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Classification and diafiltration of polydispersed particles using cross-flow microfiltration under high flow rate

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ABSTRACT

A novel particle classification system based on the integration of classification and diafiltration, both of which utilize cross-flow microfiltration under high flow rate, was successfully developed. In this system, particles could be classified into two fractions, one of which was smaller than pores of the membrane and collected as permeate and another collected as retentate. Diafiltration was then conducted to completely remove the smaller particles contained in the retentate to obtain another fraction consisting of larger particles. Importantly, classification and diafiltration should be conducted under cross-flow microfiltration at a high flow rate because the sufficiently large hydrodynamic lift force attributed to the cross-flow microfiltration operation keeps the membrane surface clean so it can act as a sieve. To show the effectiveness of our particle classification system, we demonstrated stable classification and continuous diafiltration for 26 h of bi-dispersed suspensions containing particles with diameters of 0.6 and 1.5 μm and polydispersed suspensions with particle diameters of 0.1–3 μm.

1. Introduction

Particle processing plays an important role in a variety of fields, including the food and cosmetics industries [1–5]. Because of increasing demand for monodispersed particles in the sub-micron region, particle classification techniques that fractionate polydispersed particles to obtain monodispersed particles have been extensively investigated. There are many well-established wet classification techniques; however, most of these cover particles larger than 1 μm and cannot be applied to classification of smaller particles. There have been several reports of the successful classification of particles in the sub-micron region by electrical hydro-cyclones [6,7]. Additionally, novel classification techniques using hydrodynamic forces in micro-channels with specially designed structures have been reported [8–10].

We also studied a novel particle classification system that uses a microfiltration membrane [11,12]. This classification system was designed to classify particles by using pores of membranes as a sieve. The concept of this system is very simple, with particles smaller than the pores of membranes collected in the permeate and those larger than the pores collected in the retentate. The cut-off size is determined by the pore size of the membranes, which generally ranges from 0.01 to 10 μm; therefore, this method has the potential to classify particles at any cut-off diameter simply by choosing an appropriate membrane with a pore size that corresponds to the target diameter for classification, even in the sub-micron region. Many investigators [13–15] have focused on the sieving effect of membranes for particle classification. However, none have been successful to particle classification. The greatest barrier to use of this method is membrane fouling, which occurs when particles are deposited onto the membrane surface in the early stage of microfiltration, preventing the sieve from being useful for classification. Indeed, Meier et al. [16] proposed a unique concept for particle classification in which fine particles deposited onto the membrane surface through cross-flow microfiltration of polydispersed particles can be collected by backwashing as fractions containing only smaller particles with a sharp size-distribution. However, this concept is entirely different from the basic concept of classification via the use of membrane pores as a sieve. Additionally, it is not practical from an industrial viewpoint.

In addition, diafiltration should be carried out following microfiltration of polydispersed particles. In this method, smaller substances in feed solution are completely washed away from larger substances by the continuous or discontinuous addition of the same amount of solvent that permeated though membranes to the feed tank. This purification is essential to complete classification, and has often been employed for purification of proteins and biomacromolecules [17–20]. In the case of the particle classification system, diafiltration processing should be employed to fractionate larger particles with a sharp size-distribution on the retentate side because the retentate also contains

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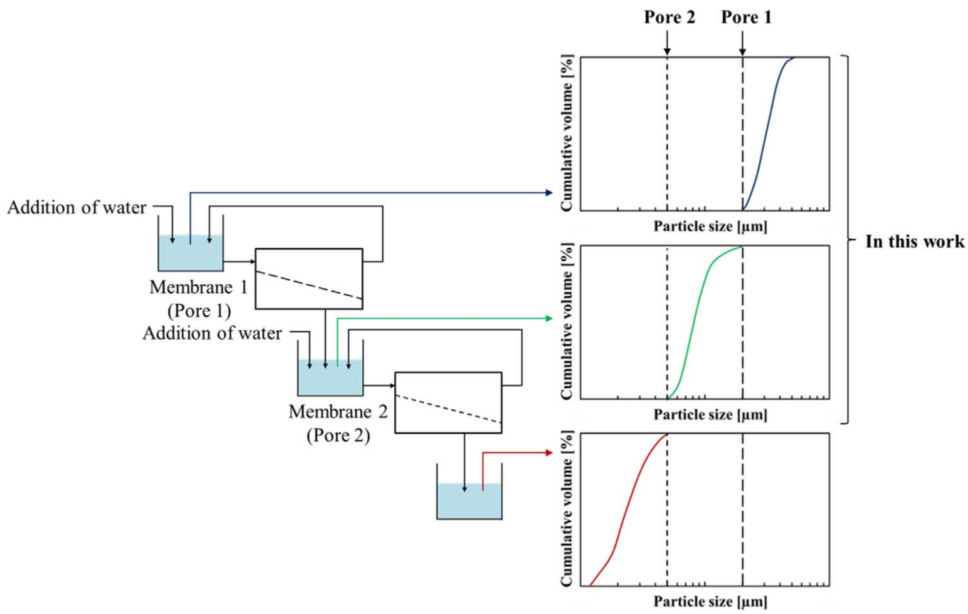

Fig. 1. A concept of complete classification system.

smaller particles, even if the rejection of smaller particles is zero throughout the first microfiltration classification. However, to the best of our knowledge there have been no reports of diafiltration operations of polydispersed particles to obtain monodispersed particles. Furthermore, fouling by particles would also become a problem to the development of stable diafiltration because fouling prevents the permeation of smaller particles through the membranes.

In our previous study, we demonstrated the complete prevention of fouling by particles in cross-flow microfiltration under a high flow rate for the first time [21]. The hydrodynamic lift force attributed to high flow rate was shown to be one of the key factors for the complete prevention of fouling. In addition, utilization of this method enabled the stable classification of bi-dispersed suspensions containing silica particles with diameters of 0.6 and 1.5 µm at a mass ratio of 1 to 9 during cross-flow microfiltration. Thus, in the present study, we developed a method of diafiltration processing bi-dispersed suspensions containing two monodispersed particles by cross-flow microfiltration under high flow rate; this method is an essential step toward the development of a complete classification system using microfiltration membrane pores, as shown in Fig. 1, none have achieved. We then extended this classification technique to polydispersed particles. We demonstrated classification and diafiltration of polydispersed suspensions by cross-flow microfiltration under a high flow rate for the first time.

2. Experimental

2.1. Diafiltration of bi-dispersed particles

The membrane housing with a channel height of 1.0 cm that we previously developed [21] was modified by replacing the porous plate with a PTFE filter plate having a pore size of 150 µm and a thickness of 5 mm. The total length and effective membrane area were 54.7 cm and 50 cm^2, respectively. Schematic diagrams of the experimental setup for diafiltration and classification are shown in Fig. 2. Cellulose mixed ester (MCE) microfiltration membranes (Toyo Roshi Kaisha, Ltd., Japan) with a nominal pore size of 1.0 µm were used. The test solutions consisted of two different monodispersed silica particles with nominal diameters of 0.6 µm and 1.5 µm (Nippon Shokubai Co., Ltd. Japan), and the initial volume of the feed solution was 6 L. The total feed concentration was 1000 ppm; the 0.6 µm and 1.5 µm particles were mixed, and the particle size distribution of this feed solution was shown in Fig. 4. The flow rate was set at 28 L min^{-1}. Under these conditions, the average cross-flow rate was calculated to be 1.9 m s^{-1}. The initial flux was set at 3.0×10^{-5} m^3 m^{-2} s^{-1} with 0.05 kPa of applied pressure. The time courses of flux and the concentration of the smaller particles in permeate were measured for 26 h and the particle size distributions in feed were measured using a laser diffraction particle size analyzer (LA-950V2, HORIBA, Ltd., Japan). After the diafiltration test, the surface and the cross-section of the membrane used were observed with a field-emission scanning electron microscope (FE-SEM, JSM-6701F, JEOL Ltd. Japan).

2.2. Classification of polydispersed particles

MCE microfiltration membranes with a nominal pore size of 0.65 µm and polydispersed silica particles with diameters ranging from 0.1 to 3 µm (Admatechs Co., Ltd. Japan) were used. The concentration in feed was 1000 ppm. The retentate and permeate were fully recycled to keep the concentration of the particles in feed constant during the classification tests under flow rates of 28, 26, 24 and 20 L min^{-1}, which corresponded to 1.9, 1.7, 1.6 and 1.3 m s^{-1}, respectively. This is because the concentration of the particles in the feed solution may affect the classification performance, so we would like to eliminate the potential influence. The initial flux was set at 2.0×10^{-5} m^3 m^{-2} s^{-1} with 0.14 kPa of applied pressure. Each classification test was performed for 180 min, and the time courses of flux and permeation ratio were measured. The permeation ratio ($1-R_{obs}$) was defined as follows:

$$1 - R_{obs} = C_p/C_f \tag{1}$$

where, R_{obs} is the observed rejection, and C_p and C_f are the concentrations in permeate and feed estimated from the mass of the particle. In addition, the particle size distributions in permeates were measured by a laser diffraction particle size analyzer at 30 and 180 min. After the classification tests, the membrane surfaces were observed using an FE-SEM.

2.3. Diafiltration of polydispersed particles

MCE microfiltration membranes with a nominal pore size of 0.65 µm and polydispersed silica particles were used. The concentration in the feed was 1000 ppm. The diafiltration tests were conducted for 26 h under a flow rate of 28 and 20 L min^{-1}. The initial flux was set at 2.0×10^{-5} m^3 m^{-2} s^{-1} and the time courses of flux and the concen-