Continuous diafiltration of fine-particle suspensions with a double-shaft disc filter

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Cake filtration and subsequent washing of the formed filter cake mostly cause problems in case of suspensions containing ultrafine particles. The problems are due to a high filter cake resistance and often an uneven flow through the filter cake, so that displacement washing, which is desirable in most cases, is inadequate. To cope with this difficult situation, diafiltration is performed to handle these types of suspensions. Thereby the suspension is initially concentrated until a high solid content is reached. Afterwards the washing fluid is added. At the same time or in a sequence, this liquid mixture is removed again by using dynamic filtration. High wall shear stresses on the filter medium with low pressure differences are desired here. Shear gap filters with rotating filter disks that are aligned on two hollow shafts and overlap meet these requirements.

The rotation of the disks and the continuous movement of the suspension can prevent the formation of a filter cake and also makes it possible to work with suspensions with a high solid content. The shear stress generated in the filter and the rheological properties of the suspension play an important role. These parameters are influenced by the type of particle class, the solid substance concentration and the process parameters. All these parameters have an effect on the washing result, the washing fluid quantity and the washing time. In the following we will report about diafiltration tests with the dynamic Krauss-Maffei Crossflow Filter DCF containing disk-shaped ceramic membranes.

1. Introduction

A suspension contains particles, e.g. crystals, precipitation products, microorganisms which are dispersed in a so called mother liquor. In process engineering it is often the task to replace the mother liquor by a washing fluid. The mother liquor often contains dissolved substances that are to be washed out with the washing fluid. These dissolved substances may either be undesired contaminations or they can be the valuable product to be recovered. Moreover in some cases, a desired or undesired extraction of substances from the particles must be considered when selecting an appropriate washing fluid.

Washing is initiated mostly after concentrating the suspension. It is beneficial to obtain a very high solid substance concentration of the material already prior washing to have the mother liquor volume already reduced substantially. During the washing stage, the mother liquor is replaced by the washing fluid. Often the target is to reach low residual concentrations of the mother liquor in the agglomerate or suspension, especially in the pharmaceutical industry. Clearly the selection of the right washing method and suitable washing liquid are of great importance. Hoffner [1] provides an overview of the washing methods and their optimum fields of application.

Washing of suspensions containing ultrafine particles is a particularly demanding task. Erk [2] and Alles [3]
show that the cakes of colloidal systems form dense packages and are highly compressible. This leads to high flow resistance, which results in long filtration and washing times. Wiedemann [4] reports about the high risk of cake cracking of compressible cakes, which deteriorates filtration and washing substantially.

In order to counteract this problem, Heuser [5] investigated jet washing of highly concentrated agglomerates with thin cakes. A resuspension of the filter cake took place upon adding the washing fluid in this case. As an alternative to this, Hoffner [6] developed an apparatus in which fluid-saturated agglomerates move bulk-like through one or several serially aligned washing chambers.

Alternatively to the aforementioned methods, washing can be performed directly with concentrated suspensions. In this context, a dynamic filtration process is performed: Washing liquid is either added subsequently or simultaneously. This method is called diafiltration. With these filters, the mother liquor exits the filter through a channel system aligned radially to the hollow shaft on the inside of the filter disks due to a pressure difference. In most cases, disk-shaped ceramic membranes are used where a suitable pore system can be created that can cope with filtering also ultrafine particles.

Taamneh [7] tested disk filters for particle classification with overlapping and non-overlapping rotating screening disks. Steinke [8] showed that it is possible to perform top layer-free filtration with a single-shaft disk filter by means of high-frequency backwashing.

2. Theoretical considerations

During dynamic filtration, different forces act on the particles due to the disk rotation. Hydrodynamic forces act as long as the particles are in motion. Adhesion and friction forces act additionally between particle and membrane on a separated particle. In this report, especially concentrate on the hydrodynamic forces. Due to these forces, the particles are in constant motion until they deposit in layers (top layer) or exit the filter as concentrate. With dynamic filtration, a dynamic equilibrium of particle feed and discharge establishes on the surface of the filter disk. According to Altmann [9], the position of the equilibrium is determined by the wall shear stress on the top layer surface on the one hand, and by the transmembrane pressure difference on the other hand.

In the course of classical cross-flow membrane, a high flow speed is enforced on the surface of the membrane, which generates a high wall shear stress. This also causes a high transmembrane pressure difference at the module feed. In contrast, disk filters do not exhibit such a strong link between the wall shear stress and the transmembrane pressure difference. The wall shear stress is primarily influenced by the rotation speed of the discs. These disk filter properties are desirable above all for suspensions with compressible filter cakes, because the throughput pressure difference curves experience a maximum according to Berndt [10].

With disk filters, the wall shear stress depends on the rotation speed of the disks and the prevailing geometric conditions. A shear gap is formed in disk filters with overlapping disks in which the wall shear stress reaches high values, especially when the gap has a small width (fig. 1). A shear gap can be formed by at least two contrarotating filter disks. With contrarotating, overlapping disks, the relative speed above the shear gap is constant in all domains, so that uniform filtration conditions prevail in the shear gap. Disk filters with and without shear gap by means of rotating disks are available with disk diameters of 300 mm and above. Today this filter type is technically feasible up to a filter surface of 20 m² and more per unit. Of course, more surface area can be realised by using a multiple set-up.
3. Experimental investigations

3.1 Pilot plant

The washing tests were performed with the dynamic Krauss-Maffei Crossflow Filter DCF 152/0.14 manufactured by Andritz KMPT GmbH. The filter consists of two horizontal hollow shafts on which up to 6 filter disks can be aligned offset to each other at a distance of 3 mm. The two outer disks consist of metal and are impermeable. The applied filtering disks are ceramic membranes with an average pore width of 0.2 μm. The outer diameter of the disks is 152 mm. Together with the inner diameter of 40 mm it results in a filter surface of approximately 0.14 m².

Throughout the test, the suspension is conveyed via a pump from an agitated 12-litre tempered feed vessel through the filter chamber in a circuit. In the filter, the suspension is divided into a retentate and a filtrate that penetrates through the disks. The volume flows are continuously determined during the tests via two flow meters positioned on the concentrate and the filtrate side. In order to be able to detect the success of the washing, the suspensions are marked by a 0.1 molar saline solution. Ergo the salt is the substance to be washed out. A conductivity cell on the filtrate side provides information about the salt concentration at any point of time. The pressure drop of filtration, which was always kept at Δp = 1 bar in the results presented here, is determined via the pressure sensors in the filter chamber and on the filtrate side.

Discontinuous and continuous washing can be realised in the disk filter. Fig. 2 shows the P&I flow chart of the test system.

3.2. Particle class

The tests were performed with aluminium oxide (Almatis GmbH, CT 3000). The suspensions were prepared using a 0.1 molar saline solution. The particle size distribution of the Al₂O₃ ranges from 0.1 μm to 9 μm, whereby the modal value is at approximately 3.5 μm. This is measured with the laser diffraction analyser Horiba LA 950.

4. Results of the experiments

With disk filters, the suspension stays in motion throughout the entire test period. Combined with the disk rotation, this leads to high shear stresses that keep the viscosity of the suspensions low. The concentrated Al₂O₃ suspensions have a viscoelastic and thixotropic behaviour. Furthermore, the centrifugal force on the rotating disks reduces the formation of a thick top layer.

The flow properties of the suspension determine the upper solid substance concentration limit at which diafiltration can be performed. One limit is that the solid substance can only be thickened up to the pumpability limit. Concentrated suspensions often have a shear thinning flow behaviour. This makes it possible to substantially reduce the viscosity through right selection of the suspension shearing situation.

Removal of larger filtrate quantities leads to an increase of the solid substance concentration of the suspension. As a result, the viscosity also increases. It is therefore important to test the influence of the suspension’s solid substance concentration prior to diafiltration.

Experiments were conducted with aluminium oxide at an initial concentration of $\phi_0 = 0.01$, a temperature of $\Theta = 20^\circ$ and a disk rotational speed of $n = 1400$ min⁻¹. Prior to continuous feed of the washing fluid at an initial volume of $V_0 = 12.5$ L, a filtrate volume of $V_f = 0$ L, $V_f = 2$ L and $V_f = 4$ L, respectively, were withdrawn from the suspension. This resulted in a concentration volume of $V_{con} = 10.5$ L, $V_{con} = 8.5$ L and $V_{con} = 6.5$ L in the individual suspensions, respectively. The specific filtrate volume flow as a function of the washing duration is displayed for the individual tests in fig. 3. The specific filtrate volume flow remains constant throughout the entire washing process during a continuous diafiltration. The concentration of the suspension has the effect that the specific filtrate volume flow decreases only slightly. Therefore one can assume a marginal top layer formation, which is also influenced by the solid substance concentration.

The great advantage of dynamic filters with rotating disks is the possibility to work with suspensions at a high solid substance concentration. The aluminium oxide suspensions were washed continuously with an initial volume of $V_0 = 14$ L and a volume concentration of $\phi_0 = 0.2$. The test temperatures and the rotational speed of the disks were kept constant at $\Theta = 20^\circ$C and $n = 1400$ min⁻¹, respectively. Prior to continuously adding the washing fluid, the suspensions were concentrated to $V_{con} = 12$ L and $V_{con} = 8$ L, respectively. In doing so, the concentration of the individual suspensions was increased to $\phi_{con} = 0.23$ and $\phi_{con} = 0.33$. Fig. 4 shows the specific filtrate volume flow during diafiltration. One can initially discern a decline of the filtrate volume flow in the concentration step. Once continuous feed of the washing fluid sets in, a steady rise of the specific filtrate flow is observed at a concentration $\phi_{con} = 0.23$, as well as at a concentration $\phi_{con} = 0.33$. One reason for this rise is the feed of the washing liquid which is added in the filter chamber. As a result, the solid substance concentration in the feed of the washing fluid sets in, a steady rise of the specific filtrate flow is observed at a concentration $\phi_{con} = 0.23$, as well as at a concentration $\phi_{con} = 0.33$. One reason for this rise is the feed of the washing liquid which is added in the filter chamber. As a result, the solid substance concentration in
the filter chamber is not homogeneously distributed in the chamber. It must also be considered that the electrolyte content of the suspension changes. These factors may also change the top layer. However, with a significantly higher solid substance concentration, the specific filtrate flow experiences a stronger decrease. This can be associated with a thicker top layer. In tests with low concentration suspensions, the described rise was not observed after adding washing fluid. This may be due to the thinner top layers which in turn explain the high filtrate flows.

The washing effect can be characterized by the non-dimensional residual salt content $c^*$:

$$c^* = \frac{c}{c_0}$$  \hspace{1cm} (1)

where $c$ denotes the current salt concentration and $c_0$ represents the concentration at the start of a washing test.

In the disk filter washing can take place in a discontinuous or continuous mode. Generally the suspension is concentrated in both washing methods initially. For this purpose, filtration is performed first and the volume is reduced from $V_{\text{in}}$ to $V_{\text{con}}$, whereby the solid content is retained basically completely by the filter medium. This is why the following concentration ratio is often referenced in diafiltration:

$$V_{\text{in}} = V_{\text{con}} \frac{\phi}{\phi_{\text{con}}}$$  \hspace{1cm} (2)

In the mode of continuous diafiltration, the washing fluid is added to the remaining suspension afterwards in such a way that the suspension volume $V_{\text{con}}$ remains constant although the filtration process is continuous. This means that the added washing fluid volume corresponds to the discharged filtrate flow:

$$V_{\text{w}} = V_{\text{f}}$$  \hspace{1cm} (3)

The concentration of the dissolved salt will decline steadily in the process. The salt is thus washed out. Under the assumption of perfect mixing, the concentration curve in continuous diafiltration can be described in dependence on the total added washing fluid volume $V_w$:

$$c^* = \frac{c}{c_0} = \frac{e^{r_{\text{conc}}}}{e^{r_{\text{conc}}}}$$

For a targeted non-dimensional residual salt content the required washing fluid quantity can be determined by this equation:

$$V_{\text{w}} = \ln(c^*) \cdot V_{\text{conc}}$$  \hspace{1cm} (5)

The required washing fluid volume is thus proportional to the residual volume of the suspension prior to adding the washing fluid. Washing at high solid substance concentrations and a low suspension volume results in less washing fluid to be added. This is beneficial if the washing fluid with the washed out substance is treated in a following process step.

The experimental results displayed in Fig. 3 together with the equation (5) are displayed in Fig. 5. There is excellent agreement indicating that the hypothesis of homogeneous mixing conditions is adequate in this set-up. In case of a desired residual salt concentration of 1%, a reduction of the suspension volume by $V_{\text{f}} = 2$ L and 4 L (which corresponds to a respective increase of the solid content of suspension) results in a reduction of 20 and 40%, respectively. It is interesting to note that the filtrate flow decreases only slightly (see fig. 3). This indicates that a high solid concentration can be more economic in terms of lower washing fluid costs and thus reduced treatment costs.

The washing curve can be mapped with the theoretical description of diafiltration. Ideal mixing of the suspension can be guaranteed even with high concentrations. This is, above all, due to the fact that the rotating disks also act as agitators, and that the filter in combination with the rather small suspension volume with reference to the filter volume can be regarded as an ideally stirred reactor.

5. Summary

The examined rotating disc filter shows excellent performance in terms of filtration and subsequent continuous washing at low and high solid concentration. The processes only limited by the pumpability of the slurry.

With low concentration suspensions, it was observed that the filtrate volume flow remains constant during continuous diafiltration throughout the entire duration of washing. The specific filtrate volume decreases slightly with increasing solid content of the suspension which indicates that only a thin top layer develops on the membrane surface that remains about constant.

However, if suspensions with higher concentrations are washed continuously, the specific volume flow decreases with increasing solid concentration during the concentration stage. There is a clear correlation between the solid concentration and the volume flow indicating that the thickness of the top layer on the membrane surface increases with higher concentrations. In the case of the subsequent continuous washing step the specific filtrate volume flow increases again. Therefore a better economical optimum can be utilized at high solid concentrations where washing is faster and the washing liquid quantity can be reduced.
The washing curve can be mapped for both concentration ranges with the theoretical description of diafiltration. Ideal mixing of the suspension can be assumed safely even at high concentrations. This is, above all, due to the fact that the rotating disks also act as agitators, and that the filter in combination with the low suspension volumes with reference to the filter volume can be considered as an ideally stirred reactor.

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Formula symbols

- $c^*$ [-] Non-dimensional residual salt concentration content
- $c_*$ [mol/L] Concentration of the salt at a specific time
- $c_0$ [mol/L] Initial salt concentration in the suspension
- $n$ [1/min] Disk rotation speed
- $t_w$ [s] Washing time
- $v_f$ [L/h] Specific filtrate volume flow
- $V_0$ [L] Initial volume of the suspension
- $V_{con}$ [L] Volume of the concentrated suspension
- $V_f$ [L] Volume of the filtrate
- $V_w$ [L] Volume of the washing fluid
- $\Delta p$ [bar] Transmembrane pressure difference
- $\vartheta$ [°C] Temperature of the suspension
- $\varphi_0$ [-] Initial solid substance volume concentration in the suspension
- $\varphi_{con}$ [-] Concentrated solid substance concentration

References


Fig. 6: Volume of washing fluid as a function of the residual salinity for continuous diafiltration processes