Centrifugal Classification In The Submicron And Nanometer Range

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Abstract. Centrifuges represent a commonly used tool for classification of particles in the liquid phase. Their main advantages are scalability and the use of both fine and coarse fraction. The access to the submicron and nanometer range requires considerably high centrifugal accelerations, however. Therefore, novel bearing and drive concepts were used to develop a tubular bowl centrifuge, which allows a relative centrifugal force of more than 100,000 for separation and classification purposes in a semi-continuous process. This paper illustrates the classification results of silica particles in the nanometer range. Moreover a comparison regarding the separation efficiency is made with a commercially available device.

Keywords: Solid-liquid separation, colloidal particles, high-speed centrifuges

INTRODUCTION

Centrifuges are applied for classification tasks in the liquid surrounding phase for some time. Larger apparatuses with a continuous process concept like decanters [1] or disc stack separators [2] are appropriate for particle systems up to the micrometer range. Besides, these centrifuges can handle large product streams. For smaller particle systems centrifugal accelerations are necessary, which are only provided by tubular bowl centrifuges. Within the last years, some experimental studies dealt with this topic [3,4]. However, the semi-continuous process concept brings disadvantages which cannot be neglected: a sediment buildup takes place in the rotor, leading to a successive degradation of separation performance and requiring a termination of the process as well as a particle discharge. The first aspect can at least be fully compensated by means of process monitoring and control techniques [5]. For a further shift of cut size to even smaller particles, constructional improvements and changes, respectively, are necessary. This is demonstrated by the presented work.

FUNDAMENTALS

As the particle size, which may be separated in a centrifuge, correlates with the inverse centrifugal acceleration, the relative centrifugal force

$$C = \frac{\omega^2 \cdot r}{g} \tag{1}$$

is a very important process parameter. ω is the angular velocity, r is the radius, and g is the gravity of Earth. The grade efficiency as a function of particle size

$$T(x) = \left(1 - f \cdot \frac{q_{Overflow}(x)}{q_{Feed}(x)}\right) \cdot 100\% \tag{2}$$

is usually used to describe the result of a classification experiment. f is the ratio of the particle concentrations in the overflow to the feed while $q(x)$ is the density distribution function of the respective particle collective. A further evaluation of the grade efficiency curve is made by the calculation of classification sharpness

$$\kappa = \frac{x_{T=25\%}}{x_{T=75\%}} \tag{3}$$

where κ is the particle size with a related separation probability. Since different centrifuges vary in rotor volume and operational speed, it is necessary to introduce a quantity for comparison. Therefore, the separation efficiency is considered for different apparatuses as a function of the ratio of flow rate to Sigma value (the ratio may be interpreted as an inverse "process effort") with

$$\Sigma = \frac{V \cdot C}{s}. \tag{4}$$

V is the volume of the liquid in the rotor and s is the sedimentation length.

METHODS AND MATERIALS

The developed centrifuge prototype uses a magnetic bearing and a magnetic drive. The stator contains switchable electromagnets while permanent magnets are mounted on the rotor. To ensure a durable connection between the rotor material and the permanent magnets, their positions are reinforced by carbon fibre composite material. Friction losses between rotor and the surrounding gas phase are reduced by the use of a helium atmosphere. The remaining thermal energy is led away by water cooling. The rotor volume is adjustable to a certain extent by means of different overflow weir sizes. The weir used in these experiments results in a volume of about 0.26 l. The highest speed which has been tested so far is 68,300 min^{-1}. This refers to C = 120,000 at the inner rotor wall.

For comparison with a commercially available centrifuge, a GLE manufactured by Carl Padberg Zentrifugenbau GmbH, Germany, was used. The maximum speed is 40,000 min^{-1} (C = 38,500).

Aerosil 200, a fumed silica made by Evonik Industries AG, Germany, was used as experimental product. The density is 2200 kg/m³ and the mean particle size is $x_{3,50}$ = 80 nm. A solids volume concentration of 0.005 was used to ensure a size dependent sedimentation. For calculation of *f* and *T(x)* samples were taken from the feed and the overflow weir.

EXPERIMENTAL RESULTS

FIGURE 1 shows the grade efficiency curves at a relative centrifugal force of C = 80,000 and varying flow rate. It becomes clear that the chosen conditions enable classification in the nanometer range: at the highest flow rate, a 50% separation probability (cut size) is obtained for about 55 nm. According to the fundamentals, a decrease in flow rate leads to a shift to a smaller cut size. A reduction of flow rate by half to 0.2 l/min results in a cut size of 35 nm. At 0.1 l/min almost the entire particle collective is separated with an efficiency above 75%. With the chosen product and under the conditions of FIGURE 1, no evaluation according to Eq. (3) is possible because grade efficiency is permanently above 25%.

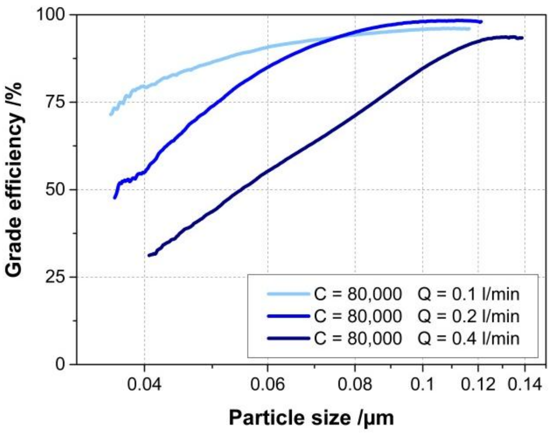

FIGURE 1. Grade efficiency curves under variation of flow rate at a constant C value of 80,000. Experimental product: Aerosil 200 in deionized water (0.5 Vol.-%).

For this purpose the relative centrifugal force needs to be lower. FIGURE 2 shows the classification sharpness under variation of flow rate. At 0.1 l/min sharpness lies within the 'sharp separation' range for all considered C values. With an increase in flow rate the sharpness drops below κ = 0.6, leading to a poorer quality of the classification experiment. Furthermore, it becomes clear that the sharpness values for experiments with C = 20,000 are lower than for processing with C = 40,000. Both observations – a decrease of sharpness with an increase in flow rate and a decrease in C value – have recently been traced back to the flow conditions in the rotor [6]. A low C value and a high flow rate promote a more intense momentum exchange in radial and axial direction. Residence time in the rotor is therefore increased as well. This is an undesired state for classification.

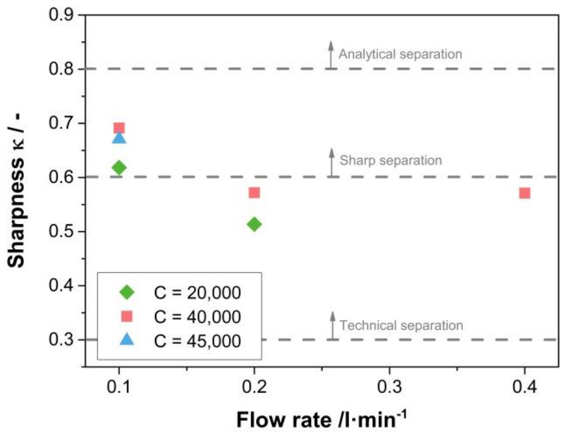

FIGURE 2. Classification sharpness as a function of flow rate. Experimental product: Aerosil 200 in deionized water (0.5 Vol.-%). The dashed lines indicate certain ranges of classification quality.

In FIGURE 3 a comparison is made between the developed prototype (blue data) and a tubular bowl centrifuge made by Carl Padberg Zentrifugenbau GmbH, Germany (red data). Separation efficiency is given as a function of the ratio of flow rate to Sigma value. Application of a higher process effort, i.e. lower flow rate and higher C value, leads to a decrease of the x-axis value. The diagram shows that for both centrifuges a lower flow rate to Sigma ratio results in better separation efficiencies. However, the commercial device works slightly more efficient, since higher separation efficiency is reached for the same process effort. A possible explanation for this observation might be a better acceleration of the suspension in the inlet region of the rotor. This is a point which can easily be modified for the prototype and will be subject of further investigations. Moreover, it must be mentioned that the data points of the prototype with a separation efficiency ≥ 80% were collected at C = 80,000, which is not the highest possible C value, whereas the respective data of the CEPA GLE result from experiments at the machine's upper limit.

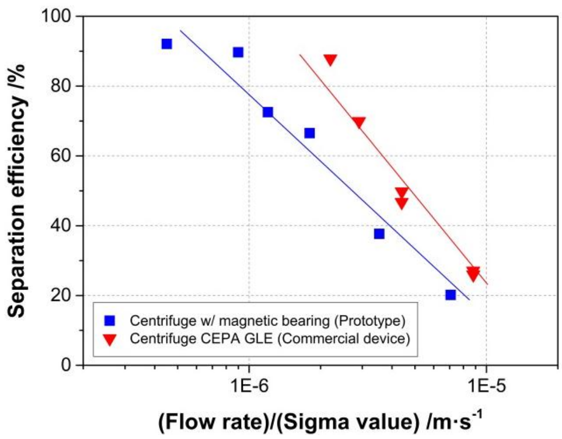

FIGURE 3. Separation efficiency vs. flow rate divided by Sigma value for comparison of the developed prototype with a commercial device. Experimental product: Aerosil 200 in deionized water (0.5 Vol.-%).

CONCLUSION & OUTLOOK

Due to the use of novel techniques and materials it was possible to develop a tubular bowl centrifuge whose speed goes beyond the state of the art of centrifuges which are available up to now. The semi-continuous process design allows separation and classification in the submicron and nanometer range. For low flow rates considerable sharpness values have been obtained. As the flow conditions significantly influence the quality of the classification experiments, further investigations and optimizations need to be made. This concerns CFD simulations to better understand the flow, which affects the particle separation process, as well as changes in rotor geometry for improved suspension acceleration.

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