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Dispersion of coagulated nano-particles in high shear field

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Well dispersion of nano-particles is necessary in order to bring out their unique properties, such as solubility, reactivity, and electromagnetic, optical and mechanical properties. However, it is difficult in general to disperse nano-particles homogeneously in liquid media because they are very prone to coagulate each other. In this study, we have developed and constructed a new apparatus for dispersing particles that basically consists of co-axial rotor and a static vessel to generate a high shear field between them. Coagulated particles are subject to high shear forces which enable to disperse the coagulated particles. In this paper we have firstly designed the dispersing apparatus, and have then carried out the dispersion tests with calcium carbonate particle which has tens nanometer size as a primary particle. The dispersion performance has been evaluated by measuring particle size distribution with taking account of shape of rotors, dimensions of the gap between rotor and vessel, rim speed of the rotor and operating time. It has been found that a smaller gap results in higher dispersing performance since higher velocity gradient is formed in the gap.
Key words: particle dispersion, nano-particles, high shear field, calcium carbonate

1. INTRODUCTION

Well dispersion of the nano-particles in liquid or gas media is necessary in order to reveal their unique properties which are owing to their small particle size (high specific surface area) such as solubility, reactivity, and unusual properties of electromagnetism, optics and mechanics different from the bulk. However, it is difficult in general to disperse nano-particles into the primary particles since they are very prone to coagulate each other due to high cohesive forces based on their high specific surface area. Therefore, the dispersion of nano-particles shall be one of the most important key techniques in so-called nano-technology. The method for particle dispersion can be categorized mainly into two approaches as follows: 1) mechanical dispersion methods in which physical energy more than cohesive forces working between particles is put to the coagulated particles to be dispersed, 2) physicochemical dispersion methods in which particle surfaces are subjected to chemical modification and the particle dispersibility would be improved. The former mechanical methods include use of ultrasonic wave illumination [1], media follow [2, 3] and so forth. The later physicochemical methods include surface modification treatment using silane coupling agents or alcohols and so forth [4, 5].

In this study, we have developed an apparatus for dispersing particles that basically consists of co-axial rotor and a vessel to generate a high shear field between them. Coagulated particles are subject to high shear forces which enable to disperse the coagulated particles. The apparatus is easy to be applied in continuous process and we would intend to combine the apparatus with chemical surface dispersion method in the next phase of our research program. In this paper we have firstly designed the dispersing apparatus, and have then carried out the dispersion tests with calcium carbonate particle which has tens nanometer size as a primary particle. The dispersion performance has been evaluated by measuring particle size distribution in the calcium slurry with taking account of shape of rotors, dimensions of the gap between rotor and vessel, rim speed and treatment time.

2. EXPERIMENTAL

2.1 Design of apparatus for particle dispersion with high speed rotational flow

Figure 1 shows a schematic diagram of experimental setup used in this study for particle dispersion with high speed rotational flow. The setup consists of a slurry tank, a pump circulating the slurry and a dispersion vessel which consists of a rotor and a static vessel. The slurry flown from the tank comes into the vessel from the bottom and goes through a gap between a rotor and a vessel and comes out from the top of the vessel and then return into the tank. The slurry tank is filled with nitrogen gas (with slight gas flow) apart from the slurry, and is equipped with a cold water jacket to prevent the slurry from overheating.

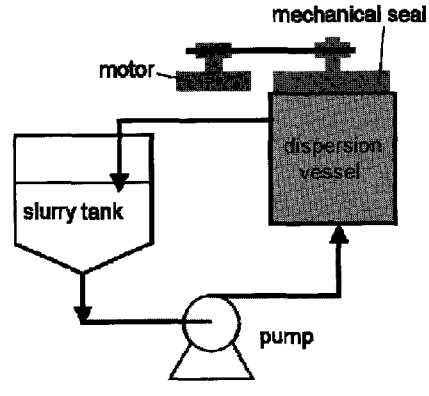
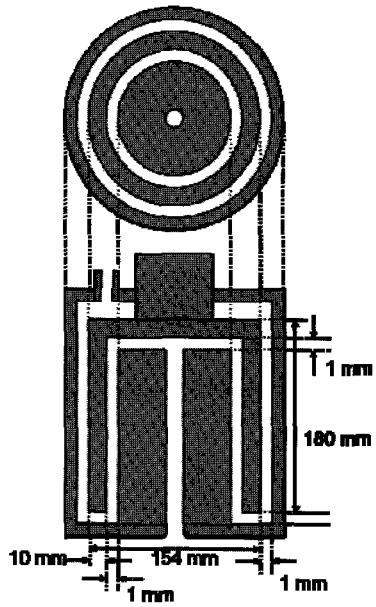
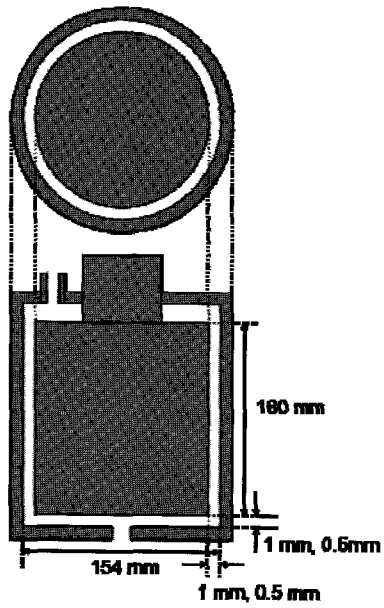


Fig.1 Schematic diagram of experimental setup for dispersing particles in slurry

The two types of rotor and vessel were designated as shown in figures 2. The first configuration is the rotor in shape of a headstand cup and the vessel in shape of a cylinder with a pin cylinder at the center of the bottom as shown in Fig.2 (a), this is called "type I" vessel in this paper. The other is a single cylindrical rotor and a vessel as shown in Fig.2 (b), this is called vessel "type II" vessel in this paper. In both the cases, rotor and vessel are in a co-axial position with the center for rotation of the rotor. Major dimensions are shown in Figs.2, and the gap between the rotor and the vessel is 1.0 mm for type I and 1.0 or 0.5 mm for type II.



(a) Type I: headstand cup rotor



(b) Type II: single cylindrical rotor

Figs.2 Schematic diagrams of rotor and vessel

2.2 Sample slurries

Calcium carbonate (average size 60nm, Nanocube 60, Nittetsu Mining, Japan) was used as a sample powder, and sodium hexametaphosphate (Kanto Chemical, Japan) was used as a dispersant. The calcium carbonate was mixed with distilled water and was stirred using a magnetic stirrer for 30 min. The solid loading was kept at 4.5 mass % (1.65 vol.%) throughout this paper. The amount of dispersant added to the slurry is 1.0 mass % to the mass of the powder in the slurry.

2.3 Particle dispersion test

2 L of the calcium carbonate slurry was put into the slurry tank and was circulated with the constant flow rate of 0.5 L/min. to the dispersion vessel and then return to the tank. The circulation was taken for 10 minutes for all the dispersion tests as a preliminary mixing. The rotor eventually began to rotate with a constant velocity in the range of 21 to 40 m/s as rim speed. At certain treatment time elapsed, the slurry was sampled from the tank and particle size distribution of the sample was measured using a laser diffraction method (Microtrac MT3000, Nikkiso, Japan).

3. Results and discussion

3.1 Effect of rotor shapes for dispersion performance

Figure 3 shows particle size distributions of slurries without dispersion treatment (original) and with the treatment using either Type I or II (gap 1 mm) rotor with the rim speed of 40 m/s for 5 minutes. The y-axis in this figure indicates frequency for each size fraction in volume percent, and the x-axis indicates particle size in a logarithmic scale. As compared to the original sample of which average size is 21.4 μm, the treated samples have smaller particle size distributions. The data of Type II shows lower frequency in larger size fractions around 10 μm, and higher frequency in smaller fractions less then several μm as compared to that of Type I, while the difference is not so significant. The Type I vessel have a longer path than Type II with the same gap of 1.0 mm, so that the result imply that only the gap with outer side of the Type I vessel can contribute to particle dispersion.

3.2 Effect of gap size for dispersion performance

Figure 4 shows the effect of gap size for particle dispersion performance. In these cases, Type II vessel was used with the same rim speed of 40 m/s. The narrower gap (0.5 mm) obviously results in smaller size distribution as compared to wider gap (1.0 mm). This is because of velocity gradient of shear flow at the gap increase with decreasing the gap size. In the range of this paper, the gap less than 0.5 mm has not been tested but would expected to be more efficient for dispersing coagulated particles.

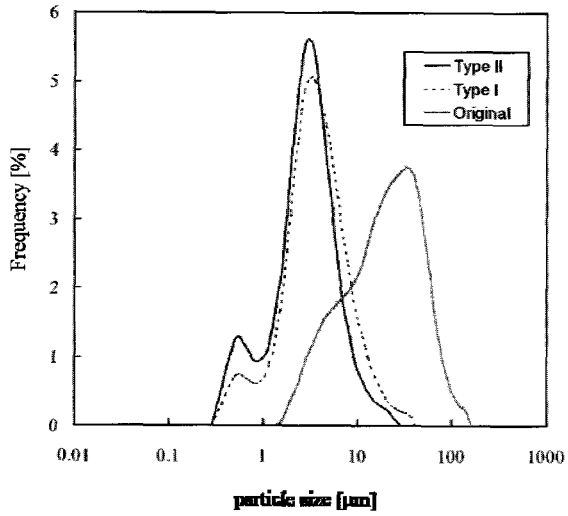


Fig.3 Particle size distribution of the slurries without dispersion treatment (original) and with the treatment using either Type I or II vessel (shown in Figs.2) with rim speed of 40 m/s. No dispersant was added to the slurries.

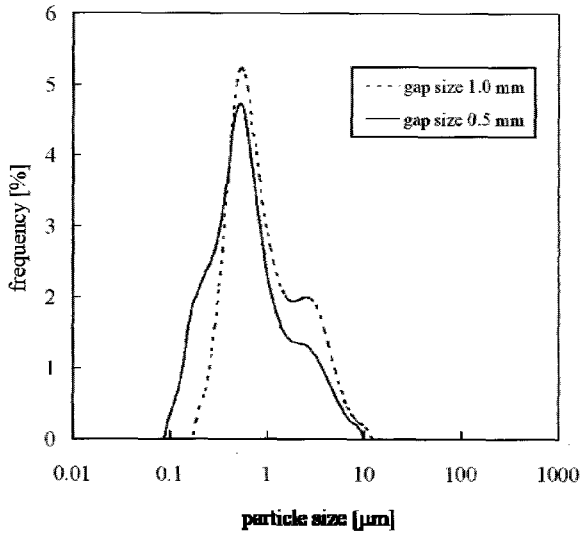


Fig.4 Particle size distribution of the slurries with the dispersion treatment using Type II rotor with different gap sizes of 1.0 or 0.5 mm (treatment time 15 min, rim speed of 40 m/s, dispersant was added to the slurries.)

3.3 Effect of rim speed for dispersion performance

Figure 5 shows relationship between particle size and rim speed. The y-axis is shown in logarithmic scale. The keys in this figure correspond to the particle size at which cumulative undersize ratio is 10 % (d10), 50 % (d50) or 90 % (d90). The particle sizes decrease with increasing the rim speed. The decrease in d10 for high rim speed (25-40 m/s) is slighter than that of d50 and d90. This might be because larger coagulated particles are easier to be dispersed but smaller particles especially in submicron size are harder to be dispersed.

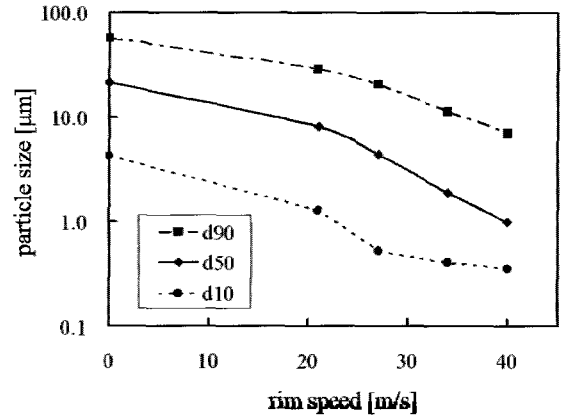


Fig.5 Relationship between particle sizes with cumulative undersize ratio of 10% (d10), 50% (d50) and 90% (d90) in volume and rim speed of the Type II vessel with gap size 0.5 mm (treatment time 5 min. dispersant was added to the slurries.)

3.4 Effect of treatment time for dispersion performance

Figure 6 shows time course of the particle sizes. The particle size decrease considerably by 5 minutes treatment, and gradually for the time 10 to 15 minutes. The 5 minutes is roughly equal a time required for all slurry input in the tank to come into the dispersion vessel and come back to the tank. Therefore the result can be interpreted that the only one-time dispersion treatment, which means one-time passing through the reaction vessel, can reduce the particle size in the level of the lowest limit that is attainable by this apparatus developed in this study.

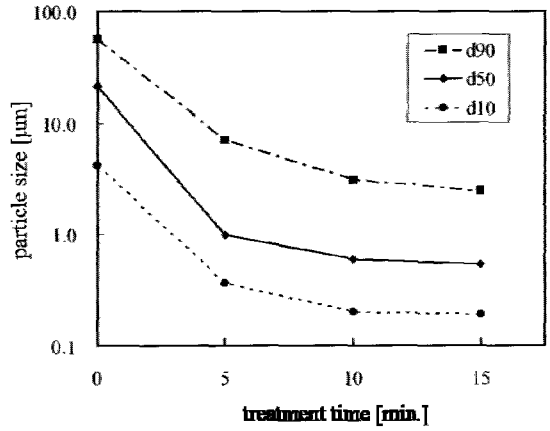


Fig.6 Relationship between particle sizes with cumulative undersize ratio of 10% (d10), 50% (d50), and 90% (d90) in volume and treatment time (rim speed 40 m/s, dispersant was added to the slurries.)

Even after dispersing treatment with rim speed 40 m/s for 15 minutes, the particle size is 540 nm as d50 at 15 min in Fig.6 and is much larger than the primary particle size (60 nm). This might be attributed to re-coagulation which might occur immediately after dispersion treatment. Therefore we have been planning that chemical surface modification to the particles using

a coupling agent is carried out in the high shear field, which would enable to enhance dispersibility of the dispersed particles and hence to prevent from re-coagulating them. This will be published elsewhere [6].

4. Conclusions

We have developed and constructed a new apparatus for dispersing particles that basically consists of co-axial rotor and a static vessel to generate a high shear field between them. The dispersion tests with calcium carbonate particle which has tens nanometer size as a primary particle have been carried out. The dispersion performance has been evaluated by measuring particle size distribution with taking account of shape of rotors, dimensions of the gap between rotor and vessel, rim speed of the rotor and operating time. It has been found that a smaller gap results in higher dispersing performance since higher velocity gradient is formed in the gap.

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