



Optimization of the wash liquor flow rate to improve washing of pre-deliquored filter cakes

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Abstract

Filter cake washing is a common way of improving the quality in purity of particulate products in solid–liquid separation processes. In industrial applications, this process step is usually treated as a black box where optimization is performed through trial and error. Decisive progress has been made in academia by the development of physical models to describe the washing phenomena inside a porous structure such as filter cakes. Although some models have led to a better understanding of the process, experimental efforts are still inevitable for determining optimization strategies. This contribution is an empirical approach to investigate the influence of important process parameters of washing pre-deliquored cakes with the focus on the wash liquor and filtrate flow rate. In washing a pre-deliquored cake, low moisture content at the time of wash liquor addition can be very disadvantageous for the wash efficiency due to inhomogeneous flow. This can be the result of a poor re-wetting process while washing at low wash liquor flow rates. An increase of the flow rate was observed to be very advantageous to set higher moisture content of the filter cake and therefore enforce a homogeneous distribution of the wash liquor. A homogeneous distribution of the wash liquor inside the cake is generally required for good washing.

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1. Introduction

Filter cake washing is a common process in solid/liquid separation. The main goal is to eliminate the impurity in the mother liquor by adding clean wash liquor. These days the wash efficiency has become very important, not only due to high purity demands but also regarding the efforts on minimizing process costs.

Two known mechanisms are the *displacement* and the *dilution* washing. During the *dilution* washing process the product is given as a suspension and mixed with clean wash liquor to allow sufficient contact time and mass exchange due to convection by agitation. The main disadvantage is the high wash liquor consumption in order to achieve a sufficient purification result in a single step. Commonly, multistage dilution is carried out with a co- or counter-current direction of the wash liquor

(Hoffner et al., 2004). In contrast, the *displacement* process allows only a limited time of contact between the mother and wash liquor during the liquid movement through a filter cake. The purification effect is achieved through the displacement of the mother liquor by the penetrating wash liquor. In most cases, the displacement washing is also accompanied by dilution in microscopic regimes of the filter cake. This method is used for example in washing crystalline products in centrifuges, where the cake is often partly dewatered and the wash liquor is distributed by a nozzle as droplets (Fuchs et al., 2003). The dilution method in centrifugal washing can be performed by reslurrying the filter cake in screenbowl centrifuges as introduced by Leung et al. (2000) and Stahl (2004).

There are some physical models which try to describe the washing process by analysing the dominating transport phenomena as presented by Wakeman (1972). Most commonly used models are the dispersion (Sherman, 1964), side channel (Kuo, 1960) and the film model (Wakeman, 1976a). They are based on the assumption that the cake is homogeneous in

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structure with ideal boundaries. A generalization of these models is very difficult due to the variety of products, machines and fluctuation of the process conditions. This is why an experimental approach is often favoured to obtain a better understanding and enable process optimization.

A known problem in washing filter cakes, especially using high driving forces for the liquid flow is the desaturation of the cake before washing (pre-dewatering). This is mostly caused by late application of the wash liquor. Examples are found in washing processes using centrifuges or in wash columns under the impact of earth gravity but also in relation with hyperbar filtration (Heuser, 2003). If the cake is already desaturated, the wash liquor flows through a two phase system of solid and air. This mostly results in an inhomogeneous flow and the formation of stagnant areas, which are inaccessible for the wash liquor (Fuchs et al., 2003; Ruslim et al., 2005). If the surface of the particles is dry, this will also make the re-wetting process difficult since wetting a surface needs energy. The liquid tends to create a minimum of surface and flows as rills on the particle surface. Thus the contact between the contaminated mother liquor and the clean wash liquor cannot be maintained intact throughout the whole surface area. A homogeneous and even distribution of the wash liquor in the cake structure is required for a good and efficient washing. The subject of this work is to determine the wash rates to achieve this homogeneous and even distribution of wash liquor.

2. Theoretical background

Wash results are mostly characterized by the wash ratio W and the remaining impurity concentration ratio c^* . These are defined as

$$c^* = \frac{c}{c_0}, \quad (1)$$

$$W = \frac{V_{wl}}{V_{\text{product}}}. \quad (2)$$

Hereby the remaining impurity concentration c is related to the initial concentration c_0 and the wash liquor consumption V_{wl} to a significant product parameter V_{product} , which can be the pore volume V_{pore} or the volume of the mother liquor that has to be removed. In this work, the cake pore volume is used as a reference considering the complete saturation of the cake at the beginning of the experiments. In some cases, where the process duration is a more significant limitation of the process rather than the amount of wash liquor, the process time can be a good parameter instead of the wash ratio. This is the case when the process is limited by diffusion and sorption mechanisms.

To compare the wash results of different products or to enable direct evaluation of the influence of several parameters, the concentration ratio is normally plotted against the wash ratio to obtain a wash curve as shown in Fig. 1.

The wash results of filter cake washing lie mostly between those of dilution washing and the calculated ideal displacement model. The three regimes of washing are the ideal displacement (I) at low wash ratios, the dispersion regime (II) and the

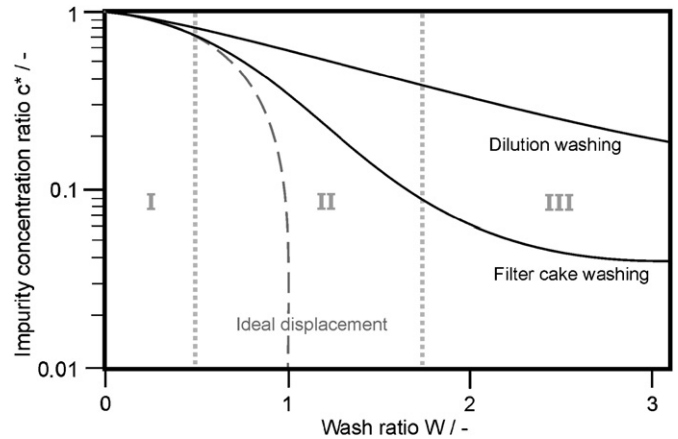


Fig. 1. The remaining impurity concentration c^* as a function of the wash ratio W .

diffusion regime (III) where no change in the remaining impurity concentration can be observed. The impurity removal takes place very slowly by means of diffusion.

Since washing in centrifugal fields is often related to desaturation processes, the properties and behaviour of particles as a porous system in the solid–liquid-apparatuses must be taken into account. The characterization of the moisture content of filter cakes can be given by the saturation S which is defined as the ratio between the moisture volume V_l and the pore volume V_{pores} according to

$$S = \frac{V_l}{V_{\text{pores}}}. \quad (3)$$

This is similar to the liquid hold up in the chemical reaction engineering but using the pore volume instead of the reactor volume as a reference parameter.

The saturation state of a filter cake is determined by the driving force which causes the desaturation of the porous system and the capillary force which holds the liquid inside the filter cake. The capillary force F_{cap} can be calculated from the capillary pressure which is predominant in interfaces between the liquid, solid and gas in a porous system. According to the capillary model (Schubert, 1982) and for small capillary radius, the pressure can be approximated to

$$p_{\text{cap}} = \frac{2 \cdot \gamma_l \cdot \cos \theta}{r}, \quad (4)$$

where γ_l is the surface tension of the liquid, θ the contact angle and r the capillary radius.

The dependency of the saturation state on the driving force for mechanical dewatering can be given as a capillary pressure diagram or a Bond diagram shown in Fig. 2 (Ruslim et al., 2006c). A useful characterization of the ratio between the acting forces is the Bond number Bo which is defined as

$$Bo = \frac{\rho \cdot g \cdot C \cdot d_h \cdot H}{\gamma \cdot \cos \theta}. \quad (5)$$

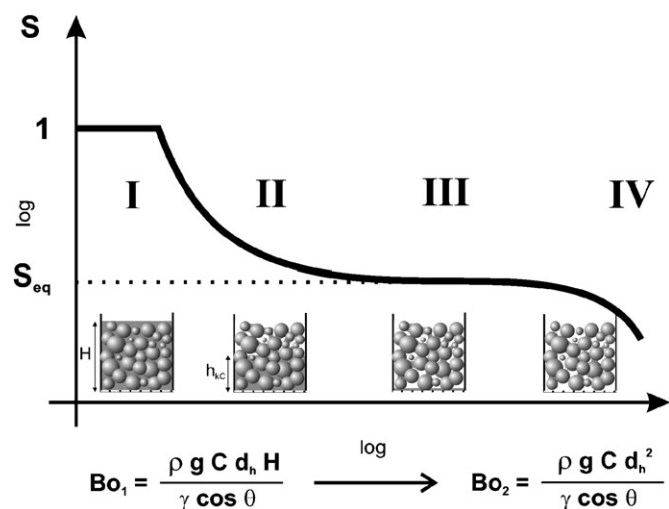


Fig. 2. The Bond diagram showing the cake saturations at different Bond numbers.

It describes a direct comparison between material parameters such as the density ρ , surface tension γ , contact angle θ and the process parameters such as G-factor C , the hydraulic diameter d_h and the cake height H .

In the first Bond regime the driving force is not sufficient to displace the liquid remaining in the capillaries of the cake. In the second regime the desaturation of the cake takes place with increasing Bond number. In experiments, this is marked by the decline of the liquid gage until the system has reached its equilibrium level in Bond regime III. Here the residual moisture stays in the contact points between particles. These liquid bridges can only be removed from the cake by increasing the driving force up to levels which are mostly no longer economical. This regime can, however, become significant if relative large particles are processed since the hydraulic diameter becomes large and thus the capillary force will be low compared to the driving force (compare to the definition of the Bond number). To describe the states in regimes III and IV usually the second Bond number Bo_2 is used where the cake height H in Bo_1 is replaced by the hydraulic diameter d_h because the cake height loses its influence if the capillaries are no longer filled with liquid (Stadiger, 1995).

3. Experimental set-up

The apparatus used for the experimental studies was an earth gravity column (Fig. 3) which enabled investigation of the influence of the parameters of washing. It consisted of a cylindrical column made of acrylic glass with a diameter of 90 mm and a height of 350 mm. The total filtration area through a multifilament sintered metal plate with a pore size of about 100 μm situated at the bottom of the column was calculated to be 63.62 cm^2 . The wash liquor was introduced by a full cone UniJet[®] nozzle manufactured by Spraying Systems with different gap diameters for a flow rate range between 4 and 40 cm^3/s . Underneath the filter cloth, a funnel-shaped collector connected the column with an elastic hose forming a u-pipe which was

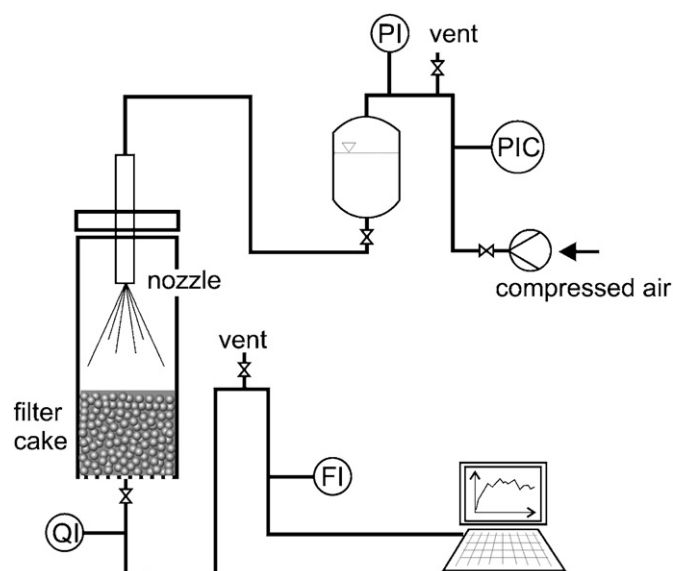


Fig. 3. Apparatus set-up of the earth gravity column (EGC) for particle washing in earth gravity.

Table 1

List of initial operating conditions for the wash experiments

Parameter	Value	Unit
Saturation	1	–
Cake height	150	mm
Initial salt concentration	0.2	mol/l

filled with liquid at all time during the experiments. The outcoming mother and wash liquor went into a beaker which was placed on a scale to measure the filtrate flow online.

The purpose of using this device was to determine tendencies and product behaviour as a result of parameter variations. An advantage of the apparatus was the good visual observation of the system, especially during the washing and flow process.

The apparatus was built to perform the washing process in a column where the filtrate drainage was driven by the earth gravity. The mass force was the cause of the liquid flow. Because the acting mass force was relatively low (earth gravity), the cake resistance had to be kept low in order to have similar saturation states as in industrial practice.

The experiment began by building the cake out of the particles and their mother liquid of a defined salt concentration (0.2 mol/l). All experiments had identical initial conditions as listed in Table 1.

The desaturation and washing steps were performed by opening the outlet valve of the apparatus allowing the driving force caused by the hydrostatic height between the liquid gage and the filter medium. An overflow system consisting of a flexible hose connected the filter medium and the filtrate outlet. This was used to prevent air bubbles, making sure that the volume underneath the filter medium was always filled with liquid.

The cake was analysed separately after the experiment in a reslurrying process to determine the impurity concentration.

Hereby a definite amount of clean wash liquor was given to the dried cake (dilution) and then the conductivity of the previously stirred liquid was measured. For products that adsorbed the impurity, the sorption isotherm had to be determined first to assure that all impurity was diluted in the liquid.

In addition to the offline measurement and as verification, the conductivity of the filtrate was measured online using electrodes. The measurement device was assembled in the Institute of Mechanical Process Engineering and Mechanics and consisted of two stainless steel electrodes which are supplied with an alternating current. The device delivered an analogue voltage signal which was converted by an A/D converter and recorded on a PC.

Since it was the cake that represented the target product, it was considered better to determine the impurity concentration inside the cake rather than the impurity concentration in the filtrate. The latter was done merely as proof that all the impurity was accounted for. Over a mass balance that was done randomly for a few experiments, the impurity concentration in the cake could be calculated for given initial concentration and measured concentration in the filtrate. Hereby, the initial liquid volume in the cake was 370 cm^3 with a concentration of 0.2 mol/l , the final liquid volume in the cake depended on the saturation and varied between 18 and 67 cm^3 . The dead volume of the apparatus consisting of the filtrate outlet system and the flexible hose was measured to be 190 cm^3 . The dead volume between the cake and the conductivity measurement cell was 60 cm^3 and had to be taken into account in the balance. For high impurity concentrations after washing, the discrepancy to the measured impurity concentration by analysing the cake was about 8 – 10% which was acceptable. However, when working at high wash ratios, the final impurity concentration was low and the relative discrepancy in the balance became unsatisfactorily high (25 – 30%). The discrepancy between the mass balance and the measurements was caused mainly by the residual moisture in the filter medium and outlet system which still contained residual impurities. By flushing the apparatus after the experiments to determine the residual salt concentration and correcting the calculation, a good accordance between the balance and the measurements could be achieved. Nevertheless, the determination of the impurity concentration in the cake was considered the best method of quantifying the results in this work instead of using the online data even though a separate analysis of the cake was more time consuming.

The particles used for the experiments were glass beads, which offered ideal properties that were important for systematic and specific investigations by excluding irregularities such as broad particle size distribution, particle shape and compressibility. These effects need to be taken into account in the applications on real products. Glass bead fractions of 3700 – $4100\text{ }\mu\text{m}$ and 2000 – $2300\text{ }\mu\text{m}$ had a very low cake resistance in earth gravity that was approximated to be around 10^8 m^{-2} while the specific cake resistance of the fraction 400 – $600\text{ }\mu\text{m}$ was measured to be approximately $8 \times 10^9\text{ m}^{-2}$ by flow experiments in earth gravity. To enable resaturation of the cake, the low cake resistance was compensated by a relative high resistance of the multifilament filter medium which was about $2 \times 10^8\text{ m}^{-1}$.

Table 2
List of varied parameters

Parameter	Value	Unit
Particle size	400–600; 2000–2300; 3700–4100	μm
Wash liquor flow rate	4–52	cm^3/s
Wash ratio	0–2	–
Pre-dewatering time	0; 5; 20; 45	s

The impurity used in the experiments was sodium chloride (NaCl) which was dissolved in demineralized water with negligible ground conductivity. The sensitivity of the conductivity change as a function of the salt concentration was an advantage used to quantify the washing experiments. The contact angle between the glass beads and the mother liquor was determined to be 46° measured using a microscope.

The main parameter of influence was the wash liquor flow rate which was adjusted by varying the pressure of the wash liquor tank for constant nozzle geometry. Very low pressures were avoided due to the indefinite flow state of the nozzle spray. Additionally, the filtrate rate was measured gravimetrically. The varied parameters are listed in Table 2.

4. Results and discussion

In the papers presented by Wakeman (1998), Fuchs et al. (2003) and Ruslim et al. (2005) the problem of inhomogeneous wash liquor distribution in washing pre-dewatered filter cakes was discussed. The cake saturation may decrease rapidly after the cake building phase where the mother liquid drains out of the cake leaving empty pores which are filled with air. These empty pores and particularly the particle surface are difficult to re-wet by the wash liquor so that not all areas are accessible for the wash liquor. Hence, the impurities remaining in these stagnant regions cannot be removed by the wash liquor and it constitutes the remaining impurity fraction. Impurity removal during washing can only take place either by displacement, dispersion or diffusion. For all three mechanisms, a sufficient contact or at least an indirect hydraulic connection such as adsorbed liquid layer, liquid bridges in the particle contact areas, filled pores, etc. must be guaranteed. An illustration of the phenomena is given in Fig. 4 where, despite an attempt at achieving an even distribution of the wash liquor on the cake surface using a full cone nozzle, the flow in the middle of the cake can still become inhomogeneous. The wash liquor which is displayed as a light grey in Fig. 4, first wets the particle surface in the top layer as a film and then it retracts into single channels and filaments. They meander and split on their way through the filter medium. It seems that the larger the distance from the cake surface is the more irregular is the flow texture and distribution of the liquid. The effect is even worse for a broad particle size distribution which corresponds to a likewise broad pore size and thus capillary force distribution. Small empty pores which have higher capillary forces will try to draw and suck the moisture in the nearby areas and leave the larger pores empty. Thus the mother liquor that is loaded by the impurity (dark areas) remains

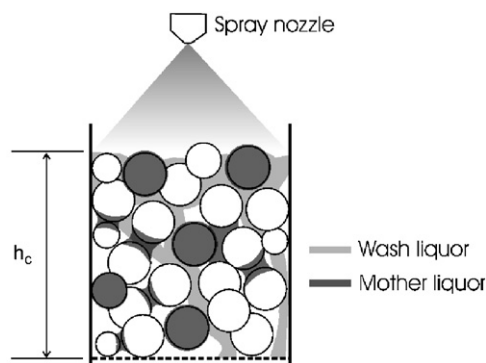


Fig. 4. Inhomogeneous wash liquor flow through a filter cake.

unaffected in areas such as in the large pores, particle surface or contact points. This liquid flow texture at low flow velocities is known as trickle flow in trickle-bed reactors (Gianetto and Specchia, 1992). These findings in washing processes were supported by measurements using magnetic resonance imaging (Ruslim et al., 2005, 2006a) and fluorescent materials such as fluorescein (Ruslim et al., 2006b). The inhomogeneous liquid distribution and problems of solids wetting were also found as typical symptoms in catalyst wetting in the trickle flow regime (Ravindra et al., 1997).

When parts of the filter cake are empty and the moisture content of the particle surface is low, the re-wetting process is energetically unfavourable. The liquid phase usually trickles by creating new interfaces. The area of the new interface is kept small due to the principle of energy minimization. The change of the free enthalpy due to surface area change is described as a coefficient of the total differential of the free enthalpy (or in literature such as Lyklema (1995) it is known as Gibbs free energy) G in

$$dG = \left(\frac{\partial G}{\partial T} \right)_{P,A} dT + \left(\frac{\partial G}{\partial P} \right)_{T,A} dP + \left(\frac{\partial G}{\partial A} \right)_{P,T} dA, \quad (6)$$

where P is the external pressure, A the surface area and T the temperature. Since the interface enlargement is affiliated to energy, interfaces tend to contract (Doerfler, 1994; de Gennes, 1985).

An increase in the flow rate leads to a higher flow velocity and thus kinetic energy. This can be used to enlarge interface and promote the re-wetting process.

4.1. Influence of the wash liquor flow rate

The increase of the wash liquor flow rate seemed to be a possible solution to the macroscopic problem of inhomogeneous flow through pre-dewatered filter cakes. Fig. 5 shows the wash results in terms of impurity concentration ratio (c^*) as a function of the wash ratio (W) at different wash rates (4.3; 12.4 and 31.7 cm³/s). The cake was made of 3700–4100 μm glass spheres which were pre-dewatered to a saturation of 0.18 which corresponded to the film flow regime. The Bond number defined in Eq. (5) was calculated to be 54 which fell into the third Bond regime (compare to Fig. 2) according to the results

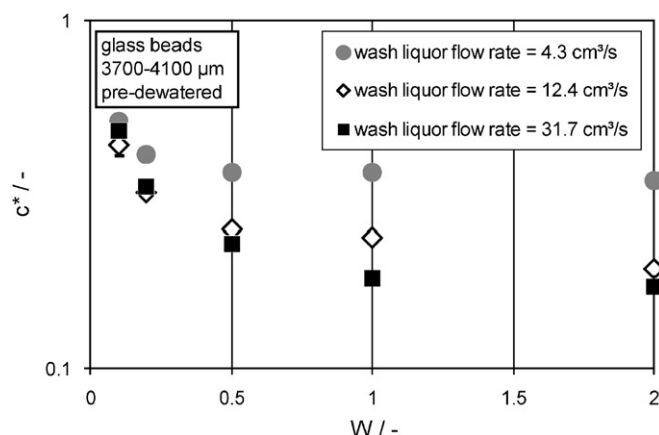


Fig. 5. The impurity concentration ratio (c^*) as a function of the wash ratio (W) of glass beads (3700–4100 μm) after pre-dewatering and washing at different wash liquor flow rates.

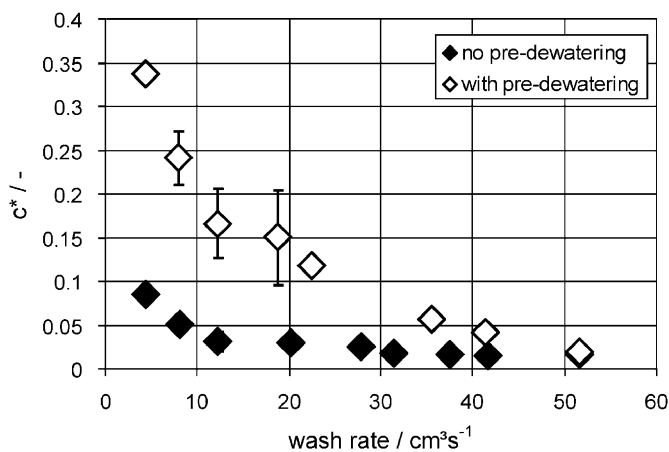


Fig. 6. The impurity concentration ratio (c^*) as a function of the wash liquor flow rate for washing experiments using initially saturated cakes of glass beads (3700–4100 μm) at a constant wash ratio of two ($W=2$) in comparison to washing of pre-dewatered cakes at the same conditions.

from Wuensch (1994) and verified by separate measurements in this work. So the residual moisture stayed as liquid bridges and surface liquid.

The results show that the concentration ratio is lower, the higher the wash liquor flow rate is. Lower concentration ratio means better washing. Higher flow rates might enforce flow not only through small pores but also larger ones, where the capillary forces are not very large. In addition, the kinetic energy of the flow could be used to force re-wetting through larger surface areas of the particles. At high wash rates, the cake was refilled and it was possible for the wash liquor to access the empty pores so that contact between the mother and the wash liquor could be assured. This could explain the better washing at higher wash rates.

A correlation between the wash rate and the impurity concentration ratio is given by Fig. 6. First the influence of the wash rate on washing *pre-dewatered* cakes will be discussed

(empty symbols). Here an increase of the wash liquor flow rate in the investigated range between 4 and 52 cm³/s always led to a decrease in impurity concentration ratio. The results were taken from experiments at a constant wash ratio $W = 2$ in the diffusion regime (compare to Fig. 1 wash curve regimes) where the remaining impurity concentration already stayed constant unaffected by further increase of the wash ratio.

As a comparison, the results of experiments using 3700–4100 μm spheres where the cake was *not pre-dewatered* before washing (black symbols in Fig. 6) will be discussed. The decrease of the impurity concentration ratio as a result of increasing wash liquor flow rate was observable for wash rates up to 12 cm³/s. Above this rate, only a slight improvement of the results by a further increase of the wash rate could be seen. So the increase of the wash liquor flow rate seemed to have stronger influence on improving washing pre-dewatered cakes, whereas the results of washing cakes without pre-dewatering were already good so that an increase of the wash liquor flow rate above 12 cm³/s did not give significant improvement.

During washing initially saturated cakes ($S_0 = 1$), a decrease in cake saturation could still happen during washing if using low wash rates, since the cake resistance of the coarse particles was relative low, so that the filtrate drained faster than the wash liquor that was applied as small droplets by the nozzle. Nevertheless, different to the mechanism in washing pre-dewatered cakes ($S_0 < 1$), here the empty space was immediately refilled by the incoming wash liquor, so the capillary surface was still wet. This was presumably the main reason for the better washing of non-pre-dewatered cakes in comparison to pre-dewatered ones.

The flow and re-wetting problems seemed to have a larger impact on washing pre-dewatered cakes since the initial saturation of the cake before washing was lower. By applying the wash liquor directly when the filtrate began to drain, the wash liquor filled and wetted the space and surface left by the draining mother liquor instantly. Thus the above mentioned problems could be avoided in the first place so that an increase of the wash liquor flow rate up to very high rates became unnecessary.

4.2. Influence of the filtrate flow rate

An increase in the wash liquor flow rate led to a higher filtrate flow rate since larger volumes of liquid were applied in the interval of time exposed to gravity and the driving force for drainage is proportional to the mass of liquid. Hence the liquid was accelerated faster. The wash liquor flow rate seemed to determine the liquid accumulation inside the cake and thus the filtrate flow rate.

For pre-dewatered cakes ($S_0 < 1$), the measured filtrate rates were, however, lower than those measured using saturated cakes. This agrees with the considerations proposed by Wyckoff and Botset (1936) who accounted for the saturation state of the cake by introducing the relationship between the liquid permeability $p_{c,L}$ and the saturation S :

$$p_{c,L}(S) = p_c \cdot p_{c,rel,L}(S). \quad (7)$$

Brutsaert (1967) proposed a power approach to determine the relative liquid permeability:

$$p_{c,rel,L} = \left(\frac{S - S_r}{1 - S_r} \right)^n \quad (8)$$

using the remanent saturation S_r as a reference. The exponent n is larger than zero and must be determined empirically. Since the cake permeability is approximately proportional to the filtrate volume rate according to

$$\dot{V}_{filtrate} = \frac{A \cdot \Delta p \cdot p_c}{\eta}, \quad (9)$$

by comparing the filtrate flow rates for different saturation states with the filtrate flow rates at the saturation of one using constant material parameters the filtration area A , the driving force Δp and the liquid viscosity η , the exponent n can be determined. In this work, the exponent was calculated to be between 1.3 and 3.3. These values conform closely with the values determined by Anlauf (1986) between 2 and 4 but are lower than those determined by Brutsaert (1967) which lie between 3 and 3.5. Anlauf assumed that the approximation function of $p_{c,rel,L}$ depends strongly on the pore size distribution of the cake as proposed by Wakeman (1976b). Wakeman introduced a pore size distribution index λ which was correlated to the exponent n in (10) by

$$n = 3 + \frac{2}{\lambda}. \quad (10)$$

He suggested for a general estimation of the relative liquid permeability for filter cakes using $\lambda = 5$ thus $n = 3.4$. The lower value of our work may refer to the narrow pore size distribution of the glass spheres used in the experiments.

The experimental washing results are plotted against the measured filtrate rates in Fig. 7, both for pre-dewatered ($S_0 = 0.18$) and not pre-dewatered ($S_0 = 1$) filter cakes. This graph shows a strong correlation between the impurity concentration ratio and the filtrate flow rate. Regardless of whether the cake

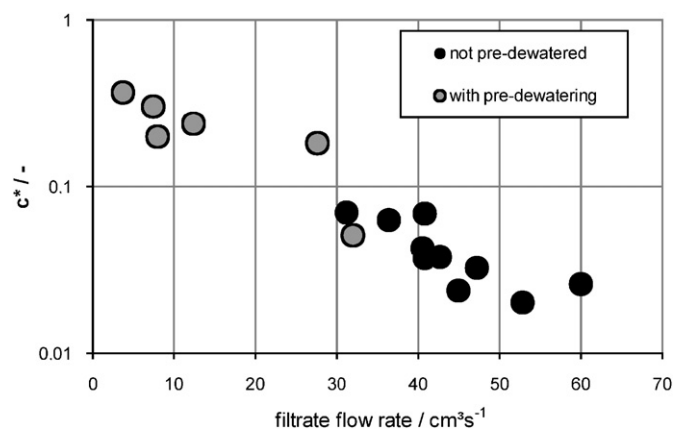


Fig. 7. The impurity concentration ratio (c^*) of washing experiments using glass beads (3700–4100 μm) with and without pre-dewatering for a variation of filtrate flow rates.

was pre-dewatered before applying the wash liquor or not, the impurity concentration ratio decreases with increasing filtrate flow rate. The better results of washing at high filtrate flow rates could be explained by the higher convection due to the liquid flow and also the better distribution of the wash liquor inside the cake. Both were caused by the liquid accumulation at high wash rates, leading to high filtrate rates.

To determine the dispersion regime of the operating conditions the Péclet number Pe was calculated according to

$$Pe = \frac{u_l \cdot d}{D} \quad (11)$$

by using the liquid flow velocity u_l converted from the liquid flow rate using the column filtration area multiplied by the cake's porosity ($A = 2.35 \times 10^{-3} \text{ m}^2$), the sphere diameter for the characteristic length d and a diffusion coefficient for NaCl in water $D = 1.61 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$ at 298.15 K according to CRC (2003). The values of the relevant Péclet numbers in these studies were calculated to be between 5×10^3 and 8×10^4 which correspond to the dispersion regime IV according to the characterization of Pfannkuch (1963). In this regime, the mechanical axial dispersion, which is mainly a back-mixing process due to liquid contact and different path way lengths of the liquid (Fried and Combarnous, 1971), dominates the flow and mass transfer process. Diffusion processes can be disregarded. The Péclet numbers calculated for non-pre-dewatered cakes are generally higher than those calculated for pre-dewatered cakes since they depend on the volumetric filtrate flow rates. These were higher for initially saturated cakes due to more liquid accumulation in the cake.

The Reynolds numbers varied depending on the flow rate between 1 and 7 indicating laminar flow. They were calculated using the hydraulic diameter of the filter cake as the characteristic length d_h , the liquid kinematic viscosity ν and the filtrate velocity in the cake. In addition, the Froude numbers ranged between 0.007 and 0.069 which were comparable to the data of a subcritical flow through obstacles. Both numbers indicated no signs of turbulence (eddies) or waves which could have supported mixing processes.

4.3. Influence of the particle size

As comparisons to the size fraction 3700–4100; 2000–2300 and 400–600 μm glass spheres were also tested. Since the fraction 2000–2300 μm showed very similar results to those of the fraction 3700–4100 μm , only the results of the smallest fraction will be discussed in this work.

A variation of the particle size affects the cake resistance and thus the drainage behaviour of the filtrate. The cake resistance of the filter cake built out of the fraction 400–600 μm was measured by a flow permeability measurement and determined using Eq. (9) to be $8 \times 10^9 \text{ m}^{-2}$. This is rather low compared to industrial relevant products that are processed in filters (Anlauf, 1986) but considering the size of the particles, this was expected. Higher cake resistances would not be practicable since the driving force for the dewatering was only the earth gravity. The Bond number calculated for this product was

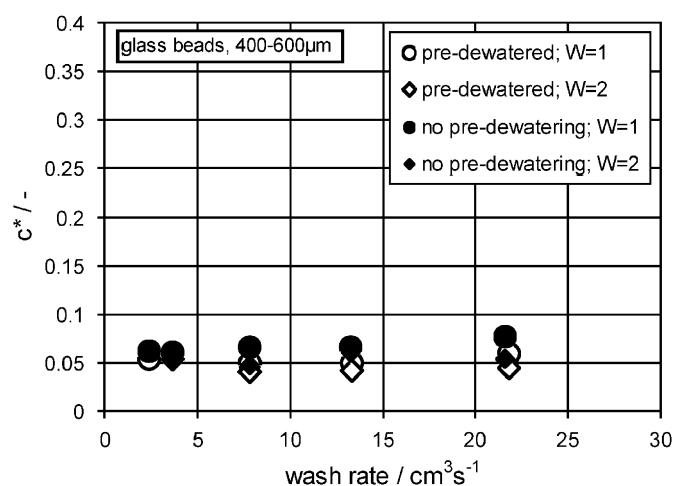


Fig. 8. The impurity concentration ratio (c^*) as a function of the wash liquor flow rate after washing initially saturated cakes and pre-dewatered cakes of glass beads (400–600 μm) at wash ratios of one and two ($W = 1; 2$).

12 which was still in the second Bond regime (compare to Fig. 2) according to literature data (Wuensch, 1994) and our own measurements. So the residual moisture in the cake at equilibrium was situated as liquid bridges, surface liquid and capillary rise. The cake was pre-dewatered down to a saturation of 0.5 which corresponded to the plug flow regime of the deliquoring kinetics.

Fig. 8 shows the impact of wash rate variation on washing pre-dewatered and initially saturated cakes for glass beads of the size of 400–600 μm . The wash results were hardly affected by the variation of the wash liquor volumetric rate. This was observed for both pre-dewatered ($S_0 = 0.5$) and not pre-dewatered ($S_0 = 1$) cakes. In both cases, the particle surface was still wet and the moisture content of the cake was sufficient, so that re-wetting was not a problem. This is the difference to the results observed while using larger glass beads, 3700–4100 μm as shown in Fig. 6 where the cake moisture content was already very low ($S_0 = 0.18$) prior to adding the wash liquor and therefore the wash results were bad at low wash rates. So the low moisture content of the cake of larger glass spheres (3700–4100 μm) was most likely responsible for the bad wash results of the pre-dewatered cakes especially at low wash liquor flow rates, whereas the cake built with the smaller beads (400–600 μm) remained wet even after pre-dewatering so that the washing was good and a change of the wash liquor flow rate did not bring further improvements.

4.4. The flow ratio FR

For practical applications it would be very advantageous to have a direct correlation between the wash results and the process parameters. Based on the results presented above, the wash results using glass spheres depend on the wash liquor flow rate and also from the filtrate rate. The filtrate rate is generally determined by the predominant wash rate and the material properties, thus also the particle size. Therefore the material

properties should be taken into account in finding a correlation between the wash results and the flow properties.

This led to the consideration of building the direct ratio between the wash liquor flow rate and the filtrate rate. This ratio can be directly used in the practice to set optimum wash rate for known material properties based on lab-scale investigations. Since the instantaneous filtrate rate may vary during the washing process, a good reference value for the ratio could be the liquid flow rate through a constantly full saturated filter cake ($S = 1$) at stationary state. This can be determined in a flow experiment at constant driving force. This flow rate is to be called the D'Arcy flow rate which is related to the cake permeability p_c according to D'Arcy (1856) for a laminar flow through a porous structure at full saturation

$$\dot{V}_{\text{Darcy}} = \dot{V}_{\text{filtrate}} = \frac{p_c \cdot \Delta p \cdot A_{\text{filtration}}}{\eta_l} \quad (12)$$

During the flow experiment, all other influence parameters such as the driving force, the dynamic viscosity η_l of the liquid, the permeability and the filtration area $A_{\text{filtration}}$ have to be maintained constant. The permeability is a function of the sauter mean diameter $x_{1,2}$ and the porosity ε according to Gupte (1970):

$$p_c = \frac{1}{5.6} \cdot x_{1,2}^2 \cdot \varepsilon^{5.5} \quad (13)$$

So the structure of the cake and the particle size distribution are taken into account in the D'Arcy flow rate as influence parameters.

The results of the application of the ratio to describe the wash results of pre-dewatered glass spheres are shown in Fig. 9. By plotting the wash results as a function of the flow ratio FR :

$$FR = \frac{\dot{V}_{\text{wl}}}{\dot{V}_{\text{Darcy}}} = \frac{\text{wash liquor flow rate}}{\text{filtrate flow rate at } S = 1} \quad (14)$$

the behaviour of the pre-dewatered cakes of different particle sizes can be compared. The ratio FR gives also indirect information on the saturation state of the cake during the washing process which will be discussed more thoroughly in the next section. First the FR regimes will be discussed.

For $FR = 1$ a flow at full saturation can only happen if the wash liquor is introduced on a saturated cake ($S_0 = 1$). This way, the filtrate flow equals the maximum filtrate flow according to Darcy and the identical flow rate of the wash liquor keeps the saturation at a constant level of $S_{\text{wash}} = 1$. In this case a homogeneous contact between the liquids can be guaranteed but axial dispersion could become relevant. If $FR = 1$ and the cake has already been pre-dewatered to an initial saturation $S_0 < 1$, the filtrate drains slower than the incoming wash liquor according to Eqs. (7)–(9) so that the saturation increases up to the stationary state S_{stat} . This is the condition where the instantaneous outcoming filtrate rate equals the incoming wash liquor flow rate. Whether this condition is achievable during washing or not, depends on the wash ratio and the wash duration. In Fig. 10 the stationary state is marked during washing (W) by the constant level of saturation.

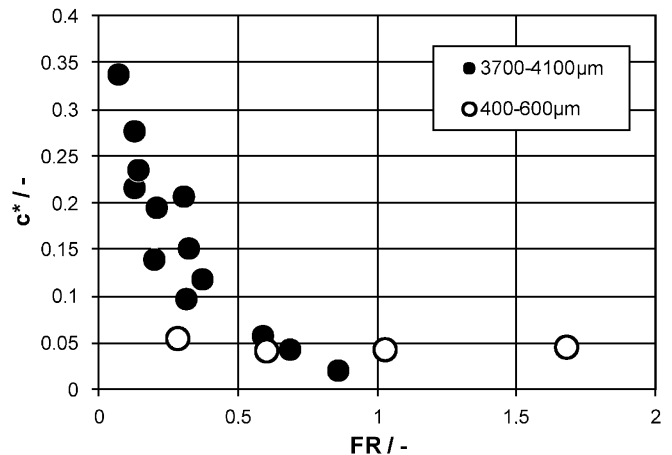


Fig. 9. The impurity concentration ratio (c^*) as a function of the flow ratio (FR) for glass beads (3700–4100 and 400–600 μm) after pre-dewatering.

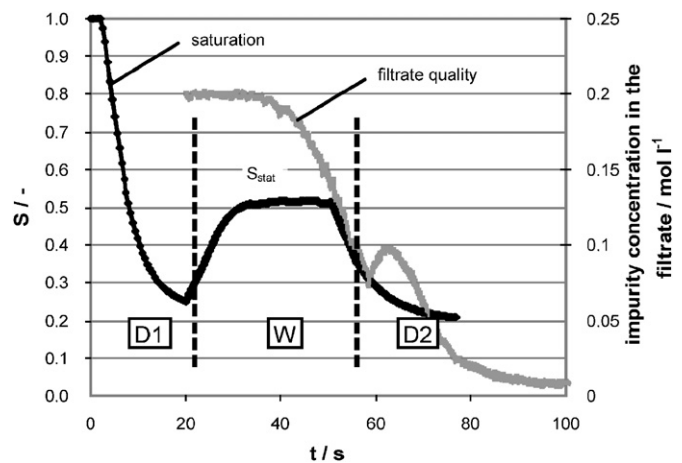


Fig. 10. The saturation progression during washing a pre-dewatered cake of glass beads (3700–4100 μm).

For high values $FR > 1$, the cake can be over-saturated in terms of flooding when the initial saturation $S_0 = 1$ which is the case for *no* pre-dewatering. Otherwise, if the initial saturation is below one ($S_0 < 1$), the liquid level will rise and the cake will be resaturated. In both cases, a high over-saturation ($S_{\text{wash}} > 1$) normally results in a mixing between the liquids before the wash liquor drains into the cake, thus the wash results are mostly very poor (Heuser, 2003).

For $FR < 1$ and if the cake is fully saturated at the beginning of washing ($S_0 = 1$), the cake will then be desaturated during washing. On the other hand, if the cake has already been pre-dewatered ($S_0 < 1$), it is not obvious whether the saturation then increases or decreases. If the initial saturation S_0 is lower than the stationary level S_{stat} , which is a function of the wash liquor and the D'Arcy flow rate, thus a function of FR , then the cake will be resaturated. Otherwise the cake will continue to desaturate down to the stationary saturation level.

The level of the stationary saturation during washing should be the same for identical wash liquor flow rates at constant material parameters as shown by Ruslim et al. (2005).

Table 3
Characterization of the saturation state of the cake from the flow rates and the related wash results

FR	Comparison	No pre-dewatering $S_0 = 1$	Pre-dewatered $S_0 < 1$
1	$\dot{V}_{wl} = \dot{V}_{Darcy}$	Single phase flow, saturation stays constant	Pores of the cake will be refilled, saturation increases
> 1	$\dot{V}_{wl} > \dot{V}_{Darcy}$	Cake will be flooded, mixing of the liquids	Increase of the cake saturation during washing, pores will be refilled
< 1	$\dot{V}_{wl} < \dot{V}_{Darcy}$	Decrease of cake saturation, consecutive film flow of mother and wash liquor	Low moisture content, two phase flow → stagnant areas, poor distribution of the wash liquor

The summary of the *FR* and the observed phenomena of the cake are listed in Table 3.

According to Fig. 9, the best wash results were achieved for *FR* near one. For larger glass beads (3700–4100 μm) the improvement of the wash results with increasing *FR* was found to be very distinct. For smaller glass beads (400–600 μm) there seemed to be a weak dependency of the impurity concentration ratio on the flow ratio *FR*. The message from these findings is to keep the flow ratio *FR* high since this is advantageous for washing pre-dewatered cakes ($S_0 < 1$). It is not advisable to wash pre-dewatered cakes with low wash liquor flow rates if the cake is very permeable since the D'Arcy filtrate rate can become larger than the wash liquor flow rate and thus the flow ratio *FR* becomes small. In this regime, the cake is washed inhomogeneously and the impurity concentration ratio becomes high.

4.5. The dynamic saturation

This last section deals with the question seeking the physical background of the analysed results. The above defined ratio *FR* is convenient in aiding process optimization but there has to be more physical aspects related to this parameter.

During a washing process the cake saturation changes. The saturation progression can be seen representatively in Fig. 10. At the beginning of the process, the cake was still saturated, meaning that the pores and void spaces were filled with the mother liquor. With proceeding time, the cake was desaturated during exposition to a driving potential such as earth gravity or centrifugal acceleration (Phase *D1*). After opening the wash liquor inlet nozzle, the cake saturation increased, meaning that the pores and void spaces were refilled by the wash liquor as shown in the phase *W*. During this phase, mass transfer processes such as dilution or mixing and displacement took place inside the cake. The saturation level stayed at a constant level during washing when the filtrate rate equalled the wash liquor volume rate. After the wash liquor input was stopped (phase *D2*), the saturation decreased in an identical way as in the pre-dewatering phase. The most significant wash effect was marked by a decrease in the impurity concentration which was shown in the washing (*W*) and post-dewatering (*D2*) phase.

Since the moisture content of the cake during washing depended on the flow ratio, it was assumed that the cake saturation during washing might be of great importance for the

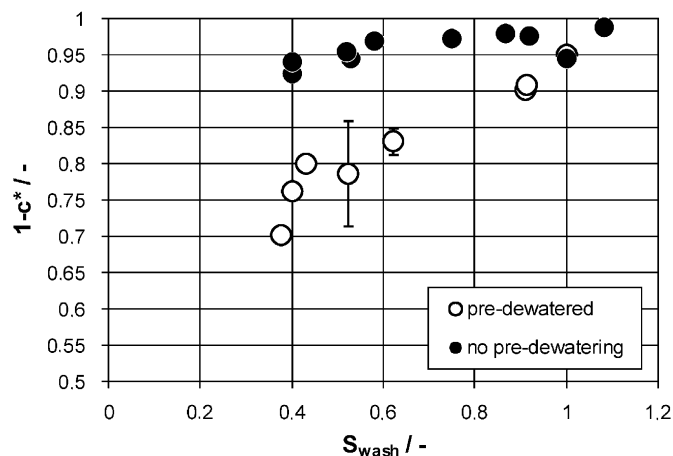


Fig. 11. The washing efficiency ($1 - c^*$) as a function of the saturation state during washing (S_{wash}) for both cases, with and without pre-dewatering.

wash results. In Fig. 11 the wash efficiency $1 - c^*$ is plotted instead of the normal concentration ratio c^* as a function of the cake saturation during washing S_{wash} . Washing initially saturated cakes ($S_0 = 1$) was always of good efficiency (nearly full efficiency) even though a desaturation of the cake during washing might occur due to low flow ratio ($FR < 1$). On the other hand, for pre-dewatered cakes, full wash efficiency could only be achieved if a complete resaturation of the cake was done. Otherwise, the efficiency dropped sharply if only a partial resaturation after pre-dewatering was practicable. This is a relevant case in the practise as in washing coarse crystalline products in pusher centrifuges. So this should arouse awareness in industrial applications since a slight pre-dewatering of the cake due to late application of the wash liquor may lead either to a significant loss in product purity or to an excess in wash liquor consumption and process duration.

The difference between the flow phenomena during washing initially saturated ($S_0 = 1$) and pre-dewatered ($S_0 < 1$) filter cake was that the saturation of the initially saturated cake remained high at all times, so that a homogeneous contact between the mother and wash liquor in nearly all cake areas was allowed. During washing pre-dewatered cake, the pores had to be filled first and this might happen inhomogeneously so that not all areas were accessible for the wash liquor. The resaturation quality and thus the wash efficiency in the regime of

low wash saturations depend on the product surface property, surface texture, particle shape and the cake structure.

A contribution and recommendation for practical applications must be the need to maintain a high saturation level before (S_0) and during (S_{wash}) washing to enable good and homogeneous distribution and thus contact between the mother and the wash liquor. This consideration is important during the machine and process design. A possible enhancement of the wash efficiency could be a periodical wash liquor application at high flow rates. The flow rate should be adjusted according to the optimum dynamic saturation and the duration of each period could be determined by calculating the time to refill all capillaries of the cake. This way, optimum wash efficiency could be achieved and at the same time the wash liquor consumption reduced to its minimum required amount.

5. Conclusions

In investigations of filter cake washing, the starting point of washing has been found to be an important influence parameter in affecting the results. In industrial processes, a late wash liquor application happens frequently. This leads to a pre-dewatering of the filter cake and thus to a low moisture content. This is disadvantageous for the wash liquor distribution inside the cake, either in the flow or in the surface re-wetting process. The poor distribution of the wash liquor inside the cake results in poor contact between the mother and wash liquor and therefore bad washing. So a late application of wash liquor usually results in worse washing when compared to an application when the cake is at an ideal state of wetness.

In this paper, possible solutions to this problem of washing pre-dewatered cake were discussed. It was found that an increase of the volumetric wash liquor flow rate led to an improvement of the results. A high wash liquor flow rate caused an increase of the cake moisture content since large volumes of liquid were applied in the interval of time. This liquid accumulation made more cake areas accessible for the wash liquor and thus led to an even distribution. Besides, the large volumes induced higher filtrate flow rates that might enforce a homogeneous flow and enhanced the re-wetting process. By means of convection during the displacement flow, the mass transfer of the impurity could be improved. All these factors contributed to better washing. A strong correlation between the impurity concentration ratio and the filtrate flow rate was found in this work, regardless of whether the cake had been pre-dewatered or not. This underlined the importance of the flow rates as important influence parameters.

The ideal state of cake wetness could be set by adjusting the wash liquor flow rate and at the same time considering the material parameters affecting the cake resistance to determine the filtrate flow rate. A ratio between the wash liquor flow rate and the maximum filtrate flow rate at full saturation was proposed as a way to determine optimum wash rates to achieve homogeneous and even distribution of wash liquor. It was found that washing at high flow ratios, where the wash rates were equal to or larger than the maximum filtrate flow rate, produced better results than washing at low flow ratios. This was also related

to the saturation state of the cake during the washing process. At high wash liquor flow rates and low filtrate flow rates, the liquid accumulated inside the cake, thus the saturation during the wash process became high. This condition around a full saturation of the cake was found to be the best condition for the wash efficiency. However, when the cake had been pre-dewatered before applying the wash liquor, a partial resaturation of the cake did not result in the same wash efficiency as of the identical level of saturation achieved during washing initially saturated cake. So there was a dependency of the efficiency on the pre-history in the process. Probably during the partial resaturation process, a complete homogeneous distribution was not possible. Thus optimum washing should be performed by applying the wash liquor onto a cake near full saturation ($S_0=1$) and maintaining the saturation during washing (S_{wash}) at a high level by setting the appropriate wash liquor flow rate considering the cake resistance.

Notation

A	surface area, m^2
c	concentration, mol/l
c^*	concentration ratio, dimensionless
d	particle size, m
d_h	hydraulic diameter, m
D	diffusion coefficient, m^2/s
D_{ax}	dispersion coefficient, m^2/s
FR	flow ratio, dimensionless
g	surface tension, N/m
G	Gibbs free enthalpy, J
p	pressure, Pa
p_{cap}	capillary pressure, N/m^2
p_c	permeability, m^2
$p_{c,L}$	liquid permeability, m^2
$p_{c,\text{rel},L}$	relative liquid permeability, dimensionless
Δp	pressure difference, bar
$\Delta \eta$	dynamic viscosity, Pa s
Pe	Peclét number, $Pe = u_l \cdot d/D$, dimensionless
S	saturation, dimensionless
S_0	initial cake saturation just before washing, dimensionless
S_r	remanent saturation, dimensionless
S_{stat}	saturation at stationary level, dimensionless
S_{wash}	dynamic saturation during washing, dimensionless
T	temperature, K
u_l	liquid velocity, m/s
\dot{V}_{Darcy}	filtrate flow rate for saturated cakes according to Darcy, cm^3/s
\dot{V}_f	filtrate flow rate, cm^3/s
\dot{V}_{wl}	wash liquor flow rate, cm^3/s
W	wash ratio, dimensionless
x	loading, kg/kg
$x_{1,2}$	sauter mean diameter, m

Greek letters

γ	interfacial tension, N/m
ε	porosity, dimensionless

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