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.

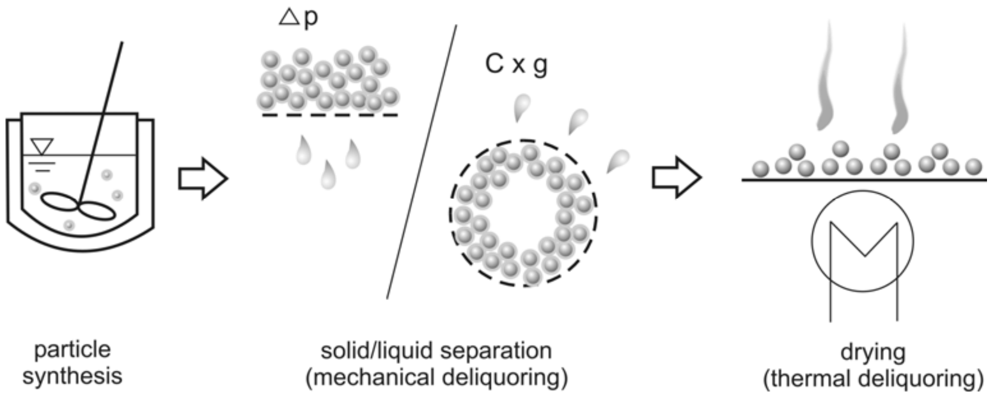

Figure 1-1. Example of a process chain in the chemical industries.

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.

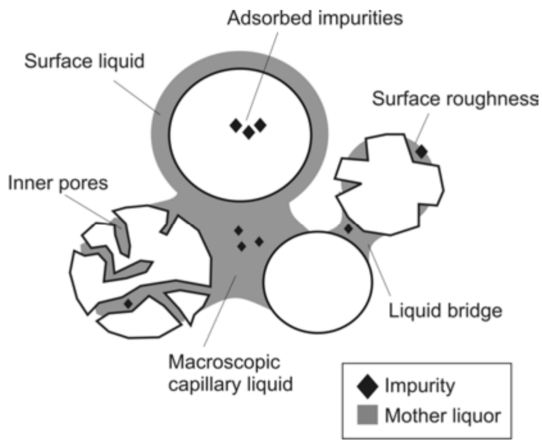

Figure 1-2. Possible locations of impurities dissolved in the mother liquor.

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 centrifugal washing 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.