Special Feature: Nanomaterials and Processing

Research Report

Fabrication of Colloidal Nanoparticles by High Efficiency Laser Ablation

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ABSTRACT Novel laser ablation methods are proposed for the production of colloidal nanoparticles. Ag nanoparticles were fabricated by laser ablation of a Ag powder target settled on the bottom of a piriform flask filled with pure water, while Pd nanoparticles were fabricated by laser ablation at the air-liquid interface using a Pd powder target agitated in pure water by a magnetic stirrer. In each case, both the nanoparticle yield and the uniformity of size were significantly improved over that achieved with a conventional laser ablation in liquid. Laser ablation at the air-liquid interface is a novel method for the fabrication of nanoparticles that is quite different from the conventional method. The mechanism for the production of nanoparticles is discussed and the stability of nanoparticles in the liquid is attributed to electrostatic repulsion from by-product ions adsorbed on the nanoparticles. In addition, the density of the plasma induced by laser ablation was estimated to be a very important factor for the fabrication of single non-agglomerated nanoparticles.

KEYWORDS Laser Ablation, Nanoparticles, Air-liquid Interface, Plasma, Colloid

1. Introduction

Much attention has been focused on nanoparticles because they exhibit different properties from their bulk counterparts. Nanoparticles have been studied for applications in various fields.(1-5) Laser ablation has been used for the generation of nanoparticles and clusters in various atmospheres. The properties of the nanoparticles produced by laser ablation in various atmospheres are influenced by the experimental conditions. For example, laser ablation in vacuum or in the presence of an inert (or particular) gas atmosphere can produce sub-nano, nano and micro sized particles,(5-7) which has made it possible to investigate the catalytic activities of various nano- and sub-nanosized particles. However, it is difficult to effectively collect nanoparticles produced by laser ablation in vacuum or in the presence of an inert gas atmosphere.

In contrast, nanoparticles produced by laser ablation in liquid can be effectively collected. One of the advantages of laser ablation in liquids is that nanoparticles can be produced without the need for chemical reagents such as stabilizers and reducing agents, which result in the formation of ligand-free nanoparticles. The high stability of colloidal nanoparticles without additional stabilizers is also an advantage. Laser ablation in liquids can be applied to produce various metal,(8-12) alloy,(13,14) semiconductor,(15-17) and organic(18,19) nanoparticles. The size distribution of nanoparticles produced by laser ablation in liquid is generally broad, and the production of monodisperse nanoparticles has not been previously reported. Low nanoparticle yield with this technique has also been a significant problem, although improvement in the nanoparticle yield has been recently reported,(20) whereby a complicated experimental setup such as a flow cell was used to improve the nanoparticle yield. In contrast, we have reported two simple methods to improve the nanoparticle yield without the use of a flow cell.(21,22) One is laser ablation of a powder target settled on the bottom of a piriform flask filled with liquid.(21) Laser ablation of a powder target in a liquid agitated by a magnetic stirrer has also been reported.(22) In the conventional laser ablation method, a focused laser is irradiated into the powder target agitated in a liquid. The important difference between our method and the conventional method is the irradiation scheme. In our method, the laser is irradiated through the bottom of a piriform flask, whereby the products are discharged into the liquid. As a result, the incident laser light cannot interact with the products because there are no products in the light path. Another method is laser ablation at the air-liquid interface using a powder target agitated by a magnetic stirrer.(22) High density plasma is known to be produced by laser ablation in liquid due to the confinement effect. However, the plasma produced by laser ablation at the air-liquid interface is expected to expand, which results in the formation of lower density plasma.

Here, we report a novel method for the production of colloidal nanoparticles, which easily allows for the production of monodisperse single nanoparticles. The stability of the nanoparticles in liquid is discussed based on the results of product analysis. In addition, the mechanism for nanoparticle generation by these novel methods is also discussed.

2. Experiments

2. 1 Laser Ablation of Powder Target Settled on the Bottom of a Flask Filled with Water

Figure 1 shows a schematic illustration of the experimental setup. A piriform flask was filled with pure water and commercially available Ag powder target (Nilaco, 325 mesh) was settled. Unfocused second harmonic light from a Q-switched Nd:YAG laser system (Spectra Physics, Pro-290, 532 nm, 10 Hz, 400 mJ/pulse, 7 ns) was irradiated for 60 min through the bottom of the piriform flask. After laser irradiation for 60 min, the Ag nanocolloid was obtained. The product was observed using scanning transmission electron microscopy (STEM) operated at 200 kV. The UV-Vis absorption spectrum of the Ag nanocolloid was measured with a spectrophotometer.

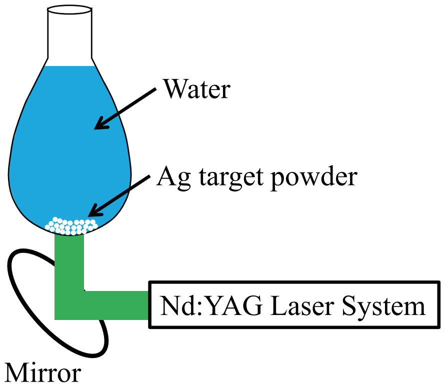

Fig. 1 Experimental setup for laser ablation of powder target precipitated on the bottom of a piriform flask filled with water.

2. 2 Laser Ablation at the Air-liquid Interface

Figure 2 shows a photograph and schematic of laser ablation at the air-liquid interface. A commercially available Pd powder target (Nilaco, 300 mesh) was agitated in water by a magnetic stirrer. The second harmonic light from a Q-switched Nd:YAG laser system (Spectra Physics, Pro-290, 532 nm, 10 Hz, 800 mJ/pulse, 7 ns) was irradiated to induce ablation at the air-liquid interface for 60 min. The incident laser light was focused onto the air-liquid interface using a quartz lens with a focal length of 200 mm. After laser irradiation for 60 min, the Pd nanocolloid was obtained. The product was analyzed using STEM, UV-Vis absorption and X-ray photoelectron spectroscopy (XPS).

3. Results and Discussion

3. 1 Laser Ablation of Powder Target Settled on the Bottom of a Flask Filled with Water

Figure 3 shows the Ag nanocolloid prepared by laser ablation of a Ag powder target on the bottom of a piriform flask filled with water. **Figure 4** shows the UV-Vis absorption spectrum of the Ag nanocolloid. The large absorption peak observed at approximately 400 nm is the plasmon band, which indicates the formation of Ag nanoparticles in water. The absorption intensity was higher than previously reported. The concentration of the Ag nanocolloid was estimated to be higher than previously reported Ag nanocolloids prepared by conventional laser ablation in liquid because the absorbance is dependent on the concentration of the Ag nanoparticles dispersed in water. The Ag nanoparticle concentration was calculated to be 0.2 g/L according to the decrease in the amount of target Ag powder, which corresponds to a nanoparticle yield of 20 mg/h and 1.39 μg/J. This is higher than that previously obtained with conventional laser ablation in liquid.(10) In conventional laser ablation in liquid, the laser is focused in the suspension, so that the incident laser light interacts with the product and target particles, even if the laser fluence is smaller than the ablation threshold. As a result, laser light is attenuated by particle interaction

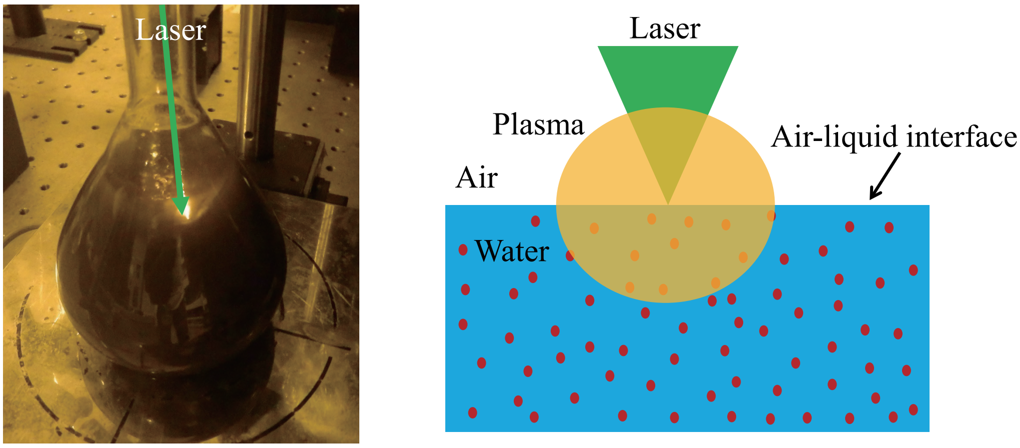

Fig. 2 Photograph (left) and schematic (right) of laser ablation at the air-liquid interface.

Fig. 3 Photograph of Ag nanocolloid prepared by laser ablation of powder target precipitated on the bottom of a piriform flask filled with water.

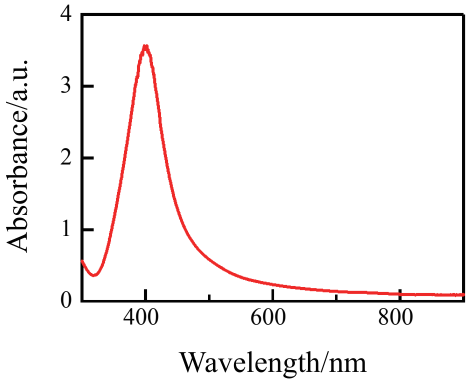

Fig. 4 UV-Vis absorption spectrum of Ag nanocolloid prepared by laser ablation.

such as absorption and scattering, which prevents the effective ablation of target particles. In contrast, both product particles and target particles cannot interact with the incident laser light in our proposed method. Therefore, the shape and size of the product particles cannot be changed by laser irradiation. In addition, stable laser light energy can be irradiated to the target particles. The ablation rate is not dependent on the irradiation time because the product and target particles cannot interact with the incident laser light; therefore, the nanoparticle yield was significantly improved.

Figure 5 shows an STEM image of the Ag nanoparticles. Monodisperse single Ag nanoparticles were observed, the average size of which was approximately 2 nm. Monodisperse nanoparticles have not been previously fabricated by laser ablation in a liquid. The results indicate that the novel method

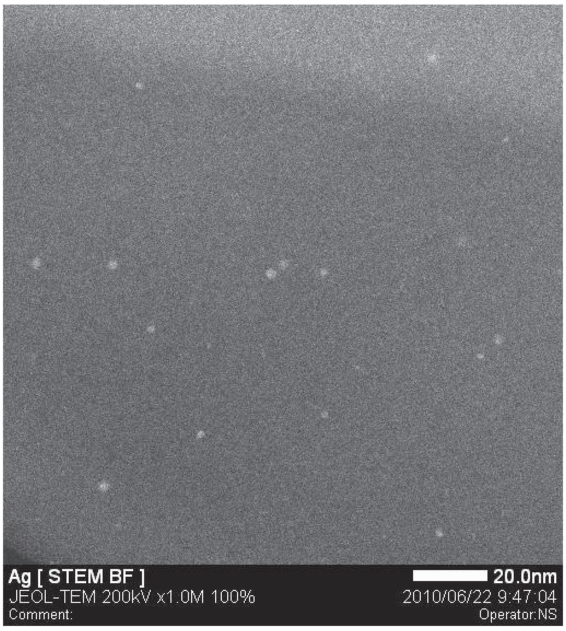

Fig. 5 TEM image of Ag nanoparticles prepared by laser ablation.

proposed here may improve not only the nanoparticle yield but also the size uniformity. The incident laser light cannot interact with product and target particles; therefore, target particles are ablated by the laser light with stable energy, which results in an improvement in the size uniformity. In addition, the Ag nanocolloid was very stable, as others have also reported.(11,12) It is thought that nanoparticles produced by laser ablation are charged and the electrostatic repulsion contributes to the stability of products. The novel laser ablation method presented here can produce a stable monodispersion of Ag nanoparticles in a liquid without the use of chemical reagents such as surfactants, which is a significant advantage over other chemical methods. For example, such surfactant-free nanoparticles could be easily incorporated into mesopores due to their smaller size.(23)

The antibacterial activity of the as-prepared nanocolloid against Bacillus coli (B. coli) was investigated. **Figure 6** shows the antibacterial activity of the as-prepared Ag nanocolloid against B. coli. The amount of bacteria decreased with time. Rather than antibacterial activity, bactericidal activity against B. coli was observed.

3. 2 Laser Ablation at the Air-liquid Interface

The target particle was confirmed to be an inhomogeneous distribution of particle sizes and shapes. The target powder was agitated in water by a magnetic stirrer and ablated at the air-liquid interface by irradiation with the second harmonic of the Nd:YAG laser system. Ablation was induced by the high-energy pulsed laser light focused at the air-liquid interface. A large sound was generated due to the laser-induced breakdown, and photoemission from the laser-induced plasma at the air-liquid interface was observed, as shown in Fig. 2. The residual target particles were separated from the product particles by spontaneous sedimentation due to gravity. **Figure 7** shows a photograph of Pd nanocolloid prepared by laser ablation at the air-liquid interface. Stable yellow colloidal nanoparticles were obtained in water. **Figure 8** shows UV-Vis absorption spectra for the Pd nanocolloids prepared by laser ablation with various intensity at the air-liquid interface. Absorption peaks were observed in the UV region. The peak intensity was dependent on the incident laser energy, while the peak wavelength was independent of the laser energy, which indicates that the efficiency of ablation and nanoparticle formation depend significantly on the pulse energy. The UV-Vis absorption spectra of

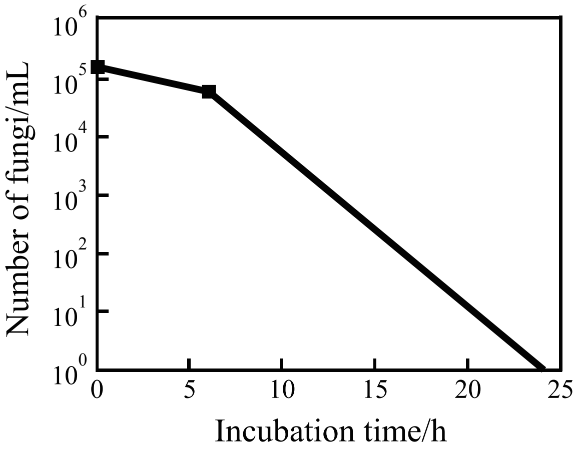

Fig. 6 Antibacterial activity of as-prepared Ag nanocolloid against B. coli.

Fig. 7 Photograph of Pd nanocolloid prepared by laser ablation at the air-liquid interface.

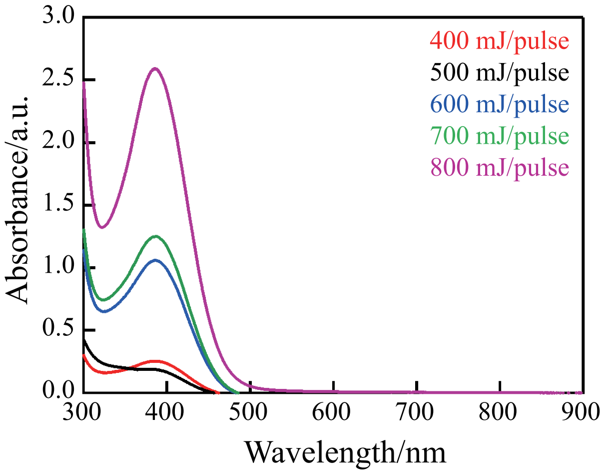

Fig. 8 UV-Vis absorption spectrum of Pd nanocolloid prepared by laser ablation at the air-liquid interface.

the Pd nanoparticles prepared by laser ablation at the air-liquid interface were clearly different from that of Pd nanoparticles produced by laser ablation in liquid.(24,25) This indicates that the chemical state of the nanoparticles produced by laser ablation at the air-liquid interface are different from nanoparticles produced by laser ablation in liquid. The absorption properties of Pd nanoparticles produced by laser ablation at the air-liquid interface are similar to those reported by Cristoforetti et al.,(26) where the absorption peaks in the UV region were attributed to Pd oxide species. However, the peak wavelength of the Pd nanoparticles prepared by laser ablation at the air-liquid interface was distinctly different from their report, thus indicating a difference in chemical state of the Pd nanoparticles.

The nanoparticle yield was estimated to be greater than 20 mg/h and it varied widely. **Figure 9** shows an STEM image of the Pd nanoparticles prepared by laser ablation at the air-liquid interface. The nanoparticles aggregated during sample preparation for STEM. The size of the primary particles was estimated to be less than 5 nm. Monodisperse Pd nanoparticles ranging between 1 and 5 nm in size, which are rarely produced by laser ablation in liquid, were observed. The plasma density produced during conventional laser ablation in liquid is known to be very high(27) due to the confinement effect of the liquid. In contrast, the density of plasma produced by laser ablation at the air-liquid interface can be expected to be much smaller because the plasma can easily expand. As a result, the collision probability is lower and the growth of particles lesser, which results in the formation of small particles.

Figure 10 shows XPS spectra for the products deposited on a Si wafer. These results show that the Pd nanoparticles were oxidized, and NO_3 and NO_2 ions were produced because the laser irradiation excites not only the target particles, but also molecules in the gas phase and in the water. Therefore, the gas phase effects on nanoparticle formation by laser ablation at the air-liquid interface were investigated. XPS spectra of Pd nanoparticles prepared by laser ablation at an Ar-liquid interface under the same conditions as laser ablation at the air-liquid interface were also measured. Pd metallic nanoparticles were obtained and nitrogen oxides were not detected due to replacement of air with Ar. These results revealed that laser light focused onto the air-liquid interface excite gas molecules in air as well as the target particles, which results in the production of nitrogen oxide. This is also evidence that laser ablation occurred at the air-liquid interface. The results also indicate that nitrate ions contribute to the stability of the nanoparticles because nanoparticles prepared by laser ablation at the Ar-liquid interface were aggregated.

The mechanisms for nanoparticle formation and the stability of the nanoparticles were considered. While the density of plasma induced by laser ablation in liquid is very high due to the confinement effect, it can be expected that the plasma induced by laser ablation at the air-liquid interface can expand, which results in the formation of lower density plasma. There are many particles activated by laser ablation at the air-liquid interface. In the plasma induced by laser ablation at the air-liquid interface, ionic NO_2^- and NO_3^- are produced by chemical reaction between nitrogen and oxygen, and these compounds are activated by laser ablation. Nanoparticles produced by laser ablation at the air-liquid interface are probably protected by these by-product ions, which results in the stability of nanoparticles due to electrostatic repulsion between adsorbed by-product ions. The pH of the Pd nanocolloid was in the range 2-3, which also indicates that laser ablation at the air-liquid interface induced a chemical reaction.

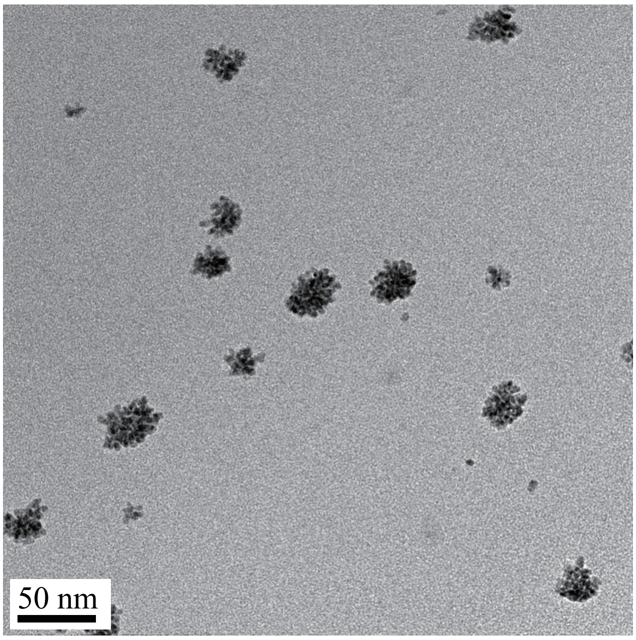

Fig. 9 STEM image of Pd nanoparticles prepared by laser ablation at the air-liquid interface.

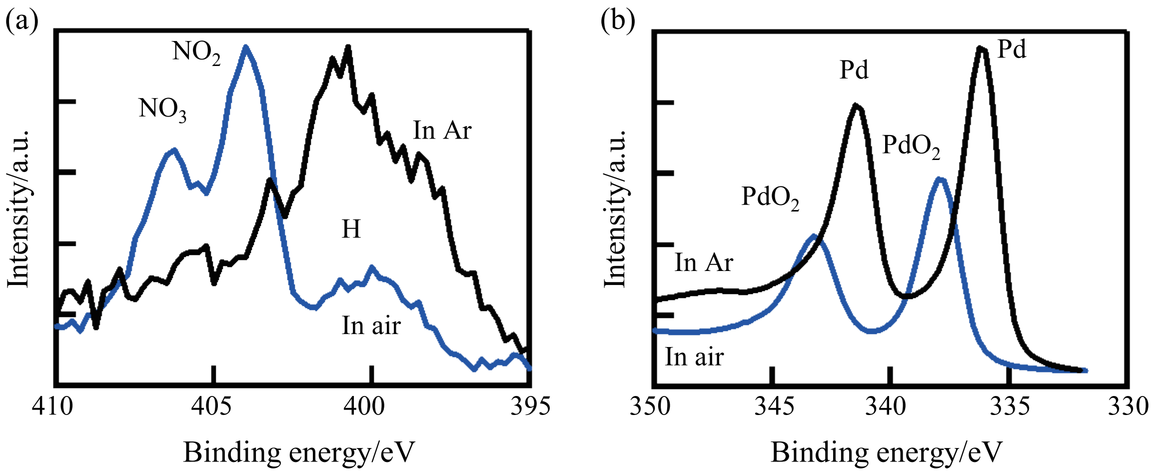

Fig. 10 (a) N 1s and (b) Pd 3d XPS spectra of Pd nanoparticles prepared by laser ablation at the air-liquid and Ar-liquid interface.

4. Conclusion

Novel laser ablation methods for the formation of colloidal nanoparticles were proposed with a focus on improving the efficiency of colloidal nanoparticle formation by laser ablation. In each method, stable nanoparticles were dispersed in water without the use of chemical reagents, and the nanoparticle yield was significantly improved. Ag nanoparticles prepared by laser ablation of a powder target exhibited large absorbance around 400 nm due to the plasmon band and bactericidal activity against B. coli. Surfactant-free monodisperse nanoparticles can be easily incorporated into the mesopores due to the smaller size.

Laser ablation at the air-liquid interface is a novel method that is quite different from the conventional laser ablation method. The size distribution of products obtained with this method were very narrow; Pd nanoparticles were stably dispersed in water. The replacement of air with Ar revealed the gas phase effect on nanoparticle generation. With Ar flow, unstable Pd metallic nanocolloids were obtained. These results indicate that the stability of Pd nanoparticles prepared by laser ablation at the air-liquid interface can be attributed to electrostatic repulsion due to by-product ions adsorbed on the Pd nanoparticles.

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Fig. 4
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