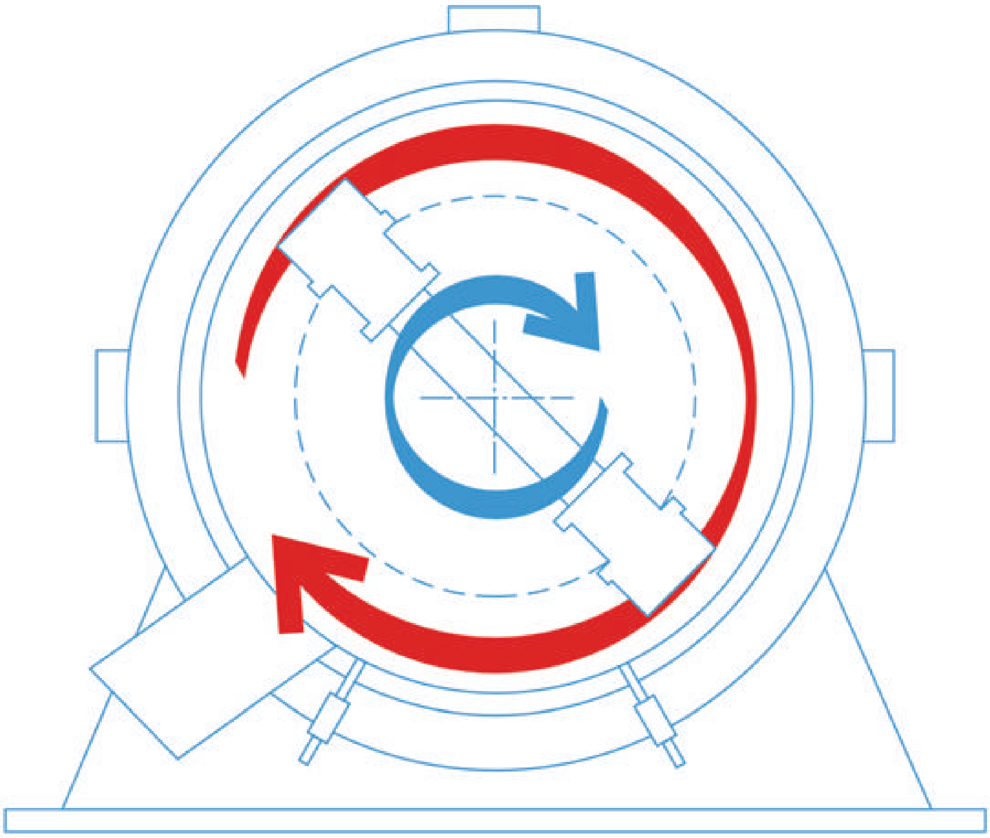
Franky Ruslim

Flow Phenomena in Cake Washing Driven by Mass Forces



Cuvillier Verlag Göttingen

Flow Phenomena in Cake Washing Driven by Mass Forces

Zur Erlangung des akademischen Grades eines
DOKTORS DER INGENIEURWISSENSCHAFTEN (Dr.-Ing.)

der Fakultät für Chemieingenieurwesen und Verfahrenstechnik der
Universität Fridericiana Karlsruhe (TH)

genehmigte
DISSERTATION

von
Dipl.-Ing. Franky Ruslim
aus Jakarta, Indonesien

Referent:	Prof. Dr.-Ing. W. Stahl
Korreferent:	Prof. Dr.-Ing. habil. H. Nirschl
2. Korreferent:	Prof. Dr.-Ing. C. Posten
Tag der mündlichen Prüfung:	01. Oktober 2008

Bibliografische Information der Deutschen Nationalbibliothek
Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der Deutschen Nationalbibliografie; detaillierte bibliografische Daten sind im Internet über http://dnb.ddb.de abrufbar.
1. Aufl. - Göttingen : Cuvillier, 2008
Zugl.: (TH) Karlsruhe, Univ., Diss., 2008

978-3-86727-788-4

Nonnenstieg 8, 37075 Göttingen
Telefon: 0551-54724-0
Telefax: 0551-54724-21
www.cuvillier.de

1. Auflage, 2008
Gedruckt auf säurefreiem Papier

978-3-86727-788-4

Preface

This work was established during my function as scientific assistant in the Institute of Mechanical Process Engineering and Mechanics of the University of Karlsruhe (TH). A work of great success is always a product of many hard-working and helping hands as well as genious intellectual contributions from many people. This also applies to this work and herefore I want to express my deepest gratitude to all these persons for their support.

I had the privilege of having great mentors in mechanical process engineering. I would like to express my deepest appreciation to Prof. Dr.-Ing. Werner Stahl, who despite his retirement always supported me with his never-ending ideas and intensive discussions. He was the one who encouraged me to pursuit my PhD in the first place.

Many thanks go to Prof. Dr.-Ing. habil. Hermann Nirschl for his unflagging challenge and continuous motivation boost. The latitude he allowed me in teachings and research has enriched my competence and above all encouraged my personality development.

I also thank Prof. Dr.-Ing. Clemens Posten who took on the honourable function as co-referent of this work, contributing with the point of view from the field of bio process engineering.

I acknowledge *Rhodia Research Center in Lyon* for providing the financial support for this cooperation project. Furthermore, the knowledge interexchange with Dr. Philippe Carvin, who is an exellent expert in solid/liquid separation, was very fertile.

It was an honour to be a part of the group *"Verfahrenstechnische Maschinen (VM)"* with such competent scientists, especially Dr.-Ing. Harald Anlauf, who has always accompanied the advances of research in solid/liquid separation over decades. It was also an honour to continue the great work of my predecessors, the *"Waschbären"* (in English: *racoons*; in Latin: *Procyon lotor*). I want to thank all my colleagues for the cooperative and pleasant companionship, both in professional and private circles. I really appreciate that. Furthermore, without the competent technical support of almost all of our co-workers, engineers, technicians, particle analysis lab (PMT), mechanical and electrical workshop, this work would not have been possible. Especially Dipl.-Ing. (FH) Friedhelm Flügel and Hans Guigas have given me more than I could ever return. Special thanks to you.

I am very grateful to the numerous young students, apprentices and interns who were enthusiastic for our research in particle purification. Their contribution in the *"Waschteam"* (research team working on washing) has brought science one step ahead. The members of the team were: Francis Lekane, Christian Brösicke, Serge Fozin-Foyet, Sandra Polovina, Kim Braun, Jenny Fleischer, Rüdiger Kostka, Benjamin Schmidt-Hansberg, Sandra Dittmann, Norbert Linkenheil, Jessica Rahardja, Ardo Adiwidjaja, Elena Kipf, Antonina Belyaeva, Matthias Bönisch, Dario Lopez, Vincent Siauw and Jennifer Landes.

My greatest thanks go to my parents, my sister and my dearest Melissa who have made all this possible in the first place. They have been my reliable personal stronghold. Thank you for your patience and for standing by me through the ups and downs, I love you sincerely.

Karlsruhe, in Autumn 2008 Franky Ruslim

Dedicated to my father and the wonderful women in my life: my mother, sister and Melissa

"It is hard to fail, but it is worse never to have tried to succeed"
Theodore Roosevelt

"A smile is a curve that sets everything straight… ☺"
Phyllis Diller

Contents

1 Introduction

Many processes in the chemical industries begin with the synthesis of particles in liquid and end with thermal drying (Figure 1-1). Between these steps, there are several important process sequences which define the quality of the final product. The solid/liquid separation is one of these unit operations. The main purpose is hereby to remove as much liquid as possible from the solid particles by means of mechanical forces such as differential pressure or mass forces. However, in most cases a complete moisture removal by mechanical forces is not possible. The lower the moisture content is the less effort must be done to eliminate the residual moisture in the subsequent thermal drying process which is mostly very energy-consuming.

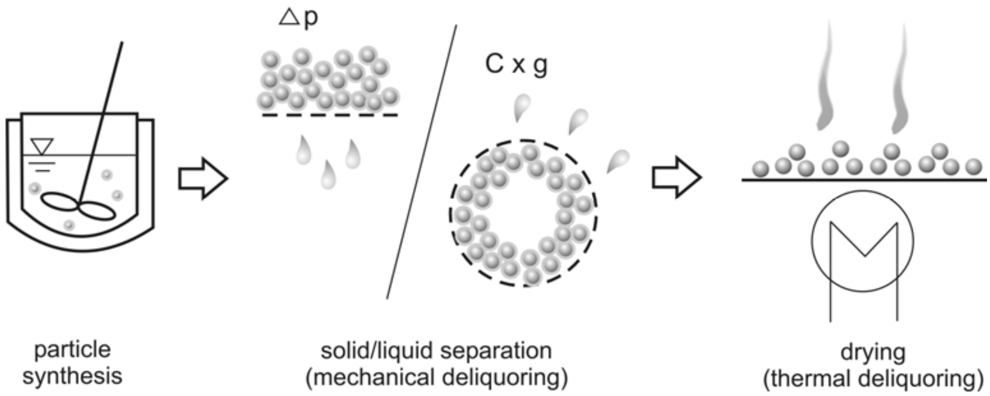


Figure 1-1. Example of a process chain in the chemical industries.

During the particle synthesis, e.g. by reactions, crystallization or precipitation, there is a chance that impurities and contaminants enter the product system. In addition, residual reactants, by-products or derivatives can still remain in the product suspension after the synthesis ends. Since these substances may influence the final product properties, their presence is undesired. Depending on the nature of these impurities, either thermal or mechanical methods of purification can be applied to remove them.

While thermal methods such as distillation, rectification and extraction are powerful methods of separating undesired liquids to achieve a very high degree of purity, mechanical methods such as filtration and washing offer a rather cost-efficient though effective way of reducing dissolved impurity concentration in the liquid phase. In cases of non-volatile impurities, mechanical washing is the sole competitive method to eliminate these before the drying process.

Washing is done by displacing the mother liquor containing dissolved impurities by the wash liquor containing less concentration of these impurities. Thus the concentration of the impurities is reduced by the addition of the wash liquor. If the impurities are soluble in the mother liquor, these can be situated in areas where the liquid is held inside the product system such as in macroscopic void areas, in a thin surface liquid layer on particles, in liquid bridges between adjacent particles, adsorbed on the particle surface or inside particle pores as shown in Figure 1-2.

The displacement process of the mother liquor by the wash liquor has to be induced by a driving force leading to a liquid flow out of the product system. The magnitude of this driving force depends on the force, which retains the mother liquor and/or the impurity in the product system. The liquid is mostly held by the capillary force. Either differential pressure or mass forces can be used to exceed the capillary force and therefore enforce a liquid discharge from the porous system. The selection between filters or centrifuges can be made dependent on the particle size. While centrifuges are suitable for deliquoring coarse particles with sizes larger than 50µm, filters are mostly used to process particles smaller than 10µm which posess high capillary pressure [Stahl, 2007]. Fortunately, the rapid development of centrifugal technologies has expanded the range of application into particle size regions of 5µm (peeler centrifuges, decanters) and even lower (0.1µm for separators). In addition, the assembling options as a low-cost machine or as a high-value complex apparatus for sterile products equipped with high-tech measurement and control system offer a broad range of applications.

Continuous centrifuges are widely spread in the chemical, pharmaceutical industries and mineral processing. Pusher centrifuges, decanters (screenbowl centrifuges), worm/screen centrifuges and separators are a few representatives of the continuous type of construction. While pusher [Ferrum AG; kmpt AG] and worm/screen [Siebtechnik GmbH] centrifuges are used for deliquoring coarse and crystalline products such as bulk chemicals, salts, minerals and plastics, separators are widely spread in fragmentation and enzyme treatment, oils and fats recovery, production of dairy products, beer, wine, fruit and vegetable juices [GEA Westfalia Separator]. Discontinuous centrifuges such as pendulum, peeler/scrapper centrifuges both in vertical and horizontal construction are often convenient solutions for processing viscous media, fine chemicals, and similar products in the chemical, pharmaceutical, agricultural, food and nutrition industries.

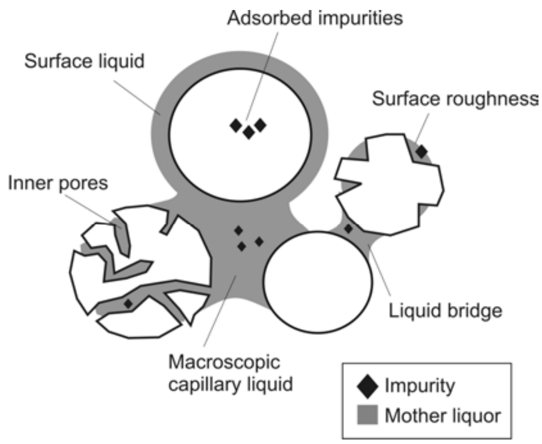


Figure 1-2. Possible locations of impurities dissolved in the mother liquor.

Approximately 90% of particulate products processed in centrifuges undergo mechanical washing, in order to achieve a sufficient degree of purity. Examples can be found in the manufacturing process of sodium chloride $NaCl$ (approximately 48 millions of tons per year) and potassium chloride KCl (approximately 28 millions of tons per year). In the mineral industries, bicarbonate, gypsum $CaSO_4$, aluminum hydroxide $Al(OH)_3$ as well as acetylsalicylic acid, penicillin and insulin in the pharmaceutical industries [Hegnauer and

Thurner, 2007] are some more particulate products subjected to washing. The diversity of the products and their properties makes it difficult to generate a general rule of thumb for an optimum washing process. It is considered necessary to distinguish the optimization strategies according to the respective product properties and the used apparatus. Common problems of washing are insufficient degree of purity, too high wash liquor consumption and change in cake structure due to application of the wash liquor.

While Heuser [2003] investigated the washing process on filters (gas differential pressure field) using particles with average sizes ranging from 20nm up to 50µm, this work aims for optimization of washing in centrifugal field. Aside from these two methods, Hoffner [2006] developed a new washing method based on principles of moving bed performed in a sedimentator. A more detailed differentiation of the main focuses of the existing works is given in chapter 2.

A major problem that emerged in former sporadic investigations on <u>centrifugal washing</u> was caused by treating the washing process separately. Most investigations focused merely on solving process, machine and product specific problems. Many studies show inconsistent results.

In this work, the centrifugal washing is examined as a partial process of an integral solid/liquid separation process. Investigations on centrifugal washing have to deal with mechanical deliquoring process, hydrodynamics, interface phenomena and mass transfer. This study begins with understanding the fundamental mechanism of cake deliquoring and washing in a macroscopic scale, supported with visual observations and physical as well as chemical material data. Afterwards, a development of a lab-scale centrifuge as a powerful tool for process parameter screening will be presented. Important experimental data acquired from the tests using this apparatus will then be discussed, especially concerning the change of cake saturation due to the interaction between deliquoring and washing process. Analogies from other fields of science are proved to be useful for the general comprehension and seen as confirmation of similar physical phenomena observed in the washing process. Furthermore, basic models with the purpose of a theoretical description of washing are analysed to determine their applicability on centrifugal washing processes. A novel modification and combination of the existing models will also be presented. The final chapter of this work deals with the technical realization of the optimization strategies developed on the base of the observed results. Moreover, new operating methods of washing will be proposed as future solutions to enhance impurity removal.

2 State of the art

Mechanical deliquoring and washing are subjects of numerous investigations worldwide. The following description is an overview of important scientific documentations which are relevant to the topic of this study.

2.1 Methods of centrifugation and washing

A solid/liquid separation process in a filtering centrifuge consists normally of several sequences which will be discussed below.

Feeding suspension and cake formation

The suspension is introduced and distributed on to the circumference of the centrifuge drum. The liquid passes through the sieve or filter cloth and the solid particles settle down on to it, bridging the pores of the filter media. This sedimentation process is part of the cake formation process, driven by the centrifugal force. The solids concentration of the suspension in the feed pipe defines the homogeneity of the cake, the solids breakthrough and the cake formation rate. Bickert [1997] investigated the influence of the solids concentration on the sedimentation velocity for monodispersed suspensions. For concentrations higher than 10% of volume, the sedimentation velocity was below the Stokes velocity of singular particle [Koglin, 1971]. Pouring the suspension onto a deliquored cake may result in inhomogeneous cake structure and unbalance. On the other hand, Müller et al. [1980] found that if too much suspension was given onto a thick cake, a large amount of liquid stayed on the cake forming circulating liquid waves. This also caused an unbalance of the rotor. Detailed calculations on the continuous liquid feed and real pulsated suspension addition were done by Borel et al. [1990]. Stahl [2004] discussed about physical effects that may influence the cake structure during feeding the suspension and cake formation. Those are particle disruption due to energy dissipation, size classification due to sedimentation, dense packing due to crossflow of attaching particles on the filter cake [Gösele, 1975] and compressibility due to electrostatic repulsion or particle disruption. A recent work done by Beiser [Beiser, 2005] dealt with the sedimentation behavior of sub-micronal particles in the centrifugal field. Focus of this work lay on the role of the solids concentration, centrifugal acceleration and physico-chemical properties of suspensions in influencing the particle sedimentation. Parallel to this, Erk [2006] investigated the consolidation behavior of suspensions containing fine particles in solid/liquid separation apparatuses, including centrifuges. He made a theoretical description of the separation process including consolidation and sedimentation using the yield point as a characterization parameter.

Flow through filter cake

If there is enough liquid on top of the cake, a laminar single-phase flow takes place, where the liquid permeates all cake pores. This can be described theoretically by the law of Darcy [1856] which will be discussed further in chapter 3. In relation to this, several correlations were proposed for calculations of the cake permeability from material parameters [Carman, 1937; Gupte 1970; Happel, 1973]. The models are valid for incompressible packed beds. For compressible cakes, the porosity and permeability vary with the solids pressure. Thus they differ along the cake height. Tiller [1999] developed a model to describe the flow through compressible and highly compressible packed beds. Alles [2000] and Heuser [2003] investigated the cake compression during filtration.

Filter cake deliquoring

The deliquoring process in the centrifugal field has been subject of several investigations, mostly with the purpose of describing the process by physical correlations. Besides empirical correlations from Bender [1971] and Redeker [1983], physical models of deliquoring as piston flow [Zeitsch, 1981, Reif et.al., 1990] and film flow [Mayer, 1986] as well as their superposition [Batel, 1961; Reif, 1990] provide a solid basis for further developments. In addition, Wakeman and Vince [1986] worked out a deliquoring model based on findings from the gravitational and differential pressure fields. Stadager [1995] combined the deliquoring process in the centrifugal field with the differential pressure field. Hultsch [1988] already showed a decrease of the equilibrium saturations due to pressure superposed centrifugation. Wünsch [1994] found an inclination of the Bond plateau which was caused by particle surface roughness. Nicolaou [1996] and Wünsch also found that the surface roughness led to slower dewatering kinetics. Peuker [2002] made a further development of the piston flow model, extended by the combination of centrifugation and steam pressure. He introduced an analytical, implicit solution of the temporal movement of the interface between steam and liquid during the steam pressure superposed centrifugation.

Filter cake washing

Filter cake washing is a step in the solid/liquid separation process which is performed either directly after the cake formation or after a partial or complete deliquoring of the cake. First, a summary of general washing methods will be given below and later in chapter 2.2, specific works on centrifugal washing will be presented.

Hoffner et al. [2003] presented a comprehensive overview of existing washing methods and apparatuses in industrial processes and academic fields. A summary of this overview adapted from their paper is presented in Table 2-1. They distinguished the states of the solid particles as freely moving particles, moving bed and packed bed whereas the washing mechanisms were classified into displacement and dilution. Displacement washing means that the mother liquor containing dissolved impurities is displaced in a piston flow and substituted by the pure wash liquor. In filter cake washing, an ideal piston flow through the porous system is desired. However, due to the inhomogeneous structure of the filter cake such as shape, porosity gradient, pore size distribution, a deviation from a piston profile is always observed. Nevertheless, displacement method is applicable to wash a particle collective with a rather small amount of wash liquor thus in an efficient way. Although a complete impurity removal is not possible [Heuser, 2003], filter cake displacement washing is a widely used method as it can be integrated in the solid/liquid process without any additional apparatuses. Caution must be taken when working with very fine particles of low permeability. This results mostly in high driving forces and extreme long washing times. Moreover, cakes formed by these fine particles are sensitive against changes in interfacial effects such as ionic strength, pH value, capillary forces, so that they tend to crack [Bender, 1983; Ruhland, 1999; Wiedemann, 1996] or shrink [Gösele, 1986; Wiedemann, 1996; Roth, 1991]. Displacement of the mother liquor can occur when single particles settle into a column filled with wash liquor. This principle is used for sedimentation columns [Dell and Preece, 1980]. Displacement washing in a sedimentator is also based on sedimentation of particles in their mother liquor forming a moving bed. The wash liquor is given transversely, perpendicular to the sedimentation direction. Due to relative particle movements, contact areas between adjacent particles are made accessible for the wash liquor leading to enormous enhancement of the washing results as reported by Hoffner [2006]. A similar mechanism can be found in conical centrifuges or in washing cakes in the area between the stages of multi-stage pusher centrifuges.

Table 2-1. Overview of existing washing methods adapted from [Hoffner et al., 2003].

	SOLIDS			
LIQUIDS	**Mechanisms**	*Freely moving particles*	*Moving bed*	*Fixed bed*
	Displacement	Sedimentation column	Sedimentator	Filter cake washing, Filter, Centrifuges
	Dilution	Stirred tank	Conical centrifuges	Centrifuges

If the rigid structure of the filter cake becomes a great barrier for a sufficient impurity removal, methods of dilution or known as reslurry washing should be applied. Here, the fixed bed is repulped in a large amount of pure wash liquor so that the rigid structure is loosened, for example in a stirred tank. Thus the particles can freely move inside the liquid. The aim is to dilute the mother liquor in order to obtain a lower impurity concentration. Theoretically to achieve a concentration of zero, an infinite volume of wash liquor would be necessary. Since this is not economical, the dilution washing is often performed in multiple stages, for example as mixer-settlers. A counter-current wash liquor flow is usually practiced in industrial processes to allow a more efficient use of the wash liquor. The main disadvantages of dilution washing method are the necessity of an additional separation apparatus between the stages and the relative high consumption of wash liquor compared to displacement filter cake washing [Bender, 1983]. Furthermore, not only a macroscopic dilution of the mother liquor such as in a stirred tank belongs to this classification. A local microscopic dilution of impurities remaining in the residual moisture in the cake such as on particle surface, in liquid bridges between adjacent particles and liquid held in internal pores is also considered as a "dilution mechanism" of washing. However, only hypotheses could be made until now, assuming that this mechanism also takes place during filter cake washing in the centrifugal field, where there is no change in the cakes rigid structure [Hoffner et al., 2003]. Exactly this is an important question to be investigated further in this work.

At last a modified classification [Ruslim, 2007c] will be presented in this place, which is more practicable for the selection of the methods for industrial applications. Instead of classifying the washing mechanism, a differentiation of the applied driving force for the liquid flow is made.

- *Gas differential pressure*

  Liquid flow can be induced by pressure forces either by means of vacuum or excess pressure generated by a vacuum pump or compressors respectively. Filters (belt, drum filters) and nutsch filters are typical apparatuses using this driving force. The pressurized gas presses the liquid held by capillary force in the cake pores enforcing a deliquoring. The applied wash liquor also flows driven by this force. As soon as the gas breaks through the largest pore and the filter cloth, the driving force may collapse due to high pressure drop. Heuser [2003] did an excellent work investigating washing of fine particles (typical products processed on filters) of submicronal size using differential pressure. The focus of his work was the influence of physico-chemical properties on the cake structure and washing behavior. In contrary, for coarse particles of over 50µm this aspect is merely of secondary significance due to their small specific surface areas.

- *Mass forces*

  In contrary to differential pressure which induces a force on a surface, mass forces such as earth gravity or centrifugal forces accelerate every single volume element due to its mass. In centrifuges, this is the driving force for liquid flow through the filter cake. A few papers deal with washing on centrifuges. These will be discussed in chapter 2.2.

- *External forces*

  The more complex the particle structure is, the better must a washing method be in order to meet requirements for higher degrees of purity. This has encouraged investigations on novel washing methods using combinations of driving forces and mechanisms. The influence of electrical field was investigated by Tarleton et al. [2004] while magnetic separation methods were used to selectively bind a substance from a multi-component mixture for further elution and enrichment [Fuchs, 2005]. Peuker [2002] applied water steam for deliquoring and washing filter cakes in centrifugal field. Using an advanced measurement technique in a bowl centrifuge, he could measure the temperature distribution over the cake height, so that the movement of the steam front-line through the cake could be acquired online. Under optimum conditions, a homogeneous distribution of the wash liquor as water condensate on particle surface was possible, inducing a mixing between the wash and mother liquor. Further more, the deliquoring process was enhanced due to additional thermal effect. In [Peuker et al., 2003] the authors reported that the optimum parameters for a good deliquoring were not necessarily identical with the ones for washing. They found that displacement or other mass transfer processes seemed to get worse with increasing steam pressure, so that poor washing results were observed. Consequently they proposed a main focus of future research dealing with conventional washing in the centrifugal field in order to understand the phenomenology. Therefore this was considered as a motivation for this current work.

2.2 Centrifugal washing

Centrifugal washing has been given attention since more and more products are processed in centrifuges and problems still occur in operating the process. The major problems are often the insufficient degree of purity due to unfavourable operating conditions and the very high liquid consumption. The large amount of wash media, e.g. acids and organic solvents are cost-intensive and this produces also a large amount of liquid waste that contains impurities. It has to be regenerated in a post-treatment process before disposition.

Some problems are caused by disadvantageous cake behavior such as cracks formation [Stahl, 1997]. Possible causes for this behavior are desaturation of the cake leading to an irregular distribution of the capillary forces for different pore sizes [Wiedemann, 1996; Nicolaou, 1996] and physico-chemical effects affecting the interaction between fine particles due to their electrical properties [Heuser, 2003]. The wash liquor given on to cracky cakes penetrates into the cracks of lower flow resistance and leaves other areas unaffected. Berger [1990] found out that the wash liquor addition might also form crater on areas near cake surface. This thickness profile can have the same negative effect as cracks formation.

Washing with a wash liquor of a lower viscosity compared to that of the mother liquor often causes fingering effects, leading to local advanced liquid flow in the cake and irregularities in flow [Bender, 1983; Heuser, 2003]. In contrary, washing with a viscous liquid was found to be advantageous.

The effects presented above are also found in washing processes in centrifuges. These were also the encouragement for practical investigations on centrifugal washing. Talmage [1966] was one of the pioneers in investigations on washing processes. He introduced the concept of one-displacement wash, which assumed that in the ideal case, all of the mother liquor left in the cake would be displaced by an equal volume of wash liquor. He defined the term of washing efficiency by the percentage of mother liquor removed by a one-displacement wash. This was applied to analyzed several filter and centrifuge cakes. Zürrer [1973] performed a qualitative study on equipments for wash liquor addition in a multi-stage pusher centrifuge. Rushton [1981] gave very useful correlations to calculate drainage time and equilibrium saturation in centrifugal deliquoring and also showed advantageous results in washing flooded cakes instead of dewatered cakes. Bender [1983] presented results of washing salt as impurity out of cakes processed in filter presses, nutsche filters, drum filters and centrifuges. In the paper of Veksler [1987] several steps to calculate the radius of the free surface of a liquid ring rotating at the rotor angular velocity from hydrodynamic parameters were given. He referred to the work of Zhukov [1984] that proposed a control of the circumferential component of the wash liquor velocity at the nozzle outlet in order to increase the washing efficiency. Wakeman [1985] considered the scale up of solids loss, recovery, dewatering and washing stages of the centrifuge cycle to industrial size machines. Voitkovskii [1988] gave attention to washing crystalline products considering the aspect of solubility. Recent developments of washing equipments and methods were discussed in important books for centrifugation such as Stahl [2004] and Leung [1998]. The newest important publications concerning washing in the centrifugal field were written by Fuchs et al. [2003] and Peuker et al. [2003] who were also active in the Institute of Mechanical Process Engineering and Mechanics (MVM) of the

University of Karlsruhe (TH), providing valuable knowledge for this work, especially concerning centrifugal washing.

Specific constructional and technical improvements of the deployed centrifuges in industrial practice were reported by some authors.

- Bakhtin [1970] dealt with rinsing processes of ammonium sulphate crystals containing sulphuric acid and water in a centrifuge,
- Crabeil [1972] concerned with the use of centrifuges in sugar factories,
- a rather exotic application was presented by Rabanda [1984] who used a special lab-scale centrifuge for washing and drying of silicon wafers,
- Leung [2000] patented cake washing technology in a continuous-feed screen-bowl centrifuge to effectively remove cake impurities. At high wash rates the cake is reslurried and dewatered in sequential stages in-situ in a screen-bowl centrifuge to remove residual impurities left in the cake. At low wash rates the new technology increases contact between the cake and the wash liquor promoting cake washing by dissolution and diffusion.
- Nielsen [1999] investigated the color removal in a refining process in sugar factories and refineries by using a real time color measurement by a colorimeter,
- Grimwood [2002] discussed the importance of safety aspects in sugar washing on centrifuges,
- Wen [2002] performed studies on a screen-bowl centrifuge for dewatering and reconstitution of an ultra-fine bituminous coal from Alabama.

Theoretical description of washing processes was also subject of several works. Kuo [1960] developed a film model, assuming the existence of a stagnant liquid film on capillary walls, from which the impurities diffuse into the moving liquid flow in the core of the capillary. Wakeman [1972] and Järveläinen [1974] substituted this stagnant liquid film by side-channels. This side-channel model was applied to describe cake washing processes on filters and centrifuges. Besides the geometrical models, there are some empirical models to describe the washing results by adaptation of experimental data. Bender [1983] formulated an empirical model for washing by taking into account 18 relevant influence parameters. He reduced these into three dimensionless parameters

- $r_c \cdot L^2$,
- W and
- $r_c \cdot D \cdot t$

where L is the cake length, W is the wash ratio, D is the diffusion coefficient and t is the wash time. Heuser and Stahl [2000a; 2000b] developed this model further to predict correlations between the limits of the washing results, the cake thickness and the pressure difference for different regimes of the wash curve. The wash curve will be discussed more thoroughly in chapter 3.

The most promising and applied models are the dispersion model [Sherman, 1964; Michaels et al., 1967; Wakeman and Rushton, 1974; Wakeman and Atwood, 1988] with its manifold applications and modifications [Heuser, 2003; Hoffner, 2006] as well as the mixing cell model [Baerns et al., 1999; Levenspiel, 1999] which both stem from the chemical reaction engineering. The fundamentals of these two models will also be discussed in chapter 3.

From the above-mentioned works two points are evident. First, sporadic up to date investigations on centrifugal washing performed or assigned mostly by the industry show that this matter is presently still relevant due to current problems and rapid growth of synthesis methods producing new products with novel properties. Second, there are indeed some published works on centrifugal washing but most of them deal with specific product- or machine-related problems and only a few of the studies can give important lead for a better understanding of the mechanism in centrifugal washing either by experimental or theoretical approach.

2.3 Scope of this work and its differentiation from existing works

There are generally two classes of purification methods. The first one is by applying thermal processes such as distillation, rectification and extraction, mainly with the purpose of elimination of liquid impurity which constitutes a separate phase in contact with the liquid product (distillation, rectification, and liquid/liquid extraction) or solids (liquid/solid extraction) respectively. This is not a subject of this work, which rather pursues the mechanical way of purification. This mechanical purification method concerns with washing and deliquoring.

Although different concepts and theories of deliquoring are discussed in this work, these should not be the main focus but rather as a related unit operation in a solid/liquid separation process chain which may influence the washing process. Further on, this work concentrates on washing in the mass force field, for example in centrifugal field. Investigations on washing in gas differential pressure field indeed give an idea of the washing mechanism but the phenomena that happen in the centrifugal field are different. Moreover, the products processed in centrifuges are mostly larger of size than those processed on filters, thus while the focus of investigating fine particles lies in the particle surface interaction due to physico-chemical properties, other phenomena are more important when dealing with larger particles.

Motivation to this work was initiated by the industrial need of optimum washing methods especially on centrifuges as well as by previous research works on this topic in academia, which had opened new challenging questions. Peuker [Peuker, 2002] provided a valuable experimental knowledge on steam washing in centrifuges where the effects of mechanical and thermal deliquoring were compared to the wash effect. Furthermore he made demands on further systematic investigations on this matter. Hoffner [Hoffner et al., 2003; Hoffner, 2006] emphasized the current need of investigations on washing undersaturated cakes as in centrifugal field. Besides he gave advice to the further use of the dispersion model for undersaturated cakes and investigations on the applicability of the mixing-cell model to describe filter cake washing as comparison to the existing models [Wakeman, 1972, Wakeman and Rushton, 1974].

Although the dilution and moving-bed washing processes have been proved to be very advantageous in obtaining a high degree of purity, some drawbacks of these methods are the