela & Oja, 2005a / Table 4, Figure 8). The only variables which need to be taken into account in modelling are the solid content of the slurry and the height of the suspension in the beginning of the compression. The value of C_e can be taken from Table 12.1, if the filtration and compression pressures are in the same order of magnitude as in the experiments shown in Table 12.1. If the pressures are greatly altered, the value of C_e has to be experimentally measured, because it is slightly pressure dependent (Sedin *et al*., 1997). However, even the 20% change in the value of C_e, has almost no effect on the modelling curve given by the Terzaghi model, which shows that the Terzaghi model is robust enough for the easy modelling of compression.

Terzaghi-Voigt models. To overcome the problems of the Terzaghi model, secondary consolidation has to be taken into account (Wakeman & Tarleton, 1999). This can be done by using the Terzaghi-Voigt models. The value of B depends on the applied pressure and the initial average void ratio, whereas the value of η is virtually constant (Sedin *et al*., 1997). In modelling of the compression dewatering of starches, the restrictions concerning the time constants (Eq.'s 6.14-6.18) are assumed to hold (Salmela & Oja, 2005a). This means that the dewatering rate due to the secondary consolidation is assumed to be clearly smaller than the rate due to primary consolidation. Figure 12.2 shows that the simplified Terzaghi-Voigt model with two parameters fails totally in the early part of the compression, but describes well the later stage of the consolidation onward from the point at which the Terzaghi's model starts to fail. The Terzaghi-Voigt model with three parameters gives a good fit to the whole dewatering period, and it can be used for the prediction of longer starch dewaterings. The problem in that model is the three experimentally measurable parameters, and the sensitivity of the measurement of these parameters, which make the use of this model difficult (Salmela & Oja, 2005a).

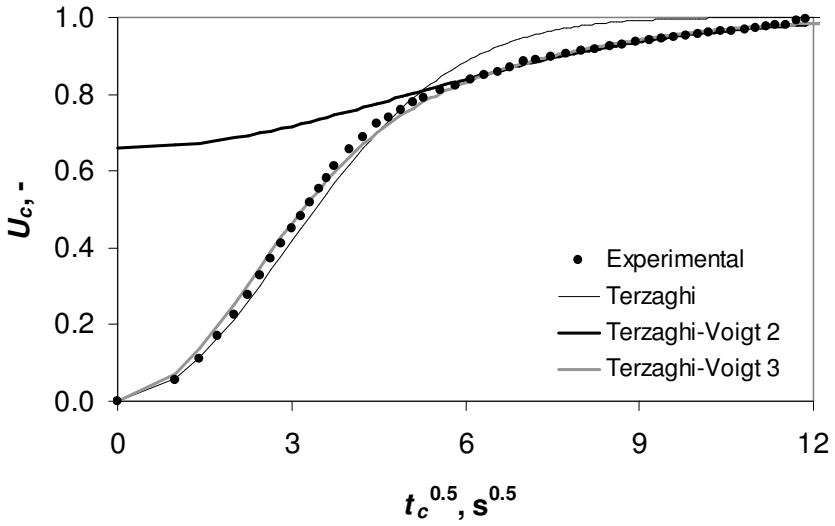

Figure 12.2 The experimental consolidation ratio (Eq. 6.8) and the consolidation ratios calculated from the Terzaghi model (Eq. 6.9), the Terzaghi-Voigt model with two parameters (Eq. 6.13) and the Terzaghi-Voigt model with three parameters (Eq. 6.12) in the filtration of native barley starch (RC 6/99) in a piston press test filter. The filtration pressure was 5 bar and the compression pressure 16 bar (corresponding figure for cationic wheat starch 215LV in Salmela & Oja, 2005a / Figure 7).

Shirato's model. The suitability of the Shirato's consolidation model for the modelling of starch consolidation is shown in Salmela & Oja (2005a / Figure 9). The model fails to predict the consolidation behaviour in the beginning of the consolidation, as was expected by Shirato *et al*. (1986b). However, the model fits well onward of the point where the Terzaghi model starts to fail (Figure 12.3). The value of T_c for the Shirato model in the modelling of starch dewatering, can be calculated by using the value of C_e measured from the Terzaghi's model (Salmela & Oja, 2005a). The Shirato model is easy to use for a quick prediction of the consolidation of starch filter cakes.

Practicality. From both an industrial and a theoretical point of view, it is preferable that a model is as simple as possible. The simpler the model, the easier it is to estimate and interpret its parameters (Sedin, 2003b). The most practical way of presenting full scale performance is to carry out small-scale tests and scale-up on the area. It is important to optimise the process variables, in order to achieve the best performance from the equipment (Carleton & Salway, 2004). For starches, the most practical way is to use the Terzaghi model for the earlier part of the consolidation and if needed, the Shirato model for the later part, as shown in Figure 12.3 (Salmela & Oja, 2005a and 2005b). The range of the measurement uncertainty in the Terzaghi model is not visible, because the Terzaghi model curve changes very little with the confidence interval of 2.5%, which is given in Table 12.2. Even a tenfold confidence interval of 25% has a very minor effect on the curve. According to Shirato *et al*. (1986a), the maximum percentage error in U_c calculated by the Shirato model with index υ=2.85 compared to the Terzaghi model is only 0.60%, and the secondary consolidation is taken into account. A typical value of υ for starches is around two.

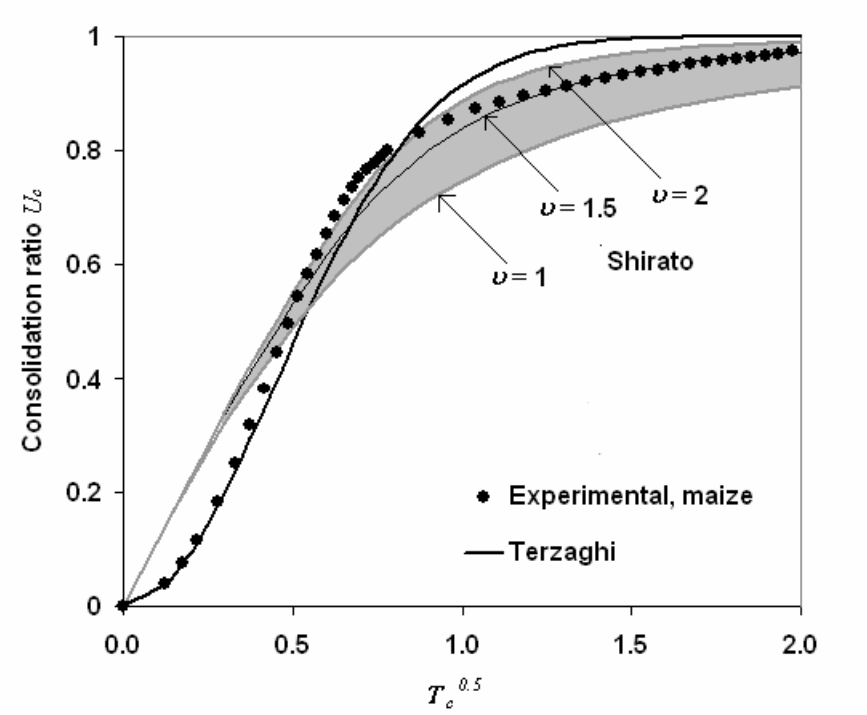

Figure 12.3 The experimental consolidation ratio (Eq. 6.8) and the consolidation ratios calculated from the Terzaghi model and from the Shirato model (with a range of υ values) for native maize starch (Amylum Meritena 100), which was filtered at 5 bar and consolidated at 16 bar. (Salmela & Oja, 2005b / Figure 3).

12.2 DISPLACEMENT DEWATERING

Starch cakes are composed of small particles, and some of the starch grades are slightly compactible. Hence starches are easily assumed to be difficult to dewater by air displacement. In this thesis, the main emphasis is given to the compression dewatering of starch filter cakes. The displacement dewatering is discussed only shortly, and its research and development are left outside the scope of this thesis. However, this does not mean that the displacement dewatering is not relevant for starch separation.

The air-drying of starch filter cakes is found to be quite ineffective and time-consuming in filtration of native wheat starch in the filter press, when air-drying is performed after the diaphragm pressing stage. Air-drying decreases the capacity of the filter, and has a very minor effect on the final solid content of the cake. It increases the solid content of the starch cake only about 0.1 – 1.5% when compared to the solid contents obtained merely with compression dewatering.

In some filtration tests with native starches the air-drying is applied after the filtration or cake washing stage without compressing the cake. With native wheat starch a couple of percent higher cake solid content is obtained with compression dewatering (about 62-65 %) than with air-drying (59-61%). With barley starch the solid content of the cakes was about 62-63% after air-drying, and slightly smaller with compression dewatering. With potato starch the solid content of the cake was about 63% after air-drying and 60-61% after compression dewatering. However, the air-drying needs a longer time to achieve this final cake solid content than does the compression dewatering, and this affects the capacity of the filter.

The displacement dewatering is introduced also in the sense that sometimes a short air-drying before washing can be used in order to empty the pipes of the filter, and to create 'a pocket of air' above the cake in the filter chamber. These in turn affect the structure of the starch filter cake, and hence washing too. The small amount of air in the filter chamber helps the wash water to flow into the filter chamber and into the cake, and facilitate the release of the cake. During the short air-dryings the air arrives above the cake, but has no time to further penetrate into its voids.

12.3 SUMMARY

This chapter introduced the starch filter cake compression and displacement dewatering results. Four different methods for determining the transition from filtration or washing to consolidation in starch cake filtration were introduced. It was shown that the selected method had almost no effect on the value of the transition point and on the values of the experimental parameters in the dewatering models. However, the choice of the starting point is critical to the success of the modelling of starch cake consolidation.

The factors affecting the starch cake expression were tested: properties of the slurry to be filtered; the particle size and the compressibility of the cake (Figure 12.1), pH of the slurry, the filtration pressure, which affect the structure of the cake to be compressed, and the demands that the washing stage after compression lay on the compression.

The applicability of theoretical models for the modelling of starch cake dewatering was presented (Figures 12.2 and 12.3) and discussed. These models were the Terzaghi model, the Terzaghi-Voigt models with two and three parameters, and the Shirato model. Finally, an overview of starch cake dewatering by air displacement was given.

13 SOFTWARE FOR FILTRATION DATA ANALYSIS (FDA)

(Appendix VI: Salmela & Oja, 2005b)

During the pressure filtration cycle, hundreds or thousands of rows of data are gathered. The analysis of this data is laborious and there is a risk of miscalculations because of the large amount of data. Also the utilisation of the models, which are introduced in the preceding chapters, is laborious and sensitive to errors, and it has to be done independently for every filtration test data. Therefore, in this study, software is developed for easy processing and modelling of the data from the filtration tests. The software is based on the theories given by this thesis and it should help in processing test data and in modelling the filtration cycle by gathering the theories of different process stages together. The structure of the software is shortly described in Salmela & Oja (2005b) and shown in Figure 13.1.

From the normal filtration run, the mass of filtrate and the filtration and compression pressures are obtained as a function of time. This data and the basic constants related to the filter, filtrate and solid material are given for the software as input. These input values needed by the software are shown in Figure 13.1 in the INPUT-box on the left.

When the input values and the data from the filtration run are given for the software, it draws a figure in which the mass of filtrate is shown as a function of time on the right y-axis, and the filtration and compression pressures as a function of time on the left y-axis. The user of the software is able to choose the process stages, which are included in the test run, by clicking them from the list given by the software. Then the user determines the start and the end point of each included process stage from the figure by moving the vertical lines showing these points on the figure. This stage in the data analysis is shown in Figure 13.1 next to the INPUT-box.

Then the software draws the experimental results and the corresponding results calculated from the models associated to each stage, and gives the average pressure during each stage, as well as the certain result parameters of each stage, as output. For the filtration stage analysis, the software uses Equation (3.3), and calculates the average specific cake resistance, α_{av}, the filter medium resistance, R_m, and the apparent filtration concentration, c, as shown in Chapter 3.2 and in FILTRATION-box in Figure 13.1.

For the washing stage, the analysed amount of chlorides retained in the cake, R_s, and the amount of wash water used for cake washing, V_w, are given for the software as input. Additionally, the user can define the value of the washing parameter w_c in the exponential decay model (Eq. 11.2). The software calculates the volume of the residual filtrate retained in the cake at the beginning of the washing, $V_{f\,0}$, according to Equation (11.1), and the wash ratio, W_R, according to Equation (5.3) for the filtration run in the analysis. Then the calculated value of R_s is plotted versus the value of W_R for that certain filtration run, and this data point is plotted in the same figure has the R_s versus W_R -curve, which is calculated from exponential decay model (Eq. 11.2) with the given w_c-value. These are shown in Figure 13.1 in the WASHING-box and related boxes. In this way, it is possible to compare the data point obtained from the test with the exponential decay model, and choose the right w_c-value for that filtration run.

For the compression dewatering stage, the height of the filter cake, L, is calculated from the mass of filtrate as a function of time from Equation (6.4). Then the consolidation ratio, U_c, is calculated from the experimental data (Eq. 6.8) as shown in Chapter 6.2.2, and from the Terzaghi model (Eq. 6.9) and Shirato model (Eq. 6.19) as shown in Chapter 6.2.4. These calculated values are plotted in one figure, as shown in the COMPRESSION DEWATERING-box and related boxes in Figure 13.1. The value of the consolidation behaviour index, υ, in the Shirato model can be fixed by the user. The software gives the fitted value of the modified consolidation coefficient, C_e, and calculated ω_0-value, as output.

For the displacement dewatering stage, the software calculated the average pressure during this stage according to the DISPLACEMENT DEWATERING-box in Figure 13.1.

If the short compression and/or air-drying stages are performed between the filtration and cake washing stage, or between the cake washing and the final compression dewatering stage, these stages are chosen to be included in the test run by clicking them from the list given by the software at the beginning of the analysis. The software calculates the average pressures during these stages, which are shown with grey colour between the main process stage boxes in Figure 13.1.

This software developed allows the systematic data analysis of each starch filtration run, and the easy gathering of the typical parameters from the runs. In the data analysis with this software, the user has only to give the input values and choose the order and the start and end times of the process stages in order to get the analysed parameters from the process stages, and the plots describing the proceeding of these stages. This saves time, minimises the amount of miscalculations, and helps to easily find out what happens during the starch filtration cycle. Also the possible faults during the cycle can be seen from the abnormal shape of the plot of Δp and $m_{filtrate}$ versus cycle time.

The different process stages of the filtration cycle are commonly treated individually. No-one has gathered them together for data analysis purposes. Although in this study the filtration cycle analysis is developed for the starch filtrations, it is possible to use this software for the filtrations of all the materials, which have a similar type of behaviour to one of the starches during the filtration cycle. It can be commonly used as the analysis tool for the systematic filtration data analysis. In the future, it could also be possible to extend the software to correlate the data for different process stages for designing a filtration cycle, and to indicate what happens, if some parameter is changed. Possibly, the software could be used for the optimisation of the separation processes in the future too.

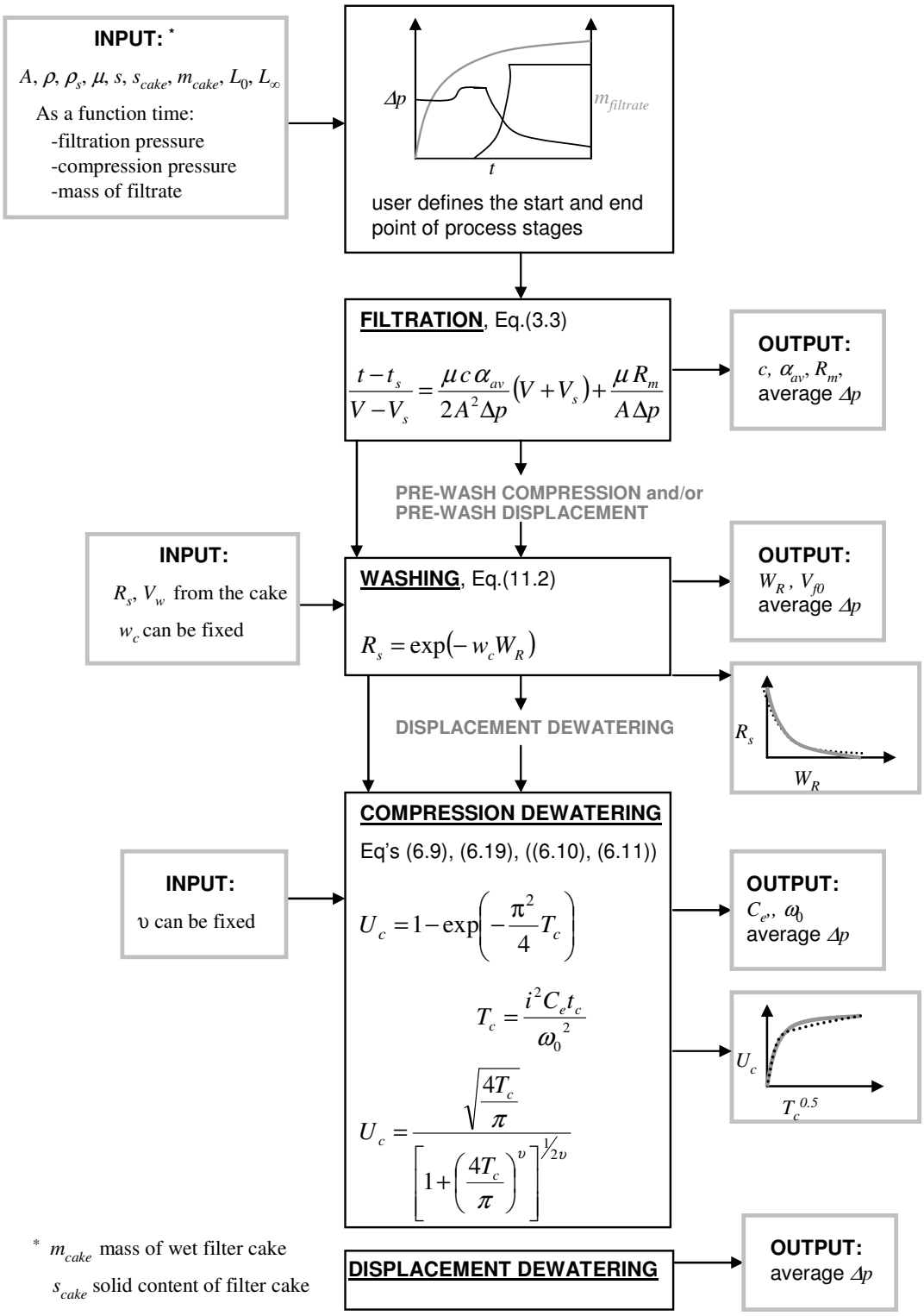

Figure 13.1 The structure of the FDA-software, which was used for the processing of the data from the starch filtration tests.

14 SUMMARY AND CONCLUSIONS

In this thesis pressure filtration technology was developed for effective production of pure starch products, the filtration sequences of that process were optimised using theoretical models, and the synthesis of the theories of different filtration stages suitable for starches was performed in the form of software. First, the studied starches and starch slurries were characterised in order to understand the differences between the starch grades and to be able to explain their filtration behaviours. Then many series of filtration measurements were performed with different types of starch slurries using different combinations of process stages and different filtration conditions during the filtration stages. The data from these tests were analysed, and compared to the data from the other tests and from the other series of measurements. Then the theoretical models were fitted to the data with the developed FDA-software in order to measure the model parameters and to get the models to describe the experimental results.

The key steps towards achieving the objectives of this thesis were the characterisation of starches, processing of each stage of the filtration cycle separately, and finally the synthesis of the theories at different filtration stages.

Characterisation. In general, the characterisation-results demonstrated that each starch grade had its own typical characteristics, which had a remarkable effects on its filtration behaviour. The characteristics studied were size, size distribution, and shape of starch particles, as well as settling velocity, viscosity, and flow behaviour of starch slurries. This study showed that the variations in the characteristics among the production lots of a starch grade were quite small, and hence, from a filtration point of view, the characteristics could be assumed to be grade-dependent. According to the results, the most important character was the particle size (shown in Table 8.2). Based on the measured particle size distributions, the decreasing mean particle size decreased the filtration rate: The potato starch, which had the greatest volumetric mean particle size (43 µm), had a very high filtration and washing rate, whereas the barley starch (17 µm) had the intermediate rate, and the maize starch (13µm) a little smaller intermediate rate. It seems also obvious that the bi-modal particle size distribution was the reason for the remarkably low filtration and washing rate of wheat starch. This bi-modality was hardly

seen in the volumetric particle size distribution (mean 17 µm / Figure 8.1), but it was easily proven by the number-based particle size distribution (mean 5µm / Figure 8.2).

The filtration stage. The results indicated that the different starch grades can be filtered in the same filter press, but the filtration technology (order of the process stages and the cycle parameters) have to be chosen depending on the starch type, in order to filter the starch effectively or even to make the filter work. The optimisation of the filtration stage is very important because it determines the structure of the starch filter cake, and hence it is the basis of the success of the whole process. In this study, the starch filtration stage was analysed by measuring the average specific filtration resistance of starch cakes at different constant filtration pressures according to Equation (3.3), and measuring the compactibilities of starches from these resistances according to Eq. (3.5). The average specific resistance of wheat starch (Fig. 9.1, Table 9.1) was found to be tenfold ($2\text{-}5\times10^{12}$ m/kg) compared to the corresponding value of maize or barley starch (0.2×10^{12} m/kg and $0.3\text{-}0.4\times10^{12}$ m/kg, respectively), and a hundredfold compared to the corresponding value of potato starch ($0.03\text{-}0.05\times10^{12}$ m/kg). These results showed clearly, that the decreasing particle size and the bi-modality of the particle size distribution increased the average specific cake resistance. Additionally, the average specific resistances of wheat and barley starch cakes showed a slightly increasing trend with increasing filtration pressure at a pressure range from 1 to 8 bar. This means that wheat and barley starch had a slightly compactible behaviour at this pressure range, whereas the average specific resistance of potato and maize starch cakes were not affected by the filtration pressure at this pressure range. (Salmela & Oja, 2000)

Filter media selection is critical to the success of the whole filtration. In this work, the simple method for comparing the different filter media in starch filtration applications was introduced. In this method, the resistances of the filter media were measured by filtering pure water through the filter media in the test filter, and measuring the resistance of the media from the water flow rate. Additionally, the media were compared by performing similar normal filtration tests with three different media, and comparing the filtrate flows and clarities of these tests, as shown in Fig. 10.2. (Salmela & Oja, 2004)

Cake washing. Based on the cake washing studies, a method for estimating a suitable duration for the washing process was developed: The washing was easily monitored online by measuring the conductivity of the out-flowing filtrate. The decrease in the conductivity indicated the point in which the wash water penetrated through the cake, and the cake was mainly washed. However, the samples from the cakes showed the final impurity contents, and gave a more reliable description of the proceeding of the washing process in the form of a washing curve (Figure 11.1). The washing results showed also that the diaphragm pressing before cake washing reduced the porosity of the cake and the amount of filtrate in the cake. Hence, a smaller amount of wash water was needed to obtain a certain purity for the cake, but the wash water flow rate in the cake decreased, which in turn decreased the capacity of the filter (Figures 11.2, 11.3). The higher the compression pressure the lower was the wash water flow rate in the cake. On the other hand, the wash water flow rate could be increased by increasing the wash water pressure (Figure 11.4). The increase was most beneficial, if the pressure was increased from the normal value, which was one bar greater than the filtration pressure, to the value which was three bars greater than the filtration pressure. The washing depended also on the height and on the average specific resistance of the cake to be washed. The thicker cakes needed longer washing times, but the cycle produced more pure starch. The higher the average specific cake resistance, the longer was the washing time or the higher was the wash water pressure needed. (Salmela & Oja, 1999a)

Compression dewatering. From a modelling point of view, it is important to know the exact transition point, in which the filtration or washing stage ends and the consolidation begins. The results demonstrated that this point could be measured from four different methods. The most reliable and straightforward method was the method in which the end point of the filtration was determined from the work cylinder pressure data given by the test filter. The value given by this method was a couple of seconds smaller than the value given by the plot of $-\Delta L/\Delta(t^{0.5})$ against dewatering time, t_c, on a logarithmic scale, or a plot of dt/dV against V. The fourth method was a plot of t/V versus V, and it gave slightly greater values for the transition point. Practically, the selected method had a very minor effect on the consolidation curve, although the correct selection of this point was critical to the success of the modelling of compression.

In this work, the progress of consolidation was monitored by measuring the mass of the out-flowing filtrate, and calculating the height of the filter cake in the filter chamber from the mass of the filtrate at small time intervals. From the cake height, the consolidation ratio was calculated according to Eq. (6.8) as a function of the square root of the dewatering time. The shape of the obtained consolidation curve depended on the particle size of the cake (Figure 12.1). The smaller it was the more gently sloping was the consolidation curve and the slower was the consolidation. The consolidation curves obtained from the piston press test filter and from the small-scale filter press were comparable, whereas the curve given by the filter press was a little more uneven. (Salmela & Oja, 2005a)

Modelling of washing. The experimentally determined washing results, the fractions of solute retained in the cakes (Eq. 5.2), were compared to the fractions given by the commonly used dispersion model (Eq. 5.8) and by the adapted simple exponential decay model (Eq. 11.2). The dispersion model fitted the experimental washing results of wheat and barley starch, when the value of the dispersion parameter, D_n, was between 1 and 10. This value was quite independent of the process conditions, although the results showed that the compression after washing (Table 11.1: Wheat 1 and 2) increased the value of D_n. The washing of potato starch could not be modelled with the dispersion model, because the curve obtained was outside the area of the dispersion model. The exponential decay model fitted well to the all washing results. The parameter w_c seemed to be more sensitive to the experimental conditions, even though the starch grades had the typical ranges of the w_c-values (Table 11.1). For wheat starch this range was 0.8-1.8, for barley starch 1.2-1.8 and for potato 2.8-4.4. The value of w_c increased with increasing filtration pressure, washing rate, or cake thickness. Both washing models failed slightly during the early parts of washing, but fitted well during the later parts of washing (Fig. 11.5). Since it is usually preferable to wash as much solute away as possible and hence continue the washing long enough, it can be concluded that both models are applicable to the modelling of starch cake washing. Also, since the exponential decay model is simpler in its application, it is therefore recommended prior to the dispersion model.

Modelling of compression. Four different theoretical models for predicting the cake consolidation were compared: the Shirato model, the Terzaghi model, and the Terzaghi-

Voigt model with two or three parameters. The results indicated that Terzaghi model fitted during the early stage of consolidation, but failed slightly when the consolidation proceeded (Fig.'s 12.2 and 12.3). The Terzaghi-Voigt model with two parameters fitted only during the later part of the consolidation (Fig. 12.2). The Terzaghi-Voigt model with three parameters fitted well during the entire consolidation (Fig. 12.2), but it was laborious to use, because it contained three parameters which had to be measured experimentally, and the fit of the model was very sensitive on the measurement of these parameters (Table 12.2). The Terzaghi model was confirmed to be adequate in many cases for the modelling of starch consolidation. Its parameter C_e was easily measured and very insensitive to the process changes (Tables 12.1 and 12.2). The value of C_e given by the piston press test filter could be used for the modelling of the results obtained from the filter press, if the possible difference in the height of cakes in the beginning of consolidation was taken into account. If needed, the later parts of the consolidation could be modelled with the Shirato model, which fitted well during the later part of the consolidation (Figure 12.3).

Stages. The order of the process stages during the starch pressure filtration was discussed during the course of this thesis. There is not one best combination for the process stages for a certain starch, but the results shown in this thesis could give some ideas of possible combinations. The testing in this respect needs to be addressed in more detail in further experimental studies.

Filtration data analysis (FDA). During the development of this work, the idea of the easy analysis and modelling of the starch filtration results was born. Earlier different process stages were analysed and modelled separately, which was time consuming and laborious. Therefore the software for filtration data analysis was developed. It congregates the theories introduced in this thesis, and works as an analysis and modelling tool (Fig. 13.1). This software could possibly be utilised in the future for optimisation too. Additionally, there seems to be no obstacles to utilise this software for other materials too. (Salmela & Oja, 2005b)

All in all, the filtration of starches is a very complex process and hence the testing should be continuous. Additionally, a large number of different starches and their modifications with their different characteristics make the process optimisation very

challenging. The change in one process condition affects the whole process. Therefore, it is always very important to know what the initial situation and the characteristics of the material are, and what kind of result is desired. Then the amount of the experimental work could possibly be reduced by developing the FDA-software to work as an optimisation tool for the processes.

The author believes that this kind of research helps to increase an understanding of the importance of the development of filtration technologies from sequence to sequence, and taking the demands of the particles to be filtered better into account. The importance of the theoretical models in predicting the experimental results is evident because the models are easily fitted to the results, and hence they can considerably reduce the amount of experimental work. Additionally, the automatic data processing with the software could be a great help in analysing the results, finding out the possible problems in the processes, modelling the process stages, and to even simulating the processes.

The following **conclusions** can be drawn from this study:

1. **Starch.** Different behaviour of starch grades in pressure filtration can be explained by their typical characteristics: The specific filtration resistance increases with decreasing mean particle size and increasing bi-modality of the particle size distribution. If the native starch is modified its filtration properties are changed. Each native and modified starch grade has to be considered as a starch type, which have its own typical properties.

2. **Starch**. From the filtration point of view, there is no need to characterise each production lot of a certain starch. If the starch grade and its particle size distribution are known, its filtration behaviour and the filtration conditions needed, can be anticipated from the former experiments.

3. **Filtration**. The different starch grades can be filtered in the same filter press, but the filtration technology (order of the process stages and the cycle parameters) have to be chosen depending on the starch type, because different starch grades have their own typical average specific cake resistances and compactibilities.

4. **Filter media.** It is very difficult or even impossible to choose the best filter medium for a certain application, but the filter media can be compared by measuring the pure water flow rate through the media, which are installed to the test filter, or by plotting the filtrate flow rate versus time from the parallel filtration experiments in which the filter media is different, but the cake formation is similar.

5. **Cake washing.** The cake washing is accurately monitored by taking samples from the filter cakes after the filtrations and by analysing them. However, an easy way to find the point at which the starch filter cake is fully washed is to measure the conductivity of the filtrate: the cake is fully washed, when the conductivity of filtrate begins to decrease. The applied wash water pressure must be from one to three bars greater than the filtration pressure in starch washing applications to ensure effective washing. The washing rate is directly proportional to the average specific resistance of the cake.

6. **Cake washing.** The washing stage can be modelled with the dispersion model or with the exponential decay model. They fit very well, when the wash ratio is greater than one. The latter takes the tail of the washing curve better into account. The parameter w_c is increased by the increased filtration pressure, and by the increase in the cake height.

7. **Cake dewatering.** The consolidation stage in starch filtration can be described with the consolidation curve. The point, at which the transition from filtration to consolidation occurs, can be chosen from four different methods (Chapter 6.2.2). The correct choice is critical to the success of the modelling of consolidation.

8. **Cake dewatering.** The early stage of consolidation can be modelled with the Terzaghi model and the later stage with the Shirato model, for which the parameter C_e is measured from the Terzaghi model. The parameter C_e measured with the piston press test filter is applicable to the results obtained with the filter press.

9. **Synthesis**. It is possible to analyse and to model each stage of the filtration process individually, and to gather the theories together and analyse the whole filtration cycle stage by stage with the FDA-software developed.

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