PROCESS OPTIMIZATION IN TUBULAR BOWL CENTRIFUGES

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Abstract: The demand for instruments which are suitable for the separation of nanoparticles and biological cells increases steadily. Various innovative particles already found their implementation in bulk products and many others are on the leap to marketability. Tubular bowl centrifuges offer high centrifugal forces at reasonable throughputs. There is a high potential for the optimization of existing processes and in the design of new tubular centrifuges. Various experiments, carried out for this work, show the separation of nanoparticles and biological cells in a semibatch tubular bowl centrifuge at high rotational speeds. The influence of the sediment on the process outcome and possibilities to enhance the separation efficiency were investigated. A boundary layer flow was indirectly detected. Structures inside the centrifuge's rotor cause defined liquid flow patterns, which influence the separation efficiency. A comparison between the separation with an undisturbed flow and the modified field of flow gave conclusions about the actual flow patterns.

Keywords: Tubular bowl centrifuges, Optimization, Boundary layer flow, Sedimentation, Bioseparation, Nanoparticles, Centrifugation, Separation

1. INTRODUCTION

There is a consistent increase in the demand for highly efficient solid-liquid separation processes during the last years. The origin is the marketability of various nanoscale products in the pharmaceutical and paint industry. Many agents are produced in biotechnological processes nowadays. The crucial task for these products is the effective purification and separation of specific components. New methods have been developed and the market for functionalized ultrafine composite particles will grow in the near future (Stolarski, et al. 2007). 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 is small. Centrifuges offer high forces and throughputs. Hence these machines are often the first choice if particles like the ones mentioned before need to be captured. Additionally a hygienic process can be realized with different types of centrifuges. The highest centrifugal forces are achieved with tubular bowl centrifuges. The solid bowl and ratio of length to diameter of approximately 5 allow high rotational speeds at a reasonable residence time and throughput, sufficient for many valuable products. An important procedure is the complete removal of the product. The flowability of the product often impedes the removal and remaining product could cause a contamination of the following batch. An optimized process can influence the properties of the sediment considerably and ease the removal of even small biological cells. Furthermore the process efficiency during a semibatch and the solids capacity of the rotor is increased due to a compaction of the sediment.

2. PROCESS OPTIMIZATION

The process efficiency could be evaluated by the measurement of the product loss (equation 1) which is the ratio of the solids volume concentration of the centrate at a certain time, $c_{V,Centrate}(t)$ and the solids volume concentration of the feed, $c_{V,Feed}$. The effect of different rotational speeds, representing different centrifugal numbers, G (equation 2), is shown for a biological product in figure 1. 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 decreases from the beginning of the process. The separation efficiency is not sufficient to capture all cells in the centrifuge. At 9700 G and 40000 G it remains almost constant for the entire duration of the batch. The excess of the rotational speed n compensates the reduced free cylinder length $L_{Cylinder}$, caused by the accumulation of product. Therefore the overall efficiency η is sufficient during the batch. This relation is shown in equation 3. The capacity of the cylinder is reached after 8 minutes at 9700 G, whereas the process at 40000 G lasts 12 min. After the capacity of the cylinder is reached, the process is instable and product gets dragged with the flow. Thus a product loss higher than 100% is possible.

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

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

$$\eta \approx \sqrt{\frac{1}{L_{Cylinder}}} \cdot \frac{1}{n} \qquad (3)$$

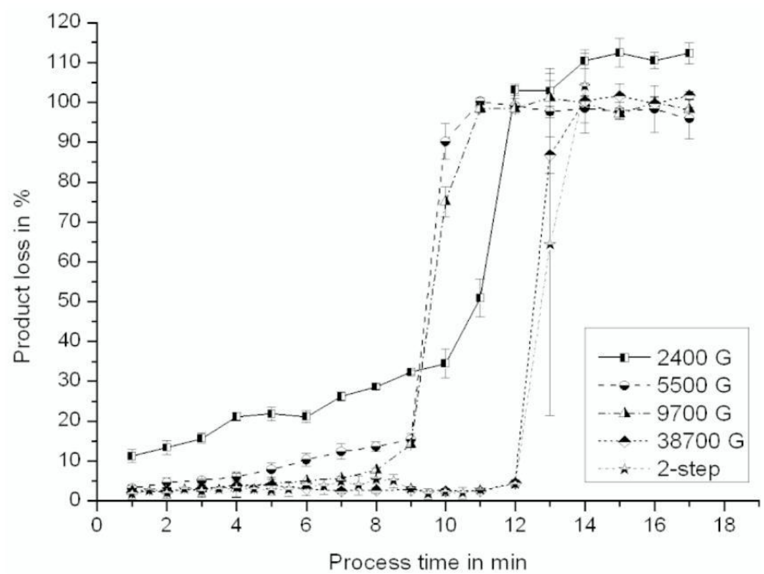

Figure 1: Process efficiency versus process time for yeast cells at a volume flux of 1 l/min

The curve “2-step” in figure 1 shows the result of further optimization. After 8 minutes of centrifugation at 9700 G, the rotational speed was increased up to 38700 G. The compression of the sediment allows further deposition of cells, resulting in a process similar to the centrifugation at 38700 G during the whole batch.

Figure 2 shows the product loss versus the processed suspension consisting of Aerosil 200 nanoparticles and deionized water. The best results are achieved with the lowest feed flux and the highest rotational speed. The separation behavior during the batch is similar to the characteristics of the yeast cells at 2400 G and 5500G. The efficiency decreases constantly due to the reduction of residence time caused by the increasing fill level. At 5500 G and a feed rate of 1 l/min the separation efficiency is the lowest. After 10 minutes of centrifugation, the remaining free length of the cylinder is not sufficient to enable further sedimentation. A complete separation of the nanoparticles is not possible within the range of the centrifuge’s operating parameters. The compressibility of the Aerosil sediment is significantly lower than the sediment build of yeast cells. Hence the capacity is barely increased with a higher centrifugal force.

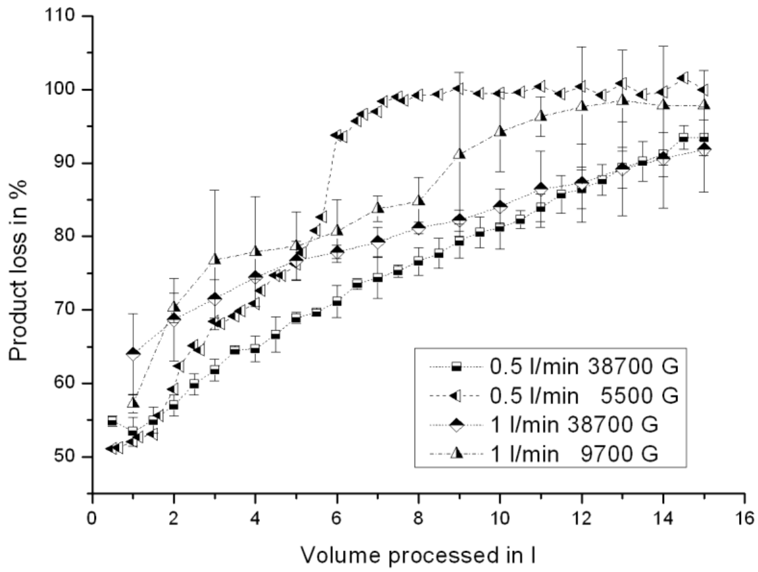

Figure 2: Product loss versus the processed suspension volume for Aerosil 200 nanoparticles

3. SEDIMENT BUILD-UP

The decrease of the separation efficiency shown in figures 1 and 2 is caused by the increasing fill level during the process. The remaining free rotor-length is reduced and so is the residence time. The effect of the reduced residence time on the separation efficiency was first described by (Stahl, et al. 2008). There were still uncertainties about the sediment build-up of submicron particles and biological cells. Figure 3 shows the sediment built of Aerosil 200 nanoparticles. The inlet is situated on the left side, the black areas are corresponding to carbon black tracer, induced at the inlet after 2 minutes, 5 minutes, 8 minutes and 15 minutes of centrifugation. The inlet geometry is not shown in the picture.

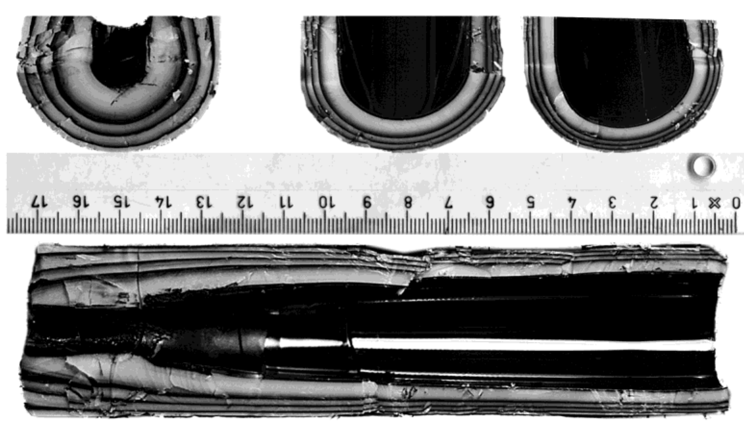

Figure 3: Sediment build-up of Aerosil 200 at a feed rate of 0.5 l/min and 38700 G

The nanoscale particles with an average size of 360 nm (volume) form a similar sediment as it was observed for titanium dioxide which has a broad particle size distribution. The accumulation of the sediment looks similar, but the characteristics are different. The Aerosil 200 particles form a sediment with a porosity of up to 90%, showing a distinctive yield point. Therefore it was possible to take photos of the induced tracer.

The yeast cells show a similar sediment build-up, see figure 4. The cells accumulate at the inlet section (left hand side in figure 4) of the centrifuge and stay at the area of deposition. Due to the hindered flow conditions, the separation efficiency decreases during the period of the batch as shown in figure 1.

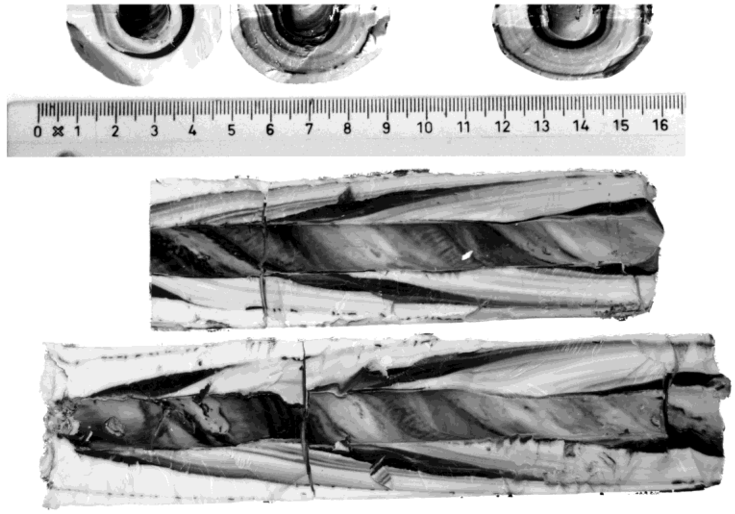

Figure 4: Sediment build-up of yeast cells at a feed rate of 1 l/min and 38700 G

4. BOUNDARY LAYER FLOW

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 (Gösele 1968) and 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 5 shows a scheme of the rotor and the different discs which can be applied at positions 2-6, center of figure 5. The top scheme in figure 5 displays a sectional drawing of the centrifuges rotor. All dimensions are given in mm. The inlet is at the left side, the fluid gets discharged via three tangential outlets at the right side. The rotor is mounted vertically in the machine.

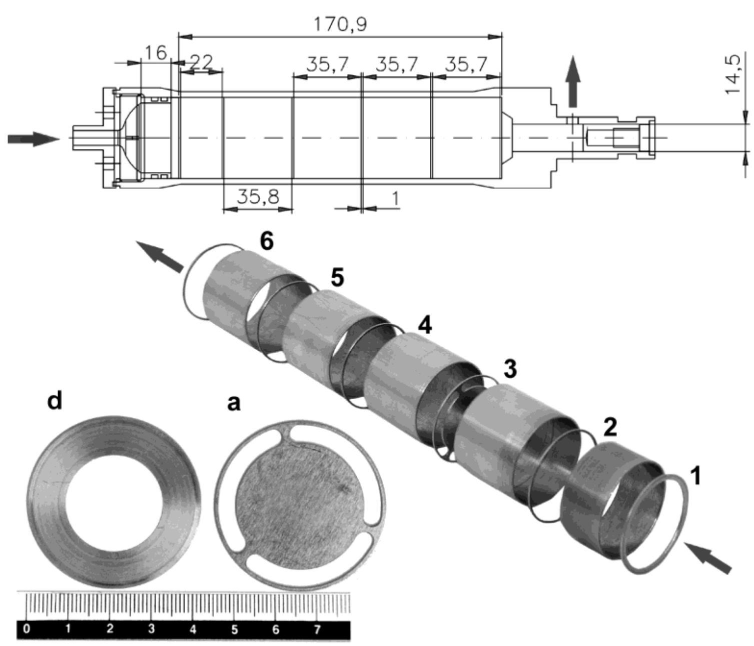

Figure 5: Scheme of the rotor and the flow affecting structures

The two discs, shown at the lower left corner of figure 5, force the flow either on a small radius close to the pool surface or at a large radius, where the centrifugal force is at maximum. If the boundary layer theory is applicable to tubular bowl centrifuges, there will be no considerable difference in the separation behavior of the centrifuge without any discs or the process with disc “d”. The separation efficiency should be enhanced by forcing the flow through disc “a”, because the centrifugal force, affecting the particles, is at maximum. Furthermore the settling distance between the cylinder wall and the flow path is smaller in comparison to the unhindered or ideal plug flow. Figure 6 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” forces the suspension to flow above it. Therefore the product loss rises after a few minutes of centrifugation. The redirection of the flow through disc “a” has no long lasting effect, but the efficiency increase of nearly 100% shows a high optimization potential. This simple geometry modification could be of interest for the separation of fine, dilute suspensions, because it may be applicable to existing centrifuges.

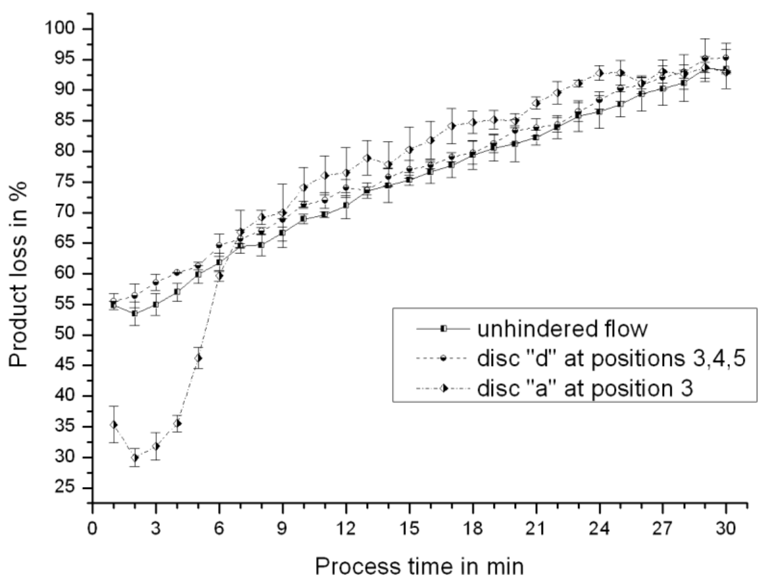

Figure 6: Product loss for Aerosil 200 at 38700 G and a volume flux of 0.5 l/min

5. CONCLUSIONS

The process outcome could be optimized by a variation of the rotational speed of the centrifuge, because the flow conditions change during the batch. The dimensioning of a centrifuge based on the separation efficiency at the beginning of the batch causes a product loss during the operation in some cases. The separation performance at the end of the batch is crucial for a satisfactorily process outcome during the entire duration of the process.

The yeast cells do not build a uniformly distributed sediment. Instead the sediment build-up of the biological product is similar to the sediment build-up of minerals. Thus the sediment affects the flow patterns and reduces the free length of the centrifuge’s rotor. Hence, the residence time and so the separation efficiency is reduced. Higher centrifugal forces could enhance the separation and the solids capacity of the rotor. Therefore it could be economic to run a process at higher rotational speeds than initially determined. A step-wise or continuous increase of the rotational speed reduces the mechanical wear at no loss of efficiency. Aerosil 200 silica nanoparticles form a stable, nearly incompressible sediment with a porosity >90%. The sediment build-up during the process is similar to the sediment build-up observed with micron scaled minerals.

A boundary layer flow was detected via applied structures inside the centrifuge’s rotor. The flow was forced to a flow path close to the pool surface. There was no significant deviation of the process outcome between the centrifugation with an undisturbed flow and the process with the discs inside. A redirection of the flow close to the outer radius of the rotor enhances the separation efficiency significantly.

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