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FLOW PATTERNS AND SEDIMENT BUILD-UP IN TUBULAR BOWL CENTRIFUGES

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ABSTRACT

Nanoparticles and biological cells like yeast and algae have gained more attention during the past years. The crucial task in the biotechnological and pharmaceutical industry is not only to advance in the formation of highly functional products, but also to assure the separation and classification of these. Semi-continuous solid bowl centrifuges offer high separating potentials and throughputs. Understanding the flow patterns and the influence of the sediment build-up on separation efficiency during a semibatch separation is essential both for the process optimization in existing apparatus and the development of new effective centrifuges. In this work, the separation efficiency and the sediment build-up in a tubular bowl centrifuge is examined for an fine inorganic and a biological product.

KEYWORDS

Bioseparation, Tubular Bowl Centrifuge, Separation Efficiency, Compaction, Capacity, Fluid Mechanics

1. Introduction

During the last years a constant increase in the demand for highly efficient solid-liquid separation processes could be observed. The origin arises from the marketability of various nanoscale products in the pharmaceutical, paint and bioengineering industry, as many agents are produced in biotechnological processes nowadays.

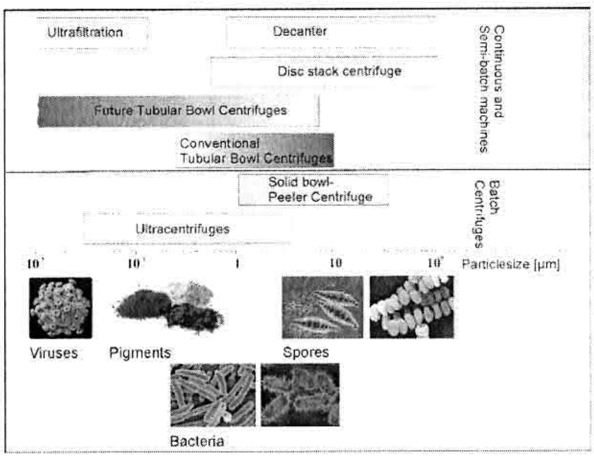

Figure 1: Overview of the application range of different types of centrifuges

The separation of nanoscale particles and biological products is a difficult task as the volume of the particles or the density gradient between the particles and the liquid are rather small. Here centrifuges offer a high clarifying potential and throughputs. Figure 1 shows an overview of the application range for some common centrifuges. The highest centrifugal forces are achieved with ultracentrifuges and tubular bowl centrifuges. If the solids concentration of the feed is very low, an economic process is only possible by using a semi continuous apparatus. Hence the tubular bowl centrifuge could be an efficient unit operation in the biotechnology.
The evaluation of the separation efficiency is essential, because high product losses reduce the profitability and could cause a contamination of subsequent unit operations. A slight increase of the biomass concentration in fermenters requires time-consuming optimization processes. Hence, it is necessary to avoid any product loss in the unit operation solid-liquid separation.

2. Methods and Materials

All experiments were carried out with a tubular bowl centrifuge with a maximal rotational speed of 40000 rpm. The length of the cylinder is 187 mm and the inner diameter of the bowl is 43 mm. The outlet weir diameter is 14.5 mm (see Figure 6). The separation efficiency of Aerosil 200 nanoparticles and a biological product, baker's yeast, was investigated. The Aerosil is a hydrophilic silica with a mean agglomerate diameter of 370 nm, whilst the mean diameter of the yeast is 5 µm. The Aerosil agglomerates are build of sintered primary particles with a mean diameter of 8 nm and a density of 2200 kg/m³, which is twice the density of the yeast with 1150 kg/m³. Both materials are compressible; the Aerosil sediment could be compacted due to the agglomerate's structure, whilst the biological cells are highly flexible and easy to deform.
The evaluation of the separation efficiency is possible via the measurement of the product loss (eq. 1) which is the ratio of the solids volume concentration of the centrate at a certain time, $c_{V,Centrate}(t)$, and of the feed, $c_{V,Feed}$. The solids volume fraction is kept constant for all experiments. The separation efficiency depends on the centrifugal number G (eq. 2) and the residence time. A higher throughput decreases the residence time and thus the separation efficiency.

$$\text{Product loss} = \frac{c_{V,Centrate}(t)}{c_{V,Feed}} \cdot 100\% \qquad (1)$$

$$G = \frac{r \cdot (2 \cdot \pi \cdot n)^2}{g} \qquad (2)$$

Magnetic resonance imaging (MRI) is used for the analysis of the sediment build up. During the centrifuge experiment, 10 ml of carbon black suspension was injected at different times at the inlet. After the experiment, the remaining unfilled core was sealed with paraffin, to avoid any changes of the sediment. The different decay of the signal of the yeast and carbon black allows a non-invasive examination of the sediment distribution at certain times (Hardy 2006).

3. Separation Efficiency

The effect of different centrifugal numbers on the separation efficiency is shown for the yeast in Figure 2. The diagram shows the product loss versus the process time at a feed rate of 1 l/min and a solids volume fraction c_V of 0.5 %. The product loss at 2400 G and 5500 G increases from the beginning of the process. The residence time decreases due to the increasing fill level of the centrifuge. The separation efficiency is not sufficient to capture all cells in the centrifuge during the semibatch. At 9700 G and 40000 G the product loss remains almost constant for the entire duration of the batch. The excess of the rotational speed n compensates the reduced residence time. After the capacity of the cylinder is reached, the process is instable. Not only the feed suspension runs through without being purified also parts of the sediment can get dragged with the flow. Thus a product loss higher than 100% occurs. The capacity of the cylinder is reached after 9 minutes at 9700 G, whereas the process at 40000 G lasts 12 minutes. Thus the solids capacity of the centrifuge is increased due to a compaction of the sediment. This effect can be observed at lower centrifugal numbers as well. The product loss curve for 5500 G and 9700 G rises at 9 minutes, although the product loss at 5500 G is higher. This means less product is accumulated in the bowl at 5500 G.

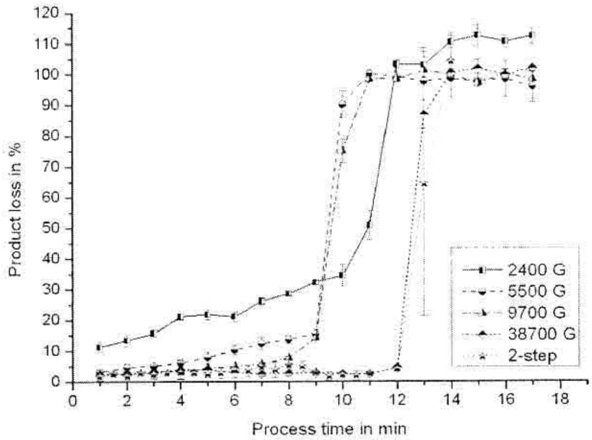

Figure 2: Product loss versus process time for yeast cells at a volume flux of 1 l/min

The curve named “2-step” in Figure 2 shows the result of an optimization. After 8 minutes of centrifugation at 9700 G, the centrifugal number was increased to 38700 G. The compaction of the sediment allows further deposition of cells, resulting in a process similar to the centrifugation at 38700 G during the whole batch. The benefit lies in a considerably reduced mechanical wear of the centrifuge.

Figure 3 shows the product loss versus the accumulated Aerosil 200 nanoparticles in grams inside the bowl. The best results are achieved with the lowest feed flux, which is 0.1 l/min, and the highest centrifugal number, 38700 G. The separation behavior during the batch is similar to the characteristics of the yeast cells at 2400 G and 5500 G. The efficiency decreases constantly due to the reduction of residence time caused by the increasing fill level. The influence of the throughput on the separation efficiency is higher than it is for the centrifugal number. This is unexpected, since in theory, the rotational speed has a higher effect on the cut size. The observed behavior can be explained by structural changes of the soft Aerosil agglomerates. The shear stress at the inlet section at 38700 G is sufficient to break up non-sintered agglomerates. This effect counterbalances the increase of the centrifugal number. Due to the larger agglomerate size the separation efficiency is even higher at 9700 G at low fill levels and little accumulated solids, respectively. The important difference in the process at 9700 G and 38700 G is the specific solids capacity of the centrifuge. Whilst the specific capacity is between 25 g and 30 g at 9700 G, up to 45 g could be captured at high centrifugal numbers. The solids volume fraction of the sediment increases from 6 % (at 9700 G and varying volume fluxes) up to 8.4 % at 38700 G.

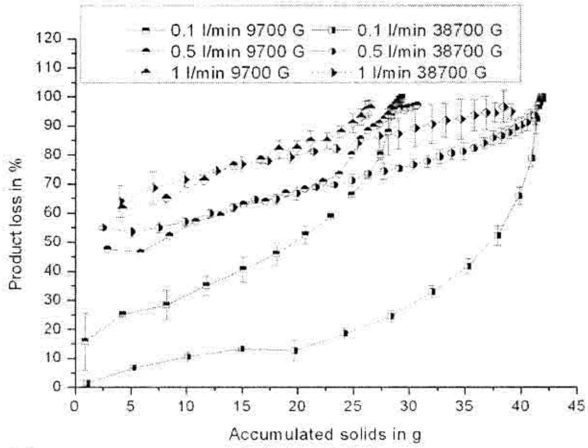

Figure 3: Product loss versus accumulated solids in g

4. Sediment Build up

The decrease of the separation efficiency shown in Figure 2 and 3 is caused by the increasing fill level during the process, which reduces the residence time. The influence of the sediment growth in a semibatch tubular centrifuge on the separation efficiency was first described by Stahl et. al. 2008. But still there are uncertainties about the sediment build-up of submicron particles and biological cells. Figure 4 shows the sediment built of yeast cells. The picture was obtained by magnetic resonance imaging (MRI). The inlet is situated on the left side; the bright areas correspond to carbon black tracer, injected at the inlet after 2 minutes, 5 minutes and 10 minutes of centrifugation. Figure 4 is build up of five single measurements; the last one was the inlet section, the most left. The black dots are caused by the metabolism of the yeast, creating carbon dioxide. The sample was chilled prior to the measurement, but warmed up during the MRI experiment, regaining its capability to produce carbon dioxide during the fifth measurement.

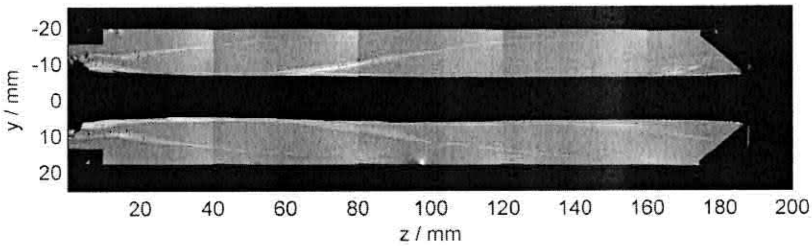

Figure 4: Sediment build-up of yeast cells at a feed rate of 1 l/min and 5500 G

The cells deposit immediately at the inlet section of the centrifuge and accumulate at the outer wall of the bowl. Due to the hindered flow or the reduced separation length along the cylinder, respectively, the cut size of the centrifuge increases during the semibatch. The separation efficiency only decreases, if the cut size exceeds the size of the smallest cells in the feed. The slope of the sedimentation front, in the following called sediment angle, depends on the ratio of the centrifugal force and the mean particle diameter. A steep angle is observed at a high ratio and low throughputs, while a low ratio or a high throughput leads to a small sediment angle. With the assumption of a boundary layer flow (see chapter 5), the effect of the sediment on the residence time is less at low sediment angles. This compensates the high initial cut size at a process with low centrifugal forces slightly.

The fill level, depending on the process time, could be determined by analyzing the MRI-pictures (i.e. Figure 4). The area of the sediment at a certain time related to the area corresponding to the total solids capacity gives the fill level in %. Figure 5 shows the fill level for yeast cells at a constant volume flux of 1l/min and different centrifugal numbers.

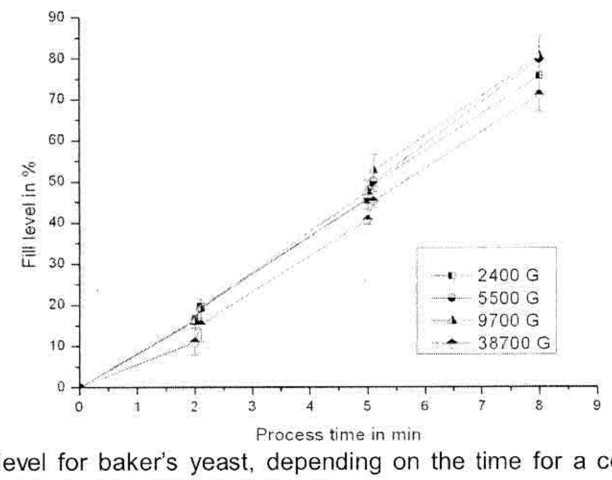

Figure 5: Fill level for baker's yeast, depending on the time for a constant volume flow of 1 l/min and varying centrifugal numbers

Within the accuracy of the measurements the filling behavior at 2400 G, 5500 G and 9700 G does not differ until 2 minutes. With increasing fill levels, the slope of the fill curve at 2400 G declines. This is due to the rising product loss as shown in Figure 2. The difference in the fill curve between the process at 38700 G and smaller centrifugal forces is due to a considerable compaction of the sediment at 38700 G. At lower centrifugal numbers, the product loss rises between 8 and 10 minutes of centrifugation up to 100 %, see Figure 2. This data corresponds to the analysis of the sediment build-up, shown in Figure 5.

5. Flow patterns

The flow patterns inside the rotor are essential for the calculation of the cut size and the effect of the sediment on the separation efficiency. A formation of a boundary layer flow has been described and measured for low rotational speeds (Bass 1959, Glinka 1983). This has been verified in recent studies (Leung 2007). Until now it was not possible to measure the flow patterns in tubular centrifuges at high rotational speeds due to the difficult experimental conditions. A new approach is the comparison of the process outcome between an experiment with a standard rotor and a rotor with structures inside. The structures induce different flow patterns which allow conclusions about the undisturbed flow. Figure 6 shows a scheme of the rotor and the different discs which can be applied at positions 2-6, center of Figure 6. The top scheme in Figure 6 displays a sectional drawing of the rotor of the centrifuge. All dimensions are given in mm. The inlet is at the left side and the fluid gets discharged via three tangential outlets at the right side. The rotor is mounted vertically in the machine.

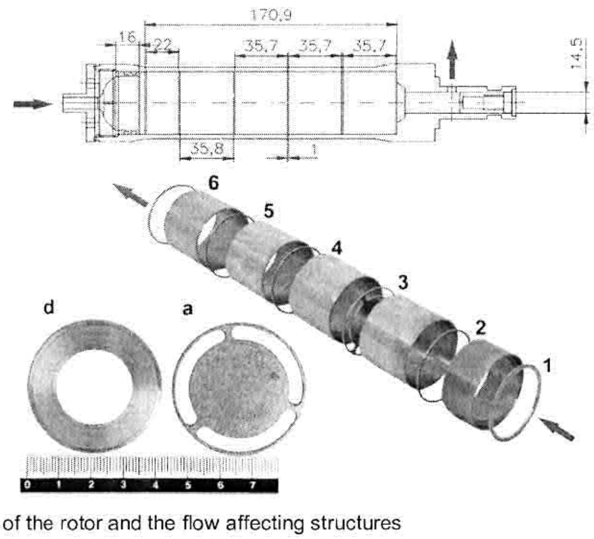

Figure 6: Scheme of the rotor and the flow affecting structures

The two discs, shown at the lower left corner of Figure 6, divert the flow either on a small radius close to the pool surface or at a large radius. The flow patterns, created with discs "d" are equivalent to a boundary layer flow. The residence time, compared to the residence time obtained with the assumption of an ideal plug flow, is less and so should be the separation efficiency. Disc "a" enhances the separation efficiency, since the settling distance for a particle is reasonably reduced and the centrifugal force is higher at an outer radius. Figure 7 shows the product loss during the process for the unhindered flow and the flow through disc "a" and "d", respectively. The product loss rises steadily throughout the duration of the batch from 55% to 100%. Disc "d" has no considerable effect on the separation behavior of the centrifuge. This supports the boundary layer theory. The layer width must be equal or smaller than the distance between the opening radius of disc "d" and the overflow weir. Disc "a" enhances the percentage of captured particles significantly. The deposition of particles behind the opening of disc "a" causes a sediment build-up, which forces the suspension to flow to an inner radius again. Therefore the product loss rises after a few minutes of centrifugation. Nevertheless, this simple geometry modification could be of interest for the separation of fine, dilute suspensions, because it may be applicable to existing centrifuges and classification tasks.

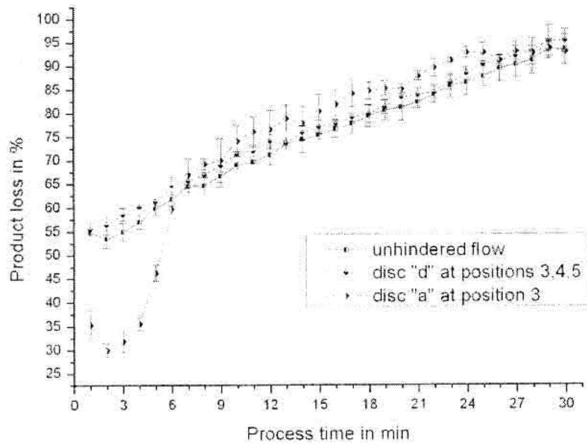

Figure 7: Product loss for Aerosil 200 at 38700 G and a volume flux of 0.5 l/min

6. Conclusions

Tubular bowl centrifuges offer a high potential for solid-liquid separation. Further developments could open up new applications, as separation and classification of nanoparticles and biological products. It was observed that yeast cells are not disrupted in the acceleration zone of a semi continuous high speed centrifuge. The cells formed a sediment similar to a sediment build up of minerals [2]. The separation efficiency could be determined via the product loss of the centrifuge, which could be reduced by higher centrifugal numbers and lower throughputs.

Depending on the properties of the suspension a change of the process parameters affects the separation efficiency in different manner. Higher rotational speeds could cause a further dispersing of agglomerates, so that the benefit of the increased centrifugal force is compensated by the generation of finer particles or agglomerates. This effect was observed during the centrifugation experiments with the partially sintered Aerosil 200 nanoparticles.

Higher centrifugal numbers cause a compaction of the sediment, leading to an increase of the total solids capacity of the centrifuge. This was observed for the Aerosil 200 sediment as well as for the yeast sediment. The ratio of process time to setup time was improved. During the centrifugation of yeast cells, a stepwise increase of the rotational speed has the same effect as running the entire process at a high rotational speed. By this method the mechanical wear is reduced.

The separation efficiency between a process without any flow affecting structures and with discs, similar to washers, was compared. Hence it was possible to confirm a boundary layer flow by applying these structures inside the centrifuge's rotor. A different disc, which forces the flow through openings near the wall of the bowl, increased the separation efficiency considerably. This setup might be applicable to existing centrifuges and may be used for classification purposes.

7. References

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