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Preparation of Carbon Nanoparticles by Long Pulsed Laser Ablation in Water with Different Laser Energies

V. Thongpool[1, 2, a], S. Denchitcharoen[1, b], P. Asanithi[1, c] and P. Limsuwan[1, d]

[1]Department of Physics, Faculty of Science King Mongkut's University of Technology Thonburi, 126 Pracha-Utid Road, Bangmod, Thoongkru, Bangkok 10140, Thailand

[2]School of Physics, Faculty of Science and Technology Rajamangala University of Technology Thanyaburi, 39 M.1 Klong 6, Thanyaburi, Pathumthani 12110, Thailand

[a]nuchphysics@hotmail.com, [b] d_somyod@yahoo.com , [c]piyapong.asa@kmutt.ac.th, [d]opticslaser@yahoo.com

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Abstract. Carbon nanoparticles were synthesized via laser ablation of graphite target in distilled water. The particle sizes of carbon nanoparticles were investigated as a function of laser energy: 1, 1.5, and 2 J/pulse. Using water as a matrix during synthesis is another potential way to control the particle size. The mean size increases when increasing the laser energy. The particle morphology is a globular shape. The sizes of the nanoparticle were evaluated from both SEM image analysis and Scherrer's equation. The sizes from the equation are smaller than those obtained from SEM, which indicates that the particles are polycrystallines.

Introduction

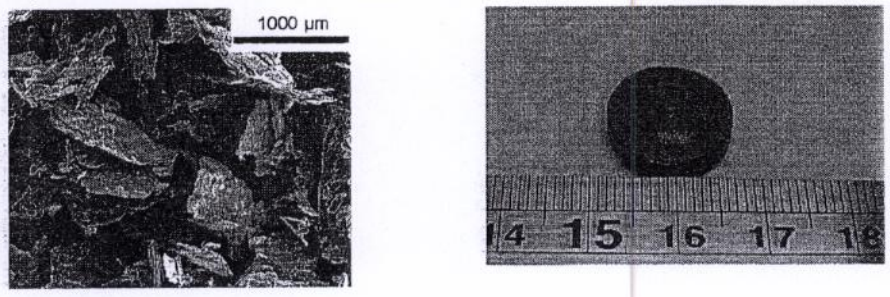
Nowadays, nanomaterials are becoming of interest due to their novel physical and chemical attributes. Carbon nanomaterials are important for many applications such as catalysis supports, oil adsorbents [1], drug delivery, hydrogen storage, junction device, and sensor [2]. Various methods have been used to prepare carbon nanostructure, e.g., arc discharge in protection gases, microwave plasma chemical vapor deposition and supersonic cluster beam deposition [3, 4]. Recently, laser ablation in liquid is widely used for synthesizing carbon nanomaterial. This technique was first reported by Patil and co-workers in 1987 by using pulsed laser to ablate a pure iron target in water [5]. After the discovery, pulsed laser ablation in liquid has been used to prepare many novel materials, such as nanodiamond and related nanocrystals, metallic nanocrystals, nanocrystal alloys and metal oxide [6]. In this method, the target is immersed in liquid solution which may also contain a suitable surfactant to stabilize the solution. Kitazawa and co-workers successfully prepared carbon particles with sizes of 1-10 μm by the Q-switched Nd:YAG laser ablation of graphite in isopropyl alcohol [7]. The Nd:YAG laser emits a laser light with a wavelength and pulsed width of 1064 nm and 3.5 ns, respectively. Moreover, Yang and co-workers recently synthesized nanocrystalline diamond by laser ablation of graphite target in water with nanosecond pulsed laser [8].

In this study, the carbon nanoparticles were synthesized by laser ablation technique of graphite target in distilled water. The effect of laser energy was reported. The carbon nanoparticles were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and Fourier transform infrared spectrometer (FT-IR).

Experimental

Synthesis of carbon nanoparticles. Carbon nanoparticles were obtained from laser ablation of a solid piece of graphite (carbon disk), as shown in Fig. 1(b). The solid piece of graphite was obtained from pressing the graphite micropowders, Fig. 1(a), under an isobaric press. The consolidation pressure is 100 bars.

The Nd:YAG laser (MIYACHI : ML-2030B) was used in this study with the wavelength of 1064 nm. The laser output energies were ranged from 0.5 to 30 J/pulse. The pulse repetition rates were ranged from 1 to 10 pulse per second (pps). The pulse durations were varied from 0.3 to 10 ms. The graphite disk was placed inside a glass vessel and filled with distilled water until the water is 0.5 cm above the disk. The graphite disk was ablated using Nd:YAG laser, focused by a 6.5 cm focal-length plano-convex lens on the surface of graphite target. Laser energies, employed in this study, were 1.0, 1.5, and 2.0 J/pulse. Pulse repetition rate and pulse duration were fixed at 1 pps and 1 ms, respectively. After 5 min of laser ablation, the nanoparticles were collected from the suspension.



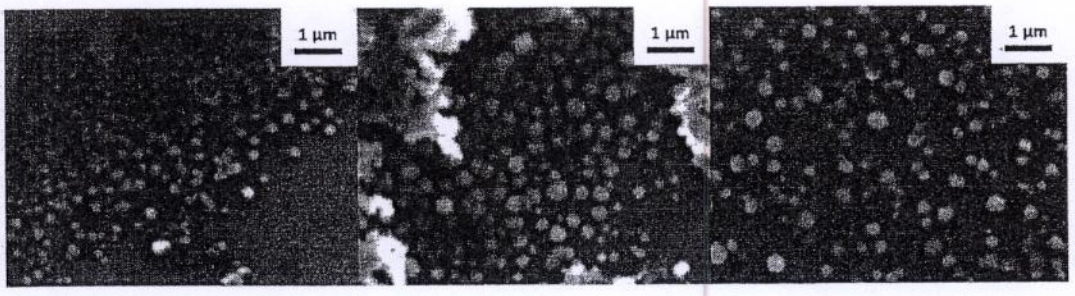
a) b)

Figure 1. a) The SEM image of graphite micropowder
b) The image of solid piece of graphite after pressing

Characterization. The morphology and size of the carbon nanoparticles were investigated using scanning electron microscopy (SEM, JEOL-JSM 6510) by droping the suspension onto silicon substrate and drying on hotplate at the temperature of 60°C. Fourier transform infrared spectroscopy (FT-IR) was used to identify the functional group. The crystalline sized was calculated by Scherrer's equation from XRD pattern.

Result and discussion

Morphology and size of carbon nanoparticles, synthesized with different laser energies, are shown in Fig. 2.



a) b) c)

Figure 2. SEM images of carbon nanoparticles synthesized with different laser energies:
(a) 1.0 J/pulse, (b) 1.5 J/pulse, (c) 2.0 J/pulse

The carbon nanoparticles were nearly spherical shape with average size about 300-400 nm after investigating by SEM image. Fig. 3 shows size distributions of carbon nanoparticles synthesized with different laser energies. They exhibit the broad size distribution ranging from less than 100 nm to about 600 nm in diameter. Moreover, the mean size of particle is bigger for higher energy.

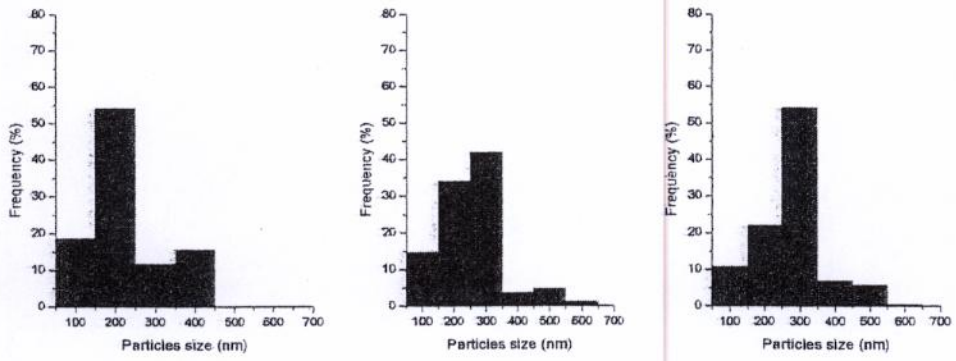


Figure 3. Size distributions of carbon nanoparticles synthesized with different laser energies (a) 1.0 J/pulse, (b) 1.5 J/pulse, (c) 2.0 J/pulse

In order to understand microstructure of the graphite target and the nanoparticles, X-ray diffraction analysis was performed. The XRD patterns are reported in Fig. 4. The graphite target exhibits the typical features of the hexagonal carbon (JCPDS Card 89-8487), i.e., two intense peaks at $2\theta = 26.69°$ and $2\theta = 54.80°$. After laser ablation with different laser energies, the phases shift to $2\theta = 27.12°$ and $2\theta = 55.19°$, respectively.

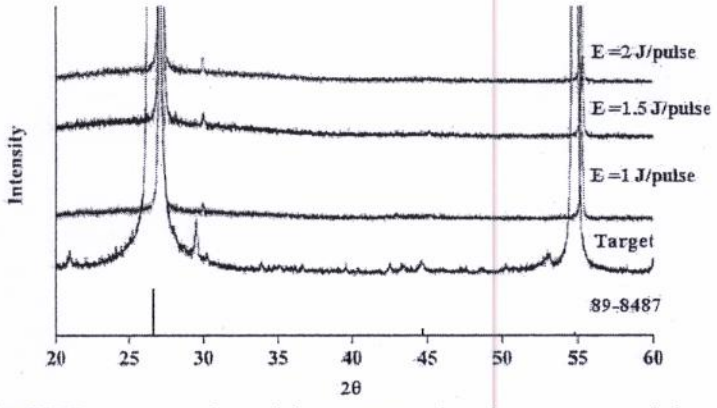


Figure 4. The XRD pattern of graphite target and carbon nanoparticles obtained from different laser energies: 1, 1.5, and 2 J/pulse

Also, the XRD pattern can be used for predicting the size of crystalline using Scherrer's equation as shown in Table 1. The general form of Scherrer's equation is $d = \frac{K\lambda}{B\cos\theta_B}$, where $0.89 \leq K \leq 1.39$. In this study, we use K = 1. Table 1 shows the crystalline sizes predicted by Scherrer's equation. However, the sizes from the calculation are smaller than those obtained from SEM, which indicates that the particles are polycrystallines.

Table 1. The crystalline size of carbon nanoparticles predicted by Scherrer's equation

Laser energy (J/pulse)	θ_B (°)	FWHM (rad)	d (nm)
1.0	27.12066	0.00186	92.81
1.5	27.14782	0.00182	95.16
2.0	27.13659	0.00202	85.59

The FT-IR spectra (Fig.5.) were used to identify the functional groups of the carbon nanoparticles at the laser energy of 2.0 J/pulse. The O-H stretching (3,400-3,450 cm^{-1}) and C-OH stretching vibration (1,020-1380 cm^{-1}) were observed. In addition, the peak located at 1,630 cm^{-1} could be assigned to C=C stretching vibration of aromatic ring.

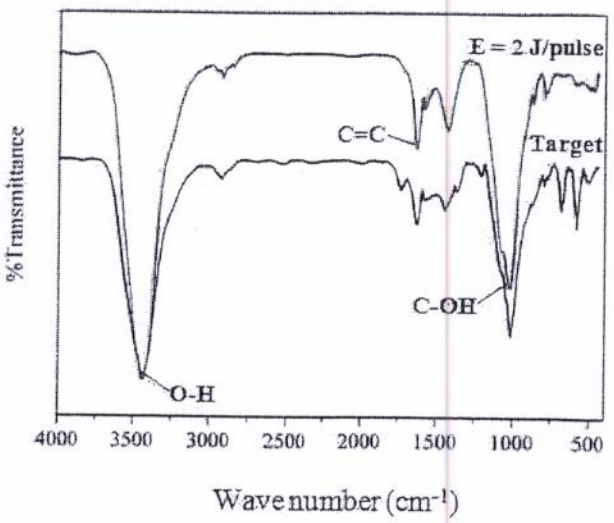


Figure 5. FT-IR spectra of the graphite target and carbon nanoparticles synthesized at laser energy of 2 J/pulse

Conclusion

The carbon nanoparticles were synthesized by laser ablation in distilled water at different laser energies. SEM, XRD and FT-IR spectra are use to investigate. The results show that the laser energy affects size of the particles. The particles size is less than 100 nm after predicting by XRD pattern.

Acknowledgment

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