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SCREENING OF COLLOIDAL PARTICLES IN CENTRIFUGES

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ABSTRACT

The screening of fine particles is an increasingly demanding task in the solid-liquid separation community. Many products are on the leap to marketability, but an undefined particle size distribution may impede the implementation of these products in the process scale. Colloidal particles may be used in ultra thin coatings, for high precision polishing and for medical purposes.
This work shows the screening of mixtures of monodisperse latices and polydisperse polystyrene. This demonstrates the feasibility of sub-micron particle screening in centrifuges. Furthermore the flow patterns are measured in a laboratory centrifuge. With the gained knowledge, directions are made for a further development of centrifuge design.

KEYWORDS

Classification, Tubular Bowl Centrifuges, Particle sizing, Fluid Mechanics

1. Introduction

The synthesis of particles is just the first step to the final product, followed by stabilization, formation and quality management. The stabilization of particles is important to assure consistent product quality during storage and usage [1]. The measurement of the particle size distribution of the initial and final product is a difficult but important task for a polydisperse particle system with a mean diameter below 1μm. The most reliable and reproducible results for the particle size distribution are obtained by small angle X-Ray scattering and analytical centrifugation. The first step towards the final product, the particle synthesis, may impede the formation of the desired product properties, especially in high throughput synthesis. The most common high throughput production techniques are flame pyrolysis and milling [2, 3]. Whereas the particles form branched aggregates during flame synthesis, compact particles are produced by milling in stirred media mills. Both products will have a broad particle size distribution, which may impede further usage as e.g. in polishing media, in ultra thin coatings and medical appliances. Hence the screening of the material after synthesis may be required. For colloidal particle systems, this unit operation as only been successfully conducted in the laboratory scale [4]. This paper describes the screening in semi-continuous centrifuges and gives some directions for the necessary developments for the industrial implementation.
The prediction of the necessary process parameters as rotational speed, throughput and solids volume fraction in the feed requires the comprehensive understanding of the flow patterns and settling behavior of submicron particles in the centrifugal field. The separation behavior and the exact value of the cut size have been determined with polydisperse products and monodisperse latices. The flow patterns in a rotor,

made of carbon fiber reinforced plastic, of a centrifuge prototype have been measured with Laser-Doppler-Anemometry. The results will support the development of an improved feeding system.

2. Methods and Materials

The determination of the screening efficiency and the cut size was conducted on a laboratory tubular centrifuge model CEPA GLE with a maximum rotational speed n of 40000 rpm and throughputs ranging from 0.1 to 1 l/min. The necessary process parameters for the semi-continuous screening were calculated using Stokes law [5].

The flow patterns were measured in an especially designed and self-made centrifuge. This centrifuge can be operated up to 17000 rpm with throughputs ranging between 0.1 and 2.5 l/min. The processor of the used Laser-Doppler-Anemometer limits the measurement of velocities up to 45 m/s, which equals a rotational speed of 9000 rpm.
It is known that a short circuit flow, called boundary layer flow, is developed in most overflow centrifuges [6, 7]. The short circuit causes a small residence time, which is disadvantageous for the separation efficiency. In centrifuges with an internal core, a plug flow profile may be observed. In the presented study the flow patterns were measured for a system with an internal core.

2.1 Particles and stabilization of the suspension

The separation behavior of different fine particle systems has been the focus of previous research activities. The next step is the screening of these particles and the exact determination of the cut size of the centrifuge. The analysis of the separation and classification behavior requires a constant particle size distribution in the feed for all experiments. Stabilization could either be made by influencing the zeta potential or by adding dispersants. Both methods could lead to a constant particle size distribution, but may affect the following processes in a different manner.

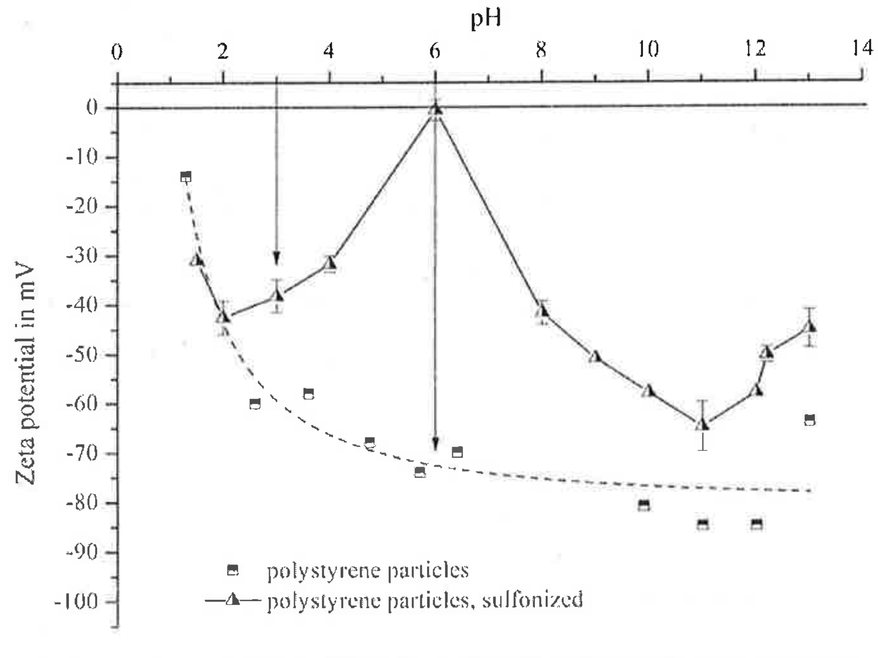

Figure 1: Zeta potential and solids volume fraction depending on pH-Value

The stabilization via the zeta potential often requires a shift of the pH-value, whereas a added dispersant could be an issue in subsequent usage of the particles. In this work, the product is stabilized via the zeta potential; the measurements of the potential are shown in figure 1. The zeta potential of the polystyrene particles is between -80 and 0 mV. The non sulfonized particles are sufficiently stabilized by the negative charge of the particles at a neutral pH, whereas the sulfonized particles are not stabilized at pH 6. Thus all experiments with the polydisperse, sulfonized particles were conducted at a pH-value of 3.

The exact value of the cut size could be determined with spherical, monodisperse particles due to Stokes's law, which describes the settling of a sphere. The calculated settling velocity will be in good agreement with the real velocity, if the solids volume fraction is very small and the settling velocity leads to a particle Reynolds number below 1. The latex particles exhibit a density difference of only 50 kg/m³, so the settling velocity is small, leading to particle Reynolds numbers between $2.8*10^{-4}$ and $1.1*10^{-3}$. The latex particles are injected with a syringe into a tube with a subsequent dispersion via a sonifier. The volume fraction of the particles in the syringe is below a solids volume fraction of 0.37 % and is diluted due to the injection into the constant feed flux of e.g. 0.1 l/min. Thus the concentration is sufficiently low to apply Stokes's equation. The particle size distribution of the overflow is then measured with a CPS disc centrifuge. The cut size was determined with mixtures of different sizes of monodisperse latices.

2.2 Laser-Doppler-Anemometry

Laser-Doppler-Anemometry is a non-invasive measurement technique which can be used for the determination of the flow velocity in gaseous and liquid streams. The advantage of the technique is the avoidance of any disturbances of the flow, because no probes have to be inserted in the flow. A scheme of the experimental setup is shown in figure 2. The laser probe is located below the centrifuge, the laser beams enter the centrifuge via a self-made optical system, consisting of two achromatic lenses and two mirrors.

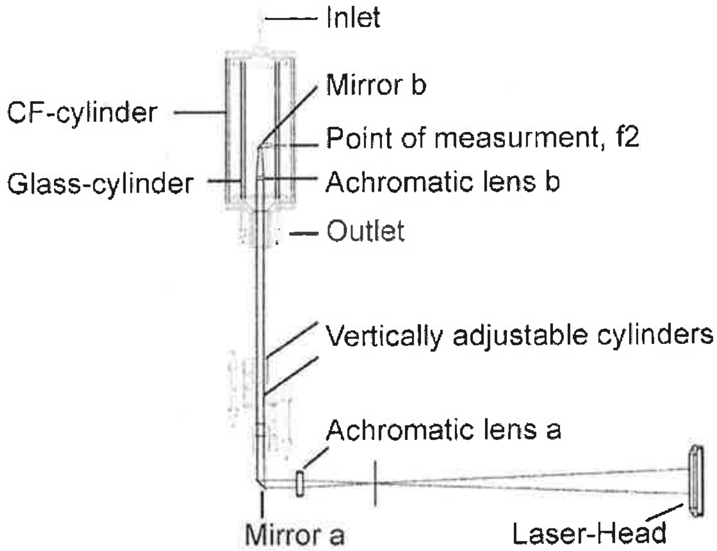

Figure 2: Experimental setup for the velocity measurement

The laser beams enter the rotor of the centrifuge via a glass cylinder. The glass cylinder rotates with the same revolutions per minute as the outer rotor does. This setup is further referred to as tandem setup. The outer radius r_i of the rotor made of glass equals 30 mm; the inner radius of the carbon fiber rotor is 50 mm. The measurement without the glass cylinder represents the process condition in overflow centrifuges, whereas the tandem setup is used in high speed centrifuges for e.g. the production of vaccines [8]. The void between both rotors is entirely filled with water. The velocity span of the used system is between 0.01 and 45 m/s. The system is a two dimensional Laser-Doppler-Anemometer from *Dantec Dynamics GmbH* with a signal processor model *F30*. The system is powered by a 6 W He-Ar laser supplied by *Spectra physics*. The experimental setup allows the measurement of the axial and tangential velocity profiles. The experimental results are compared with the calculated rigid body rotation v_{rot}, which is determined with

$$v_{rot} = \frac{2 \cdot \pi \cdot n}{60} \cdot r \,. \qquad [1]$$

The necessary power output varies due to the measurement position; if the position is close to the surface of the media, 0.5 W may be sufficient. However, if the distance of the laser beams through the media increases, higher power outputs will be necessary.

3. Screening of polydisperse particles

The screening efficiency of the tubular bowl centrifuge was determined with sulfonized, polydisperse polystyrene particles, which were synthesized via a mini-emulsion polymerization at the MVM. The dispersed particles were injected in the feed of the centrifuge and samples were taken from the feed and the overflow. The differential particle size distributions (number basis) are shown in figure 3.

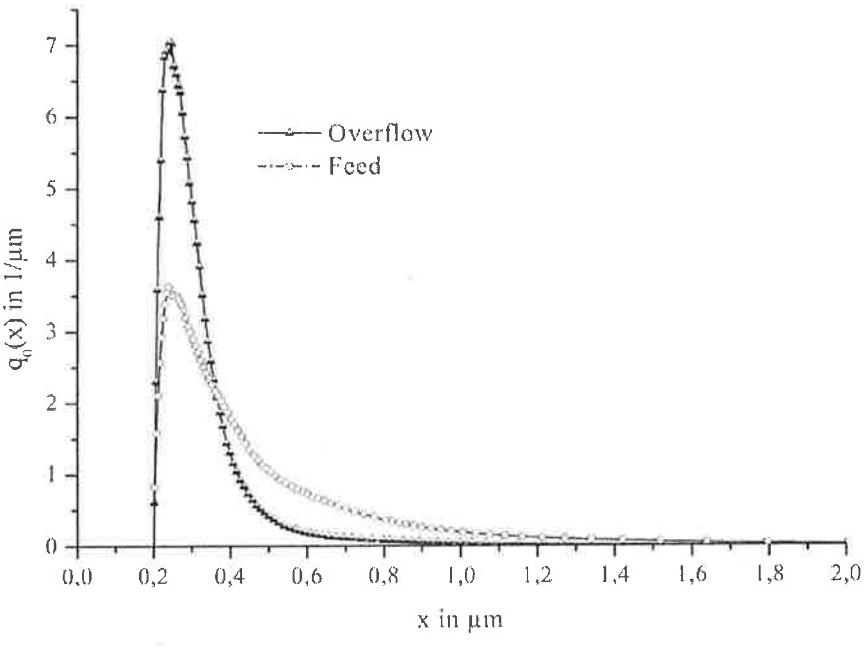

Figure 3: Differential particle size distribution (number based) of the feed and the overflow after centrifugation at 40000 rpm and 0.1 l/min

Most particles with a diameter above 700 nm are captured in the centrifuge. The calculated cut size for the given operating parameters will equal 650 nm, if a boundary layer flow with a width of 2 mm is assumed. The overflow exhibits a narrow particle size distribution.

4. Determination of the cut size

Figure 4 shows the particle size distribution of a mixture containing different fractions of monodisperse latices. After the experiment at 40000 rpm and a throughput of 0.1 l/min, all particles coarser than 720 nm are separated. Only particles below 720 nm are detected in the overflow. These results verify that the separation of two fractions with a size difference of 25 % is possible in the tubular centrifuge. The calculated cut size matches the experimentally determined one, which shows that the settling velocity of latex spheres in a semi continuous tubular centrifuge can be calculated using Stokes law. A rotational speed of 40000 rpm was used for the calculations. This presumes an ideal tangential acceleration of the liquid in the inlet zone, because a lag of the liquid of only 10 % would increase the cut size to 740 nm. Further investigations will show if the cut size for higher throughputs and different rotational speed is predictable by Stokes's law for this kind of machinery.

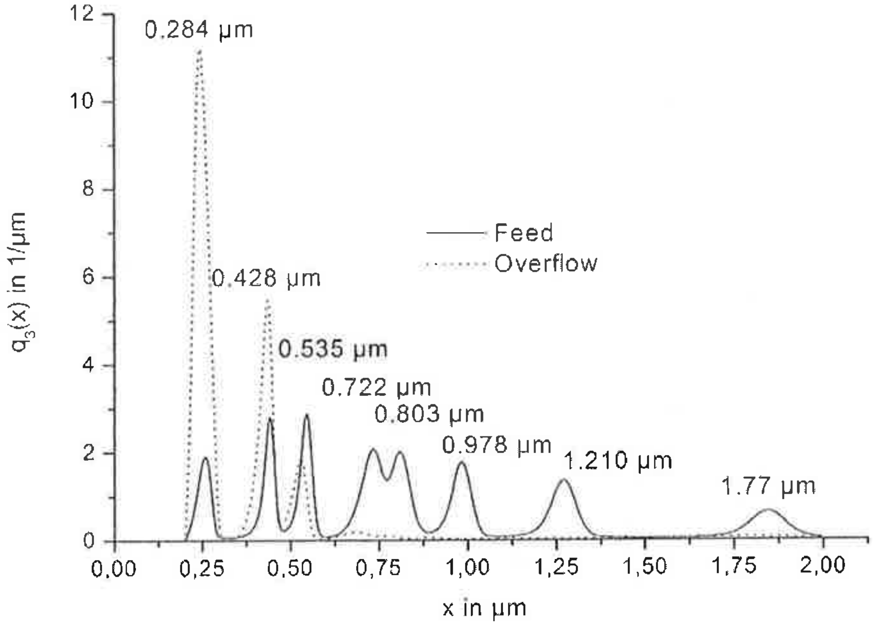

Figure 4: Differential particle size distributions (mass based) of a mixture of polystyrene particles with different sizes before and after screening at 40000 rpm and 0.1 l/min

5. Analysis of the flow patterns

The measurement of the flow patterns by Laser-Doppler-Anemometry allows the determination of the tangential and axial fluid velocity in the rotor for rotational speeds up to 9000 rpm. Figure 5 shows the tangential velocity profiles half way between inlet and outlet.

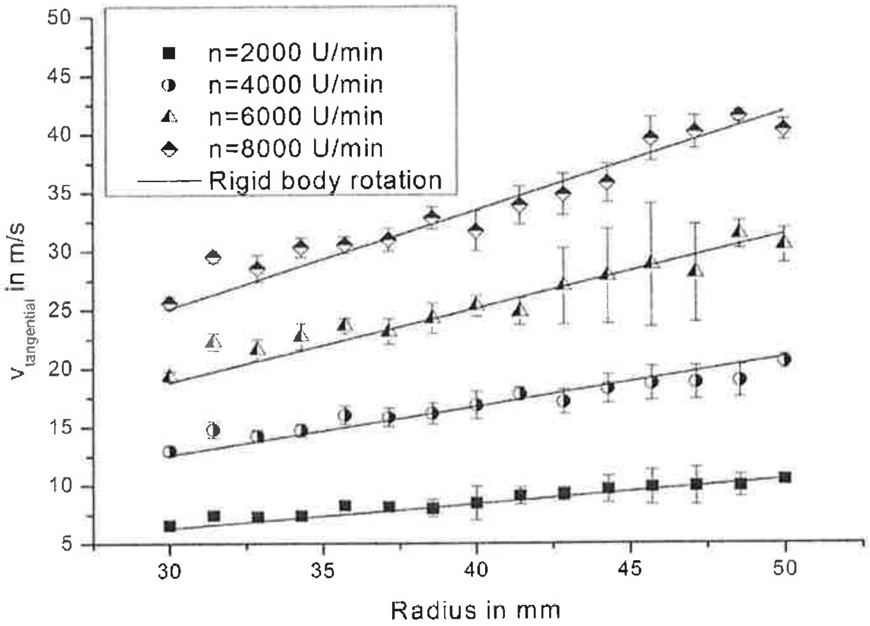

Figure 5: Tangential velocity profiles at a volume flux of 1.2 l/min

There is no significant deviation of the tangential velocity compared to the rigid body rotation within the accuracy of the measurements. The inlet via two pipes, which rotate with the same rotational speed as the rotor does, assures an ideal tangential acceleration of the feed. Due to the linear coupling of the sedimentation velocity to tangential velocity, the acceleration efficiency is very important for the process.
The second important parameter is the distribution of the flow over the desired cross section, since the residence time increases with higher crossectional area. There are only two feeding pipes in the centrifuge, thus a homogeneous distribution of the axial flow is unlikely. The axial flow profiles are shown in Figure 6. Bright areas correspond to areas of high fluid velocity and dark areas represent low fluid velocities. The left picture shows the axial fluid velocity 40 mm behind the inlet. The main liquid flow occurs in two small areas at the angles where the feed pipes are located. With increasing feed flux, the high inlet velocity causes a flow close to the wall of the rotor, as depicted in figure 6, mid. Another difference to the smaller feed flux is the quicker distribution of the feed in angular direction. At 2 l/min and a similar distance to the inlet, the liquid flows through a significant greater area covering 360°.

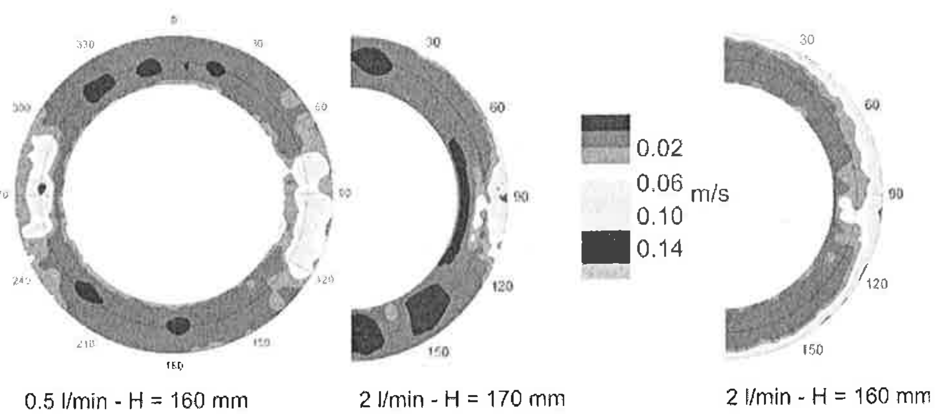

Figure 6: Axial velocity profiles at a rotational speed of 1000 1/min

6. Conclusions

The screening of colloidal particles with high speed centrifuges is feasible. For polystyrene, a cut size of at least 722 nm was determined by the experimental investigation of the separation efficiency of a laboratory, semi continuous tubular bowl centrifuge. The screening of polydisperse polystyrene was successful; the fine fraction exhibits a narrow particle size distribution and no coarse particles occur in the overflow of the centrifuge. The achieved cut size for the polydisperse particles is in agreement with the cut size, determined by the centrifugation of monodisperse fractions. Furthermore the experimentally determined cut size will match the calculated one, if an unhindered settling and a boundary layer flow with a width of 2 mm is assumed.

Laser-Doppler-Anemometry was successfully applied for the first time in a centrifuge. The measurement of the tangential fluid velocity indicates an excellent tangential acceleration of the liquid for high specific throughputs. A higher uncertainness of the gained data was observed for the measurement of the axial fluid velocity. This is due to the low axial velocity compared to the high angular velocity and scattering at the rotating glass, which causes a significant noise. Nevertheless, the measurements pointed out that the expansion of the inlet jet increases with higher feed flux. The feed is not homogeneous distributed, independently of the feed flux.

7. Outlook

The lowest achievable cut size of the laboratory centrifuge is 722 nm for particles with a density difference of only 50 kg/m³; the cut size for particles which exhibit a higher density difference to the surrounding liquid is reasonably lower. This proves the promising prospects of this kind of apparatus for the screening of colloidal particles. Further improvements for the tangential acceleration of the liquid at higher feed rates have to be made. The measurements with the Laser-Doppler-Anemometer confirmed an excellent acceleration with a rotating feed system, but the distribution of the feed has to be enhanced. This may be conducted by e.g. 6 or more feed pipes.

8. References

1. Peukert, W., et al., *Control of particle interfaces - the critical issue in nanoparticle technology.* Advanced Powder Technology, 2003. **14**(4): p. 411-426.
2. Mueller, R., L. Madler, and S.E. Pratsinis, *Nanoparticle synthesis at high production rates by flame spray pyrolysis.* Chemical Engineering Science, 2003. **58**(10): p. 1969-1976.
3. Stenger, F., et al., *Nanomilling in stirred media mills.* Chemical Engineering Science, 2005. **60**(16): p. 4557-4565.
4. Katasonova, O.N. and P.S. Fedotov, *Methods for continuous flow fractionation of microparticles: Outlooks and fields of application.* Journal of Analytical Chemistry, 2009. **64**(3): p. 212-225.
5. Stahl, S., L.E. Spelter, and H. Nirschl, *Investigations on the Separation Efficiency of Tubular Bowl Centrifuges.* Chemical Engineering & Technology, 2008. **31**(11): p. 1577-1583.

6. Glinka, U., *Die Strömung in Überlaufzentrifugen - Neue Ergebnisse mit einem elektrolytischen Markierungsverfahren.* Verfahrenstechnik, 1983. **17**(5): p. 315-318.
7. Spelter, L.E., A. Steiwand, and H. Nirschl, *Processing of dispersions containing fine particles or biological products in tubular bowl centrifuges.* Chemical Engineering Science, 2010. **65**(14): p. 4173-4181.
8. Perardi, T.E., R.A.A. Leffler, and N.G. Anderson, *K-series centrifuges II. Performance of the K-II rotor.* Analytical Biochemistry, 1969. **32**(3): p. 495-511.