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Measurement of particle size distribution of silica nanoparticles by interactive force apparatus under an electric field

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ABSTRACT

This paper describes the measurement of particle size distribution of silica nanoparticles by interactive force apparatus (IFA) under an electric field in order to suggest the application of the apparatus to the measurement of particle size distribution. The results were compared with results obtained from size measurement by dynamic light scattering. D_{50} measured by IFA was closer to the average particle size determined by TEM (5 nm). Also, when compared the results under three different supply voltage, (1) the results at 0.01 and 0.02 V were almost identical while (2) these results were different from the one at 0.04 V. The results indicate that breakage of coagulated particles possibly occur due to electric breakdown. The distribution measured by IFA (D_{50} = 5–7 nm) was larger than the one measured by DLS (D_{50} = 1 nm). The electric breakdown was explained by curve fitting of three different particle size distribution functions with particle size distribution obtained from IFA measurement.

1. Introduction

It is important to manipulate the degree of dispersion and coagulation of nanoparticles in various media for many industrial processes [1,2]. Therefore, there is a need for understanding the dispersion and coagulation of nanoparticles in the media. There are many techniques for evaluating the dispersion and coagulation of nanoparticles, i.e. size measurement [3], turbidity measurement [4], contact angle measurement [5], zeta potential measurement [6], force measurement [7,8] as well as combination of these techniques [9,10]. However, these methods usually are not suitable for evaluating the dispersion and coagulation of particles in sample solutions of high concentration and/or the solution with no optical transparency. On the other hand, these kinds of solutions are commonly used in the many industrial procedures, such as separation of fine particles [11,12] and deposition of fine particles on fibers [13].

In this study, we focused on particle size measurement for evaluating the degree of dispersion and coagulation of nanoparticles. The techniques can be divided in two categories, i.e. measurements in dry condition and measurements in wet condition [2]. In dry condition, the microscopic studies based on optical microscopy, scanning electron microscope (SEM) and transmission electron microscope (TEM) are the common techniques for size measurement of particles. In wet condition, the techniques using laser source (i.e. the dynamic light scattering and laser diffraction) are the common techniques for size measurement of particles. Although these techniques have several advantages, they have some drawbacks. The techniques in dry condition are not applicable for measuring the size of particles in solutions. On the other hand, the techniques in wet condition are not usually suitable for measuring the size of particles at high particle concentrations or no optical transparency.

The interactive force apparatus (IFA) was designed for determining the degree of dispersion and coagulation of particles suspended in a functional fluid under a magnetic or electric field [14,15]. The apparatus is a direct measurement technique, not depending on the concentration and optical transparency of the solution. Moreover, the measurement can be conducted in both the aqueous solution and organic solvent. However, the results were not fully compared with other methods and particle size distribution was not drawn due to the limited number of data acquisition. In this study, IFA was used to measure the particle size distribution of silica nanoparticles with increasing the number of data acquisition in order to evaluate the availability of IFA for the measurement. The current experimental setup allows detection of primary particles and/or aggregates of particles in sample suspension. The results were compared with results obtained by using an apparatus of dynamic light scattering and TEM.

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2. Materials and methods

2.1. Materials

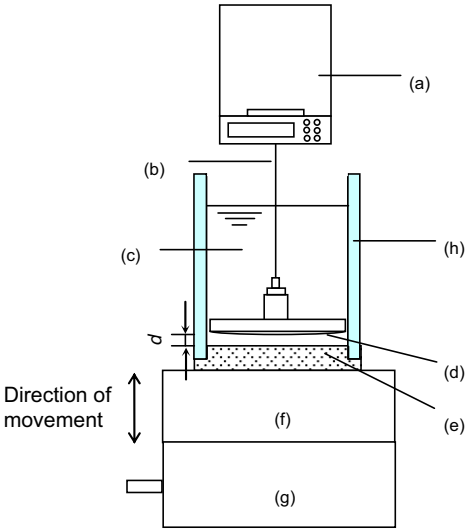
Aqueous solution containing silica particles provided by Nissan Chemical (Snowtex XS) was used for the measurement. A typical photograph of the particles taken by TEM is shown in Fig. 1. Fig. 1 indicates that particles tend to disperse, but some particles may aggregate in the solution. Averaged particle size was about 5 nm determined by analysing a series of photographs taken by TEM. The number was provided by the supplier. pH of the solution was 9, and concentration of silica nanoparticles in the solution was 30 wt.%. Spherical silica particles were chosen as a sample due to its sharp size distribution of silica particles, easiness of varying size, and simplicity of controlling dispersion and coagulation state; therefore, silica particles are suitable as model particles to evaluate the availability of IFA for the measurement of particle size distribution.

2.2. Experimental setup

2.2.1. Interactive force apparatus

The apparatus has three parts, i.e. main part (which consists of electric balance, hemisphere and flat plate), control part (i.e. personal computer, piezo-stage controller and voltage supplier), and detecting part (i.e. multi-meter and oscilloscope). The interactive force apparatus measures the interactive force between two surfaces, i.e. the gold-coated glass hemisphere and the brass flat plate, (which is fixed at the bottom of the sample cell). The main part measures the weight of the hemisphere immersed in a sample solution with decreasing the distance between the two surfaces at a certain speed. The control part is employed to adjust a supply voltage, regulates the movement of piezo-stage and collects data from the balance and the piezo-stage controller. The detecting part, on the other hand, measures the contact point where the hemisphere and flat plate attach.

Fig. 2 shows the main part of the apparatus. The hemisphere is hung to the electric balance, and remains still in the sample solvent (Fig. 2). The weight of the hemisphere is measured by using the electric balance, and recorded by the personal computer, while the flat plate moves toward the hemisphere, decreasing the distance d from a certain distance (e.g. 100 nm) to 0 nm (Fig. 2). Hemisphere was selected to make point contact to the flat plate. The movement of piezo-stage by applying voltage is used to regulate the distance between the hemisphere and flat plate (Fig. 2). The rate of movement is 1 nm/s for 100 nm distance at these experiments presented in this paper. The piezo-stage is located on a z stage. Detecting the contact point of the hemisphere to the flat plate at the bottom of the cell (i.e. the surface distance d was zero) is quite important for calculating the surface distance because the point determines a certain distance from the contact point (e.g. 100 nm) for the measurements.



(a) Electric balance
(b) Platinum wire
(c) Sample solution
(d) Hemisphere (Gold-coated)
(e) Flat plate (Brass)
(f) Piezo-stage
(g) Z stage
(h) Glass cell

Fig. 2. Schematic diagram of main part of experimental setup for interactive force apparatus.

The measured weight is converted to the interactive force by using the Derjaguin equation [16]:

$$\frac{F_{(D)_{sphere}}}{R} = 2\pi W_{(D)_{plane}} \quad (1)$$

where $W_{(D)plane}$ is the interactive free energy, $F_{(D)sphere}$ is the interactive force between the hemisphere and the flat plate, and R is the curvature radius of the glass hemisphere, respectively. The interactive force was plotted as a function of surface distance between the hemisphere and flat plate (i.e. force-distance curve) in order to determine the size of fine particles.

During the measurement, an electric field is applied between the hemisphere and flat plate, and thus dielectric particles are arranged toward the direction of electric field in the area between two plates. When the two plates are close to each other, two different forces (i.e. repulsive and attractive forces) alternately act on particles due to structure changes of the particles (Fig. 3). Under an electric field generated between two parallel plates, pearl chains of dielectric particles form towards the direction of electric field [18]. In terms of shape of the chains, linear chains form over triangular chains under an electric field since the former is more stable in terms of potential energy [19]. The repulsive force occurs when the particle chain structure is stretched by compressive force; whereas the attractive force occurs when the particle chain structure is broken. The cycle of repulsive and attractive forces is a primary size of particle or size of aggregate, which depends on the degree of agglomeration. At pH 9 silica particles tend to disperse due to the electrostatic repulsion force acting on the particles. In

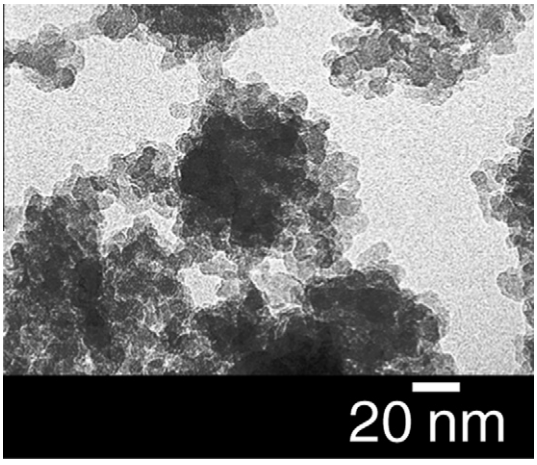


Fig. 1. TEM Photograph of silica nanoparticles.