MECHANICAL DISPERSAL OF DRY SILVER NANOPowDER IN WATER USING ULTRASONIC CAPILLARY WAVE

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ABSTRACT
In this research, an ultrasonic atomizer (1.63 MHz) was employed to build up an ultrasonic capillary wave dispersal (UCWD) system to disperse dry silver nanoparticles (in the form of powder) in water without any additional chemical phases. The distribution behavior of silver nanoparticles in the colloid was carried out by dynamic light scattering scanning electron microscope. Our findings indicated that silver nanoparticles (25 nm in diameter) were homogeneously dispersed by UCWD system in water at room temperature with the average size of dispersed particles of around 101 nm and particle size ranging between 68 and 164 nm. The average size of dispersed particles achieved by UCWD was 6 times smaller than that dispersed by sonication bath (19 kHz) and 4 times smaller than that by ultrasonic cavitation (20 kHz). Moreover, the range of particle sizes achieved by the proposed method was much narrower than that obtained by the other two conventional methods.

1. INTRODUCTION
Nanoparticles have been widely used; their application potential is very diverse and based on particle size – surface area (Hayashi et al., 2005, Jokanović et al., 2004).

Nanoparticles can be produced uniformly with respect to both shape and size. However, owing to their high surface energy, nanoparticles always tend to impact on each other and agglomerate together then exist at micro or larger scale (Gedanken, 2007). Such agglomeration strongly reduces the special functions and characteristics of nanoparticles. Therefore, nanoparticles must be well dispersed prior to use. Nevertheless, dispersing nanoparticles in liquid is always a very big challenge (Hayashi et al., 2005), especially dispersing heavy and low-melting temperature materials such as silver nanoparticles in water without any additional chemical effects (Hayashi et al., 2005).

Dispersal has been extensively studied to increase the dispersing efficiency of nanomaterials. Many mechanical dispersal methods have been proposed, where ultrasonic cavitation and sonication bath are the most powerful techniques and have been widely applied to disperse nanoparticles in liquid (Jokanović et al., 2004, Gedanken, 2007, Yang et al., 2004). However, normally the size of dispersed particles was still far from the original size of the nanoparticles, that is around twenty to hundreds times that of original nanoparticle size (Jokanović et al., 2004, Gedanken, 2007, West and Malhotra, 2006, Manh Toan et al., 2009).

This paper proposed a new technique to disperse dry silver nanopowder in water without any additional chemical phases, which is named as ultrasonic capillary wave dispersal (UCWD). This technique only applied the ultrasonic- capillary wave to create droplets of liquid in order to disperse dry silver nanoparticles in water at room temperature and without any additional chemical effects. With this technique the size of dispersed particles could be roughly predicted (Jokanović et al., 2004). On the other hand, the vibrating membrane was not eroded thus the colloid was not contaminated by membrane erosion during dispersal as the ultrasonic cavitation does. The experiment results confirmed that proposed technique could well disperse silver nanoparticles in water; with particle size approached much closer to the original size of nanoparticle (around 4 times of original nanoparticle size) and particles were much more uniform than those obtained by conventional methods.

2. EXPERIMENT

![Fig.1 Schematic of ultrasonic capillary wave dispersal system (UCWD)](image)
In the proposed dispersal, the 1.63-MHz ultrasonic atomizer was equipped with a copper probe for creating a vibrating chamber where colloid of silver nanoparticle was contained, vaporized and also cooled by cooling water as shown in Fig. 1.

The mixture of 1g dry spherical silver nanopowder with an average size of 25 nm (supplied by Yong-Zhen Technomaterial Co.LTD, Taiwan) and 1000 ml pure water, which had been premixed by magnetic stirring, was used as the material in this study. The first one-third of this mixture was dispersed by UCWD. During dispersal, the power supply of ultrasonic generator was maintained at 50 (VDC). Cooling water was circulated to maintain the colloid inside vibrating chamber at room temperature. The level of colloid inside vibrating chamber also was kept at fixed level, around 120 mm from the surface of vibrating membrane. Under that condition, the mist (micro water drops) created by ultrasonic capillary wave was very stable. This mist was pulled through pure water by a vacuum pump that drives micro water droplets containing nanoparticles to be condensed into liquid.

The other two portions of colloid pretreated by magnetic stirring were dispersed by sonication bath at a frequency of 19 kHz and ultrasonic cavitation at a frequency of 20 kHz, respectively, for 30 minutes under the same room temperature.

During these dispersals, no chemical element or chemical effect was applied; therefore they are pure mechanical dispersals. After dispersing, the samples were immediately taken to study distribution behaviors of nanoparticles in colloid. Size distribution of agglomerated particles in the dispersed colloids was investigated by dynamic light scattering. And then their structures, distribution behavior, agglomeration was also obtained by scanning electron microscope (SEM). The colloid contamination also has been examined by energy dispersive spectroscopy (EDS).

3. ANALYSIS

3.1 Dynamic light scattering analysis

As mentioned above, the size distribution of particles in colloids after dispersal was investigated by Malvern Zetasizer nano-instrument, ZS model at 25°C with square glass cuvette with square aperture and measurement position at 4.65 mm from the bottom of cuvette. The size distributions by intensity of particles in colloids were shown in Fig. 2.

Fig. 2: Size distribution of silver nanoparticles in colloids dispersed by the three different techniques.

The thin solid line shows the size distribution by the intensity of dispersed particles in the colloid achieved by the sonication bath. As can be seen, silver nanoparticles were well dispersed, but not homogeneously; the average size of particles is found to be about 600 nm. In particular, even though this dispersal could reach an average size of 600 nm, there were still about 4% of particles in the colloid whose size exceeded 5000 nm. This revealed a significant difference in size among dispersed particles, and the average size of particles was still very big.

The dashed line denotes the size distribution by the intensity of particles in the colloid achieved by ultrasonic cavitation. As can be seen, the size of particles ranged from 190 nm to 712 nm, with the average size of about 400 nm. This result also indicates that ultrasonic cavitation could obtain more uniform dispersion of silver nanoparticles, and the size distribution was more concentrated than that achieved by the sonication bath.

The bold solid curve represents the size distribution by the intensity of particles in the colloid achieved by UCWD. As can be seen, silver nanoparticles were perfectly dispersed in water. The average size of dispersed particles was 101 nm, much smaller than that obtained by ultrasonic cavitation or by sonication bath. Moreover, their size distribution concentrated within the range from 68 nm to 164 nm, a range much narrower than that obtained by the other two ultrasonic dispersal approaches. Hence, the dispersed silver nanoparticles by UCWD were truly homogeneous, a result that has never been achieved by other conventional mechanical dispersals, not even conventional ultrasonic dispersals approaches.

The result of dynamic light scattering analysis demonstrated that with UCWD, dry silver nanoparticles were deeply dispersed in water, and particles were very homogeneous. Whereas with ultrasonic bath dispersal or ultrasonic cavitation dispersal, silver nanoparticles were dispersed but the average size of particles was far bigger and particles were not homogeneous.

3.2 Nano structure analysis

In addition to dynamic light scattering - size distribution analysis, SEM was applied to study structure, distribution behavior and gathering of particles in the colloids after dispersal. Samples were mounted on a double faced adhesive carbon tape and sputter with thin gold layer in order to improve the conductivity of sample, and then surface topography was analyzed with LEO 1530 scanning electron microscope operated at an acceleration voltage of 5 kV. And the results were shown in Fig 3.

Fig 3a shows the distribution behavior of particles in colloid which were dispersed by UCWD. From this image, dispersed particle size could be easily checked, it
is around 60 – 200 nm and particles are very uniform. This image once again confirms the result of dynamic light scattering analysis and also shows how well UCWD could disperse dry silver nanoparticles in water.

Similarly, Fig. 3b shows structure of particles in colloid which were dispersed by ultrasonic sonication bath at 19 kHz. With this image, distribution of agglomerated particles in colloid is visible. First of all, particles are not uniform. Beside some small particles with their size about 400-700 nm, there are some other bigger particles with their size about 1.5 – 3 µm. This means ultrasonic bath radiation might disperse silver nanoparticles in water up to 400 nm but just few particles have their size around 400 nm. Others have size around 10 times bigger than that. In other word, with ultrasonic bath dispersal, there was a very big difference between dispersed particles.

With ultrasonic cavitation dispersal, particles seem to be dispersed more homogeneously than ultrasonic bath dispersal. This point was demonstrated by SEM image in Fig. 3c. The image presents the structure of particles with average size around 800 nm. And there was not a big difference among particles.

In order to study the contamination of colloid during dispersing process; composition of colloid before and after being dispersed were inspected by energy dispersive spectroscopy (EDS) and the results were shown in Fig 4. The result from EDS indicated that during dispersals, particles dispersed by proposed technique (UCWD) were not contaminated. In other word, there were no difference composition between original silver nanoparticles and dispersed particles obtained by UCWD

3.3 Discussions

The experiment results showed that, with heavy material like silver nanoparticles, it is very difficult to disperse by non chemical dispersal. Specially, with original mechanical methods, even conventional ultrasonic dispersion technique could not approach high dispersing efficiency: Average size of dispersed particles is large, and particles are not uniform.
Ultrasonic cavitation technique was widely applied to disperse nanoparticles in liquid. But when cavitation occurs, the generation and collapse of bubbles will generate the temperature of over 5000 K inside the bubbles (Gedanken, 2007, Manh Toan et al., 2009, Wang et al., 2007). Transient cavitations could produce an implosive impact strong enough to break up the chemical bonds of material (Gedanken, 2007, Manh Toan, 2009) thus probably making particles melted and combined or gathered. Therefore besides breaking the agglomerated particles, dispersed particles in liquid, ultrasonic cavitation can cause nanoparticles to be gathered. On the other hand, during ultrasonic cavitation, cavitation erosion will occur. It will damage the surface of horn and makes the colloid contaminated (ASTM G 32-06, 2006).

Compared with ultrasonic cavitation, sonication bath would not contaminate the colloid by surface membrane erosion, but the dispersive efficiency was not very good. For example, the average size of dispersed particles was about 1.5 times larger than that dispersed by ultrasonic cavitation, and there was significant difference in size between dispersed particles.

However with UCWD, average size of dispersed particles was small (4 times smaller than conventional ultrasonic cavitation methods) and nearly approached size of nanoparticle. Silver nanoparticles were homogeneously dispersed. Moreover, the colloid was not contaminated by membrane erosion.

4. CONCLUSION

Results from experiment pointed out that the study was very successful in finding a pure mechanical dispersing technique that well disperses silver nanoparticles in water. The particles in colloid dispersed by proposed technique are very uniform, their size in the range of 68-164 nm. This result can not be reached by using conventional methods such as ultrasonic bath or even ultrasonic cavitation.

During dispersal, temperature was maintained at room temperature so this dispersal was not the ultrasonic spray pyrolysis technique. It demonstrated that this study was different from previous studies that applied the ultrasonic spray pyrolysis technique to disperse nanoparticles in liquids.

The experiment results also indicated the ultrasonic capillary spray is a powerful technique to disperse nanoparticles in liquids without any additional chemical phases.

5. REFERENCES


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