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Application of magnetic bearing technology in high-speed centrifugation

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HIGHLIGHTS

- Magnetic drive and bearing technology was applied on a sedimentation type centrifuge.
- Separation experiments were performed on a technical scale for ultrafine particles.
- The outcome of the process is precisely adjustable via the operating parameters.

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ABSTRACT

Centrifuges represent a well-established tool in separation technology to handle high throughputs of suspensions with micron-sized particles. However, extraordinarily high centrifugal accelerations are required for even smaller particles. For this purpose, we make use of magnetic bearing and drive technology. We report the first application of this contactless technique in separation experiments. The presented prototype is based on a semi-continuous principle, where sediment is built up within the rotor while the liquid (which contains the fine fraction in case of classification) is discharged at the overflow weir. The new centrifuge allows rotational speeds of more than 64k min^{-1}. A broad variation of parameters with centrifugal accelerations of up to $C = 100{,}000$ and flow rates of up to 0.4 l/min provides a sound experimental basis for this study. The cut size and the product loss of three particle systems between 10 nm and 1 μm (silver nanoparticles, silica nanoparticles, and polystyrene) are precisely adjustable via the mentioned parameters. Furthermore, a good comparability with a commercially available device was found regarding the specific efficiency. We consider our approach to be a promising contribution to nanoscale separation technology where scalable high-throughput methods are rare.

1. Introduction

Applications of submicron and nanoscale solid particle systems are increasingly gaining in importance. In case that the production process, for instance crystallization, comminution or other syntheses, takes place in the liquid phase, a subsequent separation is required. In addition to a full-phase separation, it may be used to further influence size distribution width in the form of a classification step. However, a fundamental problem of this particle size range has been identified by Spelter et al. (2012) and Segets et al. (2013): Separation processes in the nanometer range are usually limited to extremely small throughputs and are not scalable, whereas established large-scale units in the micrometer range are not applicable to finer particle systems due to apparatus-based limitations.

An overview of the most common separation processes in the nanometer range is given by a topical review article by Kowalczyk et al. (2011). The latest developments of the techniques, which have the potential to be also used as a classification (separation into different size classes) or fractionation step (separation into fractions according to other properties than size), are discussed hereafter. Yeap et al. (2014) reported the small-scale batch fractionation of magnetic nanoclusters by use of low-gradient magnetophoresis, although a scale-up perspective is not outlined. Cyclical magnetic field flow fractionation (CyMgFFF) overcomes the major drawback of irreversible adsorption in standard MgFFF techniques, which leads to an improved sharpness (Bi et al., 2011). The throughput of this continuous process is below 1 ml/min and

Abbreviations: CyEFFF, Cyclical electrical field flow fractionation; CyMgFFF, Cyclical magnetic field flow fractionation; MgFFF, Magnetic field flow fractionation; NPs, Nanoparticles; SSP, Size-selective precipitation; PPE, poly(p-phenylene ether)

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the proof of concept was made with micrometer particles only. A similar principle was applied by using an electrical field (CyEFFF) instead of a magnetic field (Tasci et al., 2013). The authors achieved a good resolution even in the nanometer range, but the low throughput is comparable to that found by Bi et al. (2011). Membrane ultrafiltration was used to remove silica NPs from wastewater (Springer et al., 2013) as well as for separation and classification of CdTe quantum dots (Wu et al., 2014). Both publications point out that membrane fouling is a critical point in this kind of application. Doucet et al. (2004) proved feasibility of separation by size in a crossflow filtration device (microfiltration), although a large amount of fines remained in the retentate, which means a high product loss and low sharpness. The impact of diffusive effects and size-dependent membrane-particle interactions should also not be underestimated. Under certain conditions, shear flow diffusion may have a positive effect by an enrichment of smaller particles near the membrane, which enables classification even with pores larger than the largest particle size (Kromkamp et al., 2006). On the other hand, an undesired enrichment of larger particles near the membrane may be the consequence of size-dependent membrane-particle interactions (Bendixen et al., 2014).

Another approach to nanoparticle classification is size-selective precipitation (SSP). It plays a special role among the mentioned methods, since it is no real separation technique. It functions independently of a flow field, but combination with a separation step is necessary. For the first time, Segets et al. (2013) established an extensive evaluation procedure for the fine and coarse fractions of ZnS quantum dot classification. This promising – since basically scalable – technique has been improved regarding the choice of solvent and the redispersion (Segets et al., 2015).

The large-scale separation and classification of particles in the lower micrometer range is dominated by hydrocyclones and centrifuges. The principle of both is a rotating flow, which creates high centrifugal forces that lead to particle sedimentation. Hydrocyclones are continuously operated devices without rotating parts. As a decrease in cut size is obtained by a reduction of the cyclone diameter ($x_{T,50\%} \propto D^{0.41 \cdots 0.5}$) and an increase in pressure drop ($x_{T,50\%} \propto \Delta p^{-0.27 \cdots -0.25}$), the technical effort considerably rises for fine-particle systems (Bradley, 1965).

Using barium sulfate crystals (ρ=4.5 g/cm³) as an experimental product, cut sizes below 1 μm have recently been realized by means of a 10 mm hydrocyclone and a pressure drop of up to 50 bar. However, the occurrence of the so-called fish-hook effect, where small particles are unintentionally transported to the coarse fraction, led to a significant degradation of sharpness (Neesse et al., 2015).

A reduction of cut size is furthermore obtained by certain design features, such as conical installation in the upper cyclone part, but the effect is low (Hwang et al., 2012). A combination of these installations with a suitable inlet geometry may at least result in better classification sharpness values (Hwang et al., 2013). Cubic meter scale centrifuges are principally able to separate below 1 μm, too. Examples are decanter centrifuges (Müller et al., 1993) and disc stack separators (Wang et al., 2003, 2001). The drive components and the geometrical complexity of the parts within the process chamber, however, set limits on faster rotational speeds. In contrast to that, tubular bowl centrifuges have a simpler design. Our work focusses on this type. To realize extraordinarily high speeds, we use magnetic bearing and drive technology instead of conventional drive concepts. The only reference to an application to centrifuges so far is restricted to a standard speed range and does not contain any separation results (Werfel et al., 2001). Therefore, our work is a novel and guiding contribution to ultrafine particle separation and classification on a technical scale.

2. Theory

2.1. Centrifugation and sedimentation

Assuming a laminar flow and no hydrodynamic interactions between different particles, the settling velocity of a spherical particle is given by the Stokes equation:

$$u_0 = \frac{\Delta\rho \cdot x^2 \cdot C \cdot g}{18 \cdot \eta} \quad (1)$$

In Eq. (1), $\Delta\rho$ is the density difference between solid and liquid phase, x is the particle size, g is the gravity of Earth, and η is the liquid's viscosity. The relative centrifugal acceleration

$$C = \frac{\omega^2 \cdot r}{g} \quad (2)$$

represents an important operating parameter for sedimentation-type centrifuges. While Eq. (1) is only valid for sufficiently diluted systems, Richardson and Zaki (1954) found a correlation to consider the influence of an increase in concentration on the settling velocity (in a Reynolds number range <0.2):

$$u = u_0 \cdot (1-\phi)^{4.65} \quad (3)$$

2.2. Scalability and comparability

2.2.1. Sigma approach

Different types of centrifuges may be suitable for a certain separation task. Moreover, the operator might be interested in the scalability of the process if parameters need to be changed or a scale-up from laboratory to technical scale is planned. For this purpose, Ambler (1959) developed a model to classify results, which are derived from a parameter variation or different centrifuges, respectively. Assuming Stokes' sedimentation conditions, a particle size with a 50% separation probability is considered. Its settling velocity

$$u = \frac{Q}{2 \cdot \Sigma} \quad (4)$$

is directly related to a constant flow rate Q and the Sigma value

$$\Sigma = \frac{V \cdot \omega^2}{g} \cdot \left[\ln\left(\frac{2 \cdot r_2^2}{r_2^2 + r_1^2}\right)\right]^{-1} \quad (5)$$

which contains the angular velocity ω and furthermore combines the geometric properties of the apparatus. In Eq. (5), V stands for the rotor volume, g is the gravity of Earth, r_1 is the weir radius, and r_2 is the radius of the inner rotor wall. While Ambler (1959) gives correlations for several centrifuge types, Eq. (5) is valid for tubular bowl centrifuges only. For comparability, the ratio of flow rate Q to Sigma value Σ is formed. Similar results for centrifuges of the same type from 1 to n are expected when the condition

$$\frac{Q_1}{\Sigma_1} = \frac{Q_2}{\Sigma_2} = \cdots = \frac{Q_n}{\Sigma_n} \quad (6)$$

is fulfilled (Ambler, 1959).

A modified version

$$\frac{Q_1}{\mu_1 \cdot \Sigma_1} = \frac{Q_2}{\mu_2 \cdot \Sigma_2} = \cdots = \frac{Q_n}{\mu_n \cdot \Sigma_n} \quad (7)$$

of Eq. (6) is given by Svarovsky (1985), where a correction factor μ is introduced to allow a comparison of different centrifuge types. The centrifuge-specific parameter μ therefore corrects for non-ideal flow field and separation conditions. The main reasons for the discrepancy between theoretical and experimental separation