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Semicontinuous nanoparticle screening and applied Laser-Doppler-Anemometry in tubular bowl centrifuges

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Abstract: It is possible to control the particle size during production in the laboratory scale, but most bulk products are synthesised in industry scale, where it is difficult to avoid reasonable amounts of particles with an undesired size. This work shows the successful screening of submicron latex particles and fine industrial silica in a semi-continuous tubular bowl centrifuge for different process conditions.
In order to understand and improve the separation of colloidal suspensions in process machinery, it is necessary to determine and predict the flow patterns. A novel self-constructed tubular bowl centrifuge allows the measurement of the flow profile up to 8000 rpm, which corresponds to 3600 times the earth gravitational force. The fluid velocity is measured with laser-doppler-anemometry, in which the laser beams enter the centrifuge via an optic through the base bearing. The axial and tangential flow profiles for different rotational speeds and throughputs are investigated in this work. The rotor of the centrifuge is made of carbon fibre reinforced plastic. This material could increase the achievable centrifugal numbers in the future due to its high tensile strength and low density. The combination of high centrifugal numbers and improved flow channelling will enable the separation and screening of fine particulates in the process scale.

Keywords: centrifugation, colloid, fluid mechanics, separation, suspension.

1 Introduction

Fine particulates including nanoparticles are used in various products nowadays. Examples for recent developments are ultra thin coatings and composite particles for the cancer treatment and the selective bioseparation [1-4]. Ultra fine particles exhibit a high ratio between the surface and the volume, which allows a cost-effective usage of e.g. expensive catalytic materials. Furthermore some nanoparticles offer unique properties such as quantum-dots and invisible sunscreens. In most of these applications, coarse particles interfere with the desired properties of the final product, since e.g. the thickness of a layer is limited by the diameter of the coarse particles.

Whereas it is possible to control the particle size during production in the laboratory scale, it is often difficult to avoid the formation of any coarse particles in the process scale [5]. Hence, the implementation of nanoparticles in bulk products presumes a size classification of the particles. Since the product is very fine, the hydrodynamic resistance of even the coarse fraction will impede the separation in most industrial solid-liquid separation devices. Screen scroll and disc stack centrifuges exhibit a high clarifying potential, but these machines are not sufficient to capture particles below 1µm in diameter. Tubular bowl centrifuges offer the necessary separation efficiency and might be used for the classification of submicron particles. This work shows the successful screening of fine particulates in a semi-batch tubular bowl centrifuge at a throughput of 0.1 l/min and rotational speeds between 20000 and 40000 rpm.

The further development of effective tubular centrifuges requires the understanding of the flow patterns in dependency of the throughput and the rotational speed. The flow patterns are determined by Laser-Doppler-Anemometry for throughputs between 0.5 and 1.8 l/min and rotational speeds up to 8000 rpm. This knowledge aids the design of tubular centrifuges with significantly higher solids capacity, throughput and separation efficiency. The screening of ultra fine particles in the process-scale will be possible with these kind of centrifuges, which fulfils the demand of the industry for particles with a designed size distribution below 1 µm.

2 Method and Materials

2.1 Screening

The screening of dispersed particles bases on the different settling velocity ν of a coarse and a fine fraction. The settling velocity itself depends corresponding to equation (1) on the particle size x, the dynamic viscosity of the liquid μ, the density difference Δρ between the dispersed solids and the liquid, the acceleration due to gravitation g or, as the case may be, the centrifugal force.

$$\nu = \frac{\Delta\rho \cdot g \cdot x^2}{18 \cdot \mu} \tag{1}$$

The centrifugal acceleration depends on the rotational speed n and the radius of the centrifuge r and is often stated as the centrifugal number Z, which is according to equation (2) the ratio between the centrifugal acceleration and the acceleration due to gravity.

$$Z = \frac{\left(\frac{2 \cdot \pi \cdot n}{60}\right)^2 \cdot r}{g} \tag{2}$$

The sedimentation of fine particulates is impeded by the low mass and the large surface of the particles. This challenge can be met by high speed centrifuges. Since the centrifugal number is increased by the square of the rise of the rotational speed but only linearly dependent on the diameter of the rotating bowl, high rotational speeds are preferable.

The cut size of a centrifuge follows by the coupling of the residence time τ, the settling velocity - with the rotational speed as the operational parameter - and the ratio between the liquid-gas interface r_i and the radius of the boundary layer flow r_b. This is described in equation (3).

$$x_t \approx \sqrt{\frac{1}{\tau} \cdot \ln\left(\frac{r_i}{r_b}\right)} \cdot \frac{1}{n} \tag{3}$$

The residence time as well as the radii at which the settling behaviour is determining the process depends on the flow patterns in the centrifuge. A boundary layer flow is developed in solid-bowl centrifuges with a two-phase system of gas and liquid. The liquid flows only in a thin layer with the width r_b-r_i above a stagnant pool [6]. Hence the residence time and the settling distance are determined by the width of the boundary layer flow. Therefore the determination of the flow patterns has been the emphasis of many research projects during the past decades [8, 9]. In this work the flow patterns inside the rotating bowl are revealed by Laser-Doppler-Anemometry.

The separation efficiency of the centrifuge has already been investigated for fine particulates and yeast cells [6, 7]. In this work, the screening of two different products in a semi-batch tubular bowl centrifuge is analysed.

The determination of the screening efficiency of a product which exhibits a small density difference to the surrounding liquid was conducted with polystyrene. The polystyrene particles exhibit a wide particle size distribution; the coarse particles are smaller than 1 µm. The density difference between the liquid and the solid is only 50 kg/m³ and the mean particle diameter by volume is 330 nm. The second product is industrial silica, Aerosil 200, synthesised by flame pyrolysis, with a mean particle diameter by volume of 375 nm and a density difference to the liquid of 1200 kg/m³. The sintered agglomerates contain primary particles with a diameter of 8 nm. The settling behaviour of these agglomerates differs considerably from that of spherical particles.

2.2 Laser-Doppler-Anemometry

Laser-Doppler-Anemometry is a non-invasive measurement technique which allows the velocities in a gas or liquid to be measured in a broad range. The measurement of a velocity with a laser will be possible, if a particle crosses the beam and scatters the laser light. The light is scattered with a shifted frequency, which contains the information of the velocity of the particle. A measurement of the flow patterns in a gas or liquid flow requires particles with a size, similar or slightly higher than the wavelength of the laser. Furthermore the particles must follow the flow lines of the liquid without inertia, so that the velocity vector of the particle equals the one of the liquid. In the presented studies the mean diameter by number of the tracer particles was 600 nm with a density close to the one of the water.

The centrifuge could be operated at circumferential speeds up to 80 m/s. The tensile strength of transparent materials as Plexiglas® is not sufficient to withstand the high tensile stresses due to the centrifugal acceleration. Due to this constrains, carbon reinforced plastic is used for the construction of the rotor. This material exhibits a high tensile strength and a low density, so that the tensile stresses, created by the centrifugal acceleration and the mass of the rotor, are smaller than the tensile stresses of a rotor made of steel or alloy.

The carbon fiber (CF) rotor is not transparent. Hence it was not possible to measure the flow patterns directly. Instead a self-constructed optic is used to modify the laser beams and to guide them through the foot bearing of the centrifuge. The optic is shown in Figure 1. It consists of two achromatic lenses, two mirrors and the primary focussing lens of the Laser-Doppler-Anemometer itself. The laser beams are parallelised by the achromatic lens a, reflected vertically by mirror a, focussed after 80 mm with the achromatic lens b and reflected radially by mirror b. The measurement takes place in the secondary focal point f2.

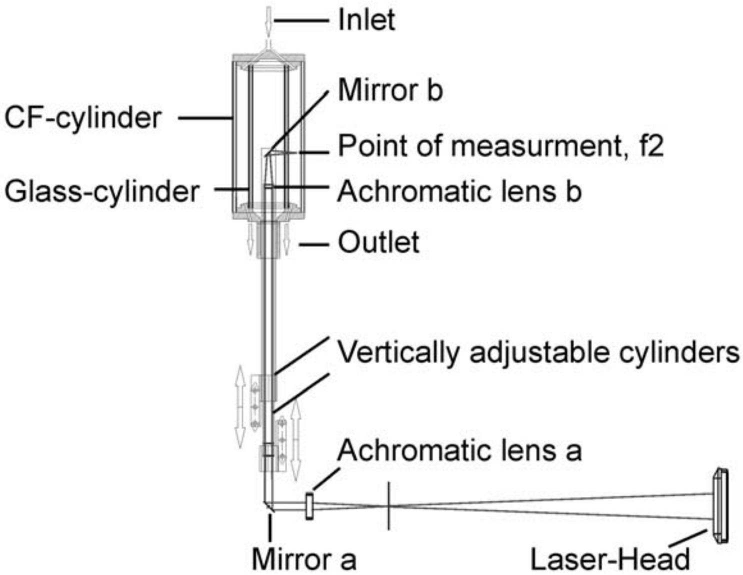

Figure 1: Laser optic and tandem-rotor of the centrifuge

A second rotor made of Duran® Glass is placed in the outer CF-Rotor. The glass cylinder rotates with the same revolutions per minute as the CF-Rotor does. The volume between both rotating cylinders is entirely filled with water. This tandem arrangement will be applied if a homogeneous plug flow profile is desired [10]. Tubular centrifuges without a tandem arrangement exhibit a boundary layer flow profile [6, 10, 11], which is disadvantageous for the separation efficiency. Furthermore the rotating glass exhibits a smooth surface, which eases the measurement with the laser-doppler-anemometer. An illustration of the centrifuge and the implemented optics is shown in Figure 1. The liquid enters the rotor at the top via two rotating filling pipes with a diameter of 3 mm and leaves the rotor at the bottom via two pipes with the same diameter. The CF-rotor has a length of 200 mm and an inner diameter of 100 mm. The outer diameter of the glass cylinder equals 60 mm.

The purpose of the centrifuge is the measurement of the flow patterns for different process conditions. A study of the separation efficiency for different products was not conducted.

Due to the vibrations of the machinery and the technical constrains of the laser-doppler-anemometer, the flow patterns were measured up to 8000 rpm until now. This corresponds to a centrifugal number of 3600.

3 Results

3.1 Screening

The screening efficiency of the semi-batch tubular bowl centrifuge was determined for two products. Figure 2 shows the particle size distribution of Aerosil 200 for the feed, the overflow and the sediment. The particle size distributions were measured with a Beckman Coulter photon correlation spectrometer type N4 plus. The feed is split into a coarse (sediment) and a fine (overflow) fraction at a rotational speed of 20000 rpm and a volume flux of 0.1 l/min. The fine fraction of the feed seems to be coarser than the overflow. This may be caused by the limitation of the measurement technique. Photon correlation spectroscopy bases on the Brownian motion of a single particle. The analysis of the autocorrelation-function enables the measurement of polydisperse products, but the standard deviation significantly increases. Furthermore the amount of coarse particles is overestimated. Thus the measurement of products with a polydisperse or bimodal particle size distribution causes a high deviation compared to the true data. Nevertheless Figure 2 proves the successful screening of submicron particles, since the fine fraction does not contain particles with a diameter above 390 nm. The coarse fraction is entirely separated in the centrifuge.

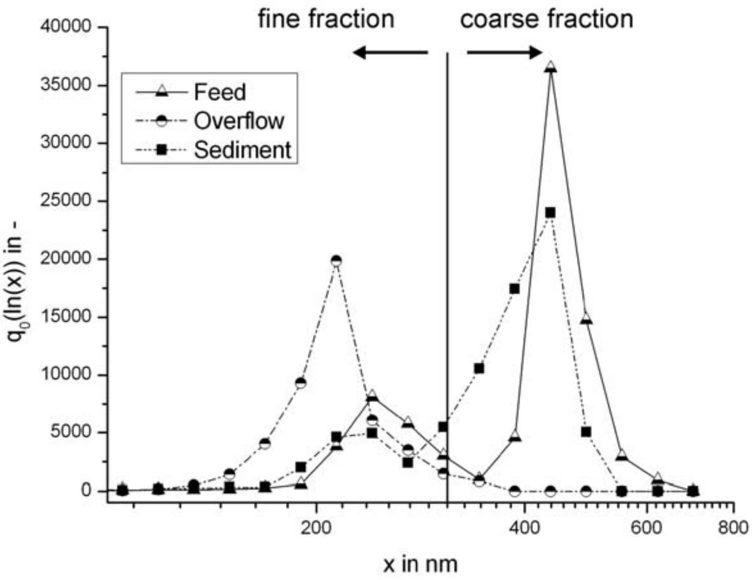

Figure 2: Screening of fine SiO_2 particles

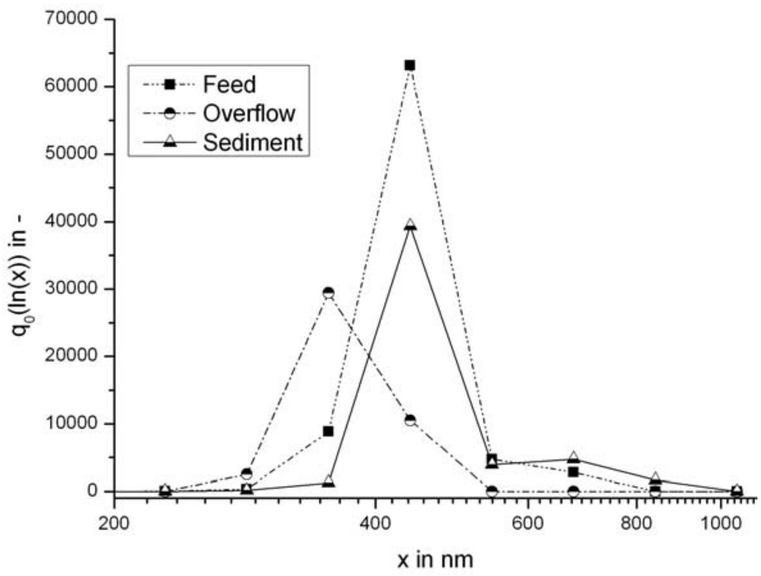

Figure 3: Screening of Polystyrene particles

The separation and screening of polystyrene particles is impeded by the low density difference between solid and liquid. Figure 3 shows the particle size distributions of polystyrene of the feed, the overflow and the sediment after the process at 40000 rpm and a feed flux of 0.1 l/min. Particles smaller than 350 nm could not be captured and occur only in the overflow. The obtained fine fraction contains particles ranging from 220 nm to 550 nm, but compared to the feed the amount of coarse particles is reduced considerably.

The calculated cut size of the centrifuge for the process with Aerosil equals 310 nm, which corresponds well to the experimentally determined cut size. The sintered agglomerates settle more slowly due to the high surface area than a single particle would do. This may cause the difference of approximately 80 nm between the calculated and the experimentally determined cut size. The same calculation for the process with the polystyrene leads to a cut size of 680 nm, which is 100 nm higher than experimentally determined. The calculation underestimates the cut size, which might be caused by the width of the boundary layer. If the assumed width is too small, the predicted cut size will be too high, due to the reduced residence time.

3.2 Tangential velocity profiles

Figure 4 shows the measured tangential velocity profiles inside the tubular centrifuge with the tandem arrangement of the glass and CF-rotor for rotational speeds of 2000, 4000, 6000 and 8000 rpm at a constant throughput of 1.2 l/min. The profiles were measured at an axial position of 100 mm, which is halfway between inlet and outlet. The solid lines are the calculated rigid body rotations. There is no deviation of the measured velocity compared to the rigid body rotation within the accuracy of the measurements. The standard deviation increases with higher radii. This is caused by the long distance between the glass and the point of measurement. The scattered light has to travel back to the detector, which is located in the laser head, so the quality of the signal decreases with increasing radius.

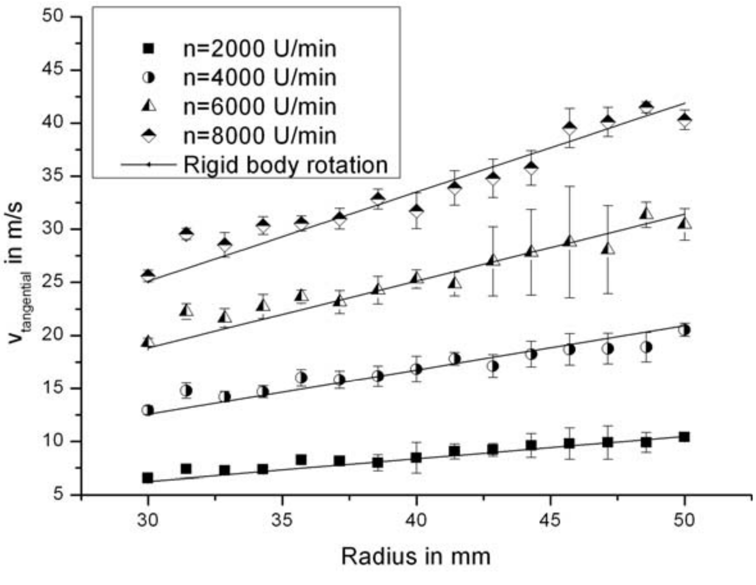

Figure 4: Tangential velocity profiles for rotational speeds of 2000 - 8000 rpm at a flow rate of 1.2 l/min, H=100 mm

The tangential velocity profiles have been measured for throughputs ranging from 0.5 l/min to 1.8 l/min. The obtained flow profiles are depicted in Figure 5. As done for the results shown in Figure 4, the measured profiles are compared with the rigid body rotation. There is no deviation of the measured velocity from the ideal behaviour within the accuracy of measurement.

Experiments with a laboratory centrifuge pointed out, that the fluid is not sufficiently accelerated tangentially with non rotating feed systems [6]. The inlet of the self-constructed centrifuge rotates with the same angular velocity as the rotor, assuring an efficient tangential acceleration of the liquid. Furthermore the inlet is submerged, which reduces unwanted turbulences as they occur in two-phase systems and a non-submerged inlet. For this reason a submerged inlet is often used in industrial centrifuges which exhibit a liquid-gas interface.

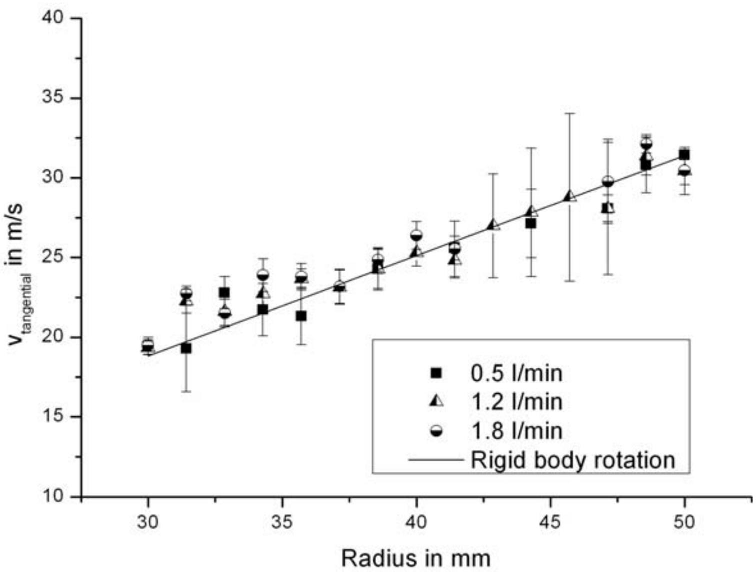

Figure 5: Tangential velocity profiles for flow rates of 0.5 - 1.8 l/min and a rotational speed of 6000 rpm, H=100 mm

3.3 Axial velocity profiles

The axial velocity profiles are measured for varying throughputs at a rotational speed of 1000 rpm. Figure 6 shows the dimensionless axial fluid velocity against the angle of rotation 30 mm behind the inlet. The fluid velocity is divided by the maximum fluid velocity, detected at a throughput of 1 l/min, yielding the dimensionless velocity. Since the fluid is fed to the centrifuge via two rotating filling pipes, the small inlet area causes a high fluid velocity at the inlet region. The water jet expands until the feed flows almost through the entire area of the rotor.

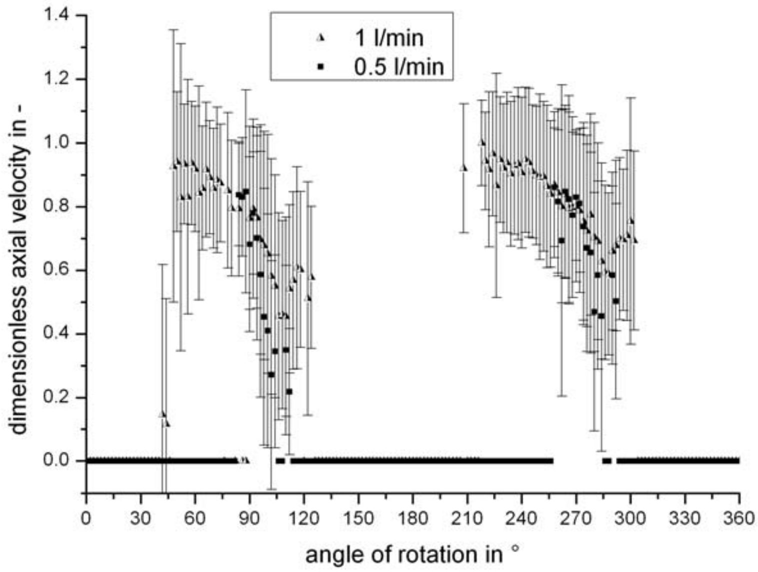

Figure 6: Dimensionless axial fluid velocity against angle of rotation for 1000 rpm

An increase of the throughput does not increase the axial fluid velocity, if the water jet expands more quickly. This behaviour was observed for throughputs increasing from 0.5 to 1 l/min. The liquid flow is detected at an angle of 30° for 0.5 l/min, whereas the angle is doubled at a feed flux of 1 l/min. The maximum velocity remains almost the same, which shows the dimensionless ordinate. The axial fluid velocity is doubled only at the section of homogeneous flow behind the influences of the inlet. So the length of the inlet section is important for the precise prediction of the residence time of the fluid in the centrifuge and depends on the inlet geometry. The inlet section may be improved by further inlet pipes which assure a homogeneous feed.

4 Conclusions

The screening of particles smaller than one micron in diameter is feasible with process machinery. Tubular bowl centrifuges reach the necessary centrifugal numbers for the successful screening of fine silica and polystyrene particles. The feed suspensions were split into a fine and a coarse fraction. Particles with a density difference of 50 kg/m³ and a size smaller than 350 nm could not be separated in the centrifuge. Higher centrifugal numbers and an improved channelling of the flow are necessary for the separation and screening of this kind of particles.

The analysis of the flow patterns was successfully conducted by Laser-Doppler-Anemometry. The fluid exhibits no lag of the tangential velocity compared to the rigid body rotation due to the rotating filling device. Thus the inlet geometry of the centrifuge seems to be appropriate for the effective tangential acceleration in fast rotating centrifuges.

The influence of the disturbances of the inlet section in dependency of the throughput was determined. The axial velocity decreases throughout the first 30 percent of the rotor length and then remains constant. This is caused by the expansion of the free jet at the rotating inlet pipe. Due to the quicker expansion of the inlet jet, the axial fluid velocity remains constant for varying throughputs below 1 l/min at the inlet section. An improvement may be made by adding more than two inlet pipes as it is in the presented case. This will enhance the homogeneous distribution of the feed and reduces the disturbances of the inlet and the axial expansion of these inhomogeneities.

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