

CHAPTER 3 MATERIALS AND METHODS

3.1 Synthesis of alumina nanoparticles

3.1.1 Preparation of aluminum target by isobaric pressing

The aluminum powder (99.999% purity, American elements, US) and average size of 30 μm was pressed by a hydraulic with a pressure of 100 bars to obtain the aluminum pellet with a diameter of 15 mm and 4 mm thick. Figure 3.1 show the photograph of aluminum powders and aluminum pellet, respectively.



Figure 3.1 Photograph of aluminum powder and aluminum pellet

3.1.2 Nd:YAG laser

The Nd:YAG laser (MIYACHI : ML-2331B) was used in this study with a wavelength of 1064 nm. The laser output energies were ranged from 0.3 to 50 J/pulse. The pulse repetition rates were ranged from 1 to 50 pulses per second (pps). The pulse durations were varied from 0.1 to 20 ms. Figure 3.2 shows photograph of the Nd:YAG laser used in this study.



Figure 3.2 Photograph of the Nd:YAG laser used in this study

3.1.3 Laser ablation of aluminum target in deionized water with different conditions

In this study, the aluminum pellet was placed at the bottom of a glass vessel, filled with a liquid until its level was approximately 5 mm above the target surface. The laser used for ablation was Nd: YAG laser (Miyachi, ML-2331B), equipped with a 5 cm focal-length plano-convex lens. Therefore, the focusing lens was held at a position of 5 cm above the aluminum target surface. The experimental setup of the laser ablation is shown in Figure 3.3. In present study, the laser ablation of aluminum powder was carried out in deionized water. Flashlamp voltage employed for laser ablation in this study was 300, 400 and 500 V with 1, 3, 5 and 7 J/pulse in each voltage. Pulse repetition rates are 2, 10 and 18 Hz. The number of laser pulses for ablation was 5000 pulses. After laser ablation, all prepared particles dispersed in deionized water were dropped on silicon substrates and dried on an oven at 40 C for 2 hour. The dried nanoparticles samples were then obtained. The morphology of nanoparticles was investigated by field emission scanning electron microscope (Hitachi, S4700).

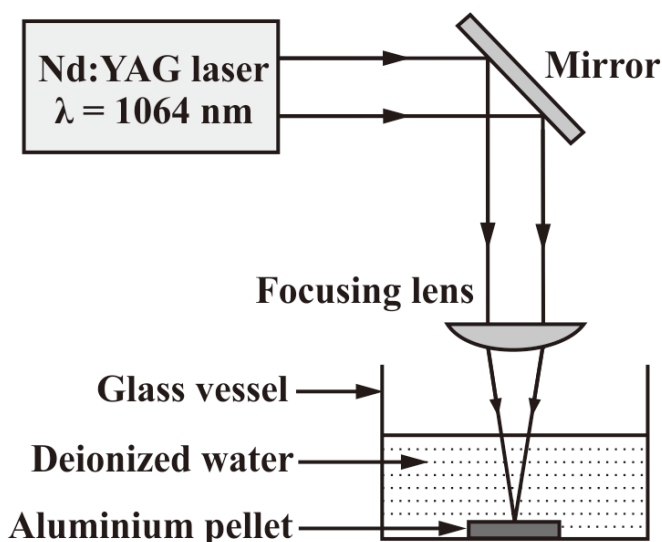


Figure 3.3 Experimental setup for synthesizing Al_2O_3 nanoparticles by laser ablation of Al in deionized water.

3.2 Sample characterization

3.2.1 Field emission scanning electron microscope (FE-SEM) and energy dispersive spectroscopy (EDS)

The morphology of the synthesized nanoparticles were investigated with field emission scanning electron microscopy (FE-SEM, Hitachi, S-4700) with 1.5 nm resolution and accelerating voltages of 5-15 kV. The chemical compositions of synthesized nanoparticles were carried out by energy dispersive spectroscopy (EDS2006, 550i). The samples were mounted onto metal stub with double-sided carbon tape and coated with platinum. The photograph of FE-SEM and EDX are shown in figure 3.4



Figure 3.4 Field emission scanning electron microscope (FE-SEM)

3.2.2 X-ray diffractometer (XRD)

The crystalline phase of the synthesized carbon nanoparticles were identified by X-ray diffraction (XRD, PANalytical's X'Pert PRO) using CuK radiation ($\lambda = 0.15418$ nm) over the 2θ ranges of 20° - 60° . The XRD data were recorded with a resolution of 0.01° . The operating voltage and current are 40 kV and 30 mA, respectively. The photograph of XRD is shown in figure 3.5.



Figure 3.5 X-ray diffractometer

3.2.3 UV-visible spectrophotometer

The prepared carbon nanoparticles dispersed in each liquid were used for the optical absorption measurements. The optical absorption spectra in the wavelength range 190 to 700 nm were recorded at room temperature using a UV-visible spectrophotometer (Jasco, V570). After setting the deionized water as background, a small amount of the collected suspension (containing ablated alumina nanoparticles) was decanted into a quartz cuvette with a path length of 10 mm. The photograph of UV-visible spectrophotometer is shown in figure 3.6.



Figure 3.6 UV-visible spectrophotometer

3.2.4 X-ray photoelectron spectroscopy (XPS)

The chemical composition of sample surface was investigated by X-ray photoelectron spectrometer (XPS; AXIS ULTRA^{DLD}, Kratos analytical, Manchester UK.) The base pressure in the XPS analysis chamber was about 5×10^{-9} torr. The samples were excited with X-ray hybrid mode 700x300 μm spot area with a monochromatic Al $K_{\alpha 1,2}$ radiation at 1.4 keV. X-ray anode was run at 15kV 10mA 150 W. The photoelectrons were detected with a hemispherical analyzer positioned at an angle of 45° with respect to the normal to the sample surface. The photograph of XPS is shown in figure 3.7

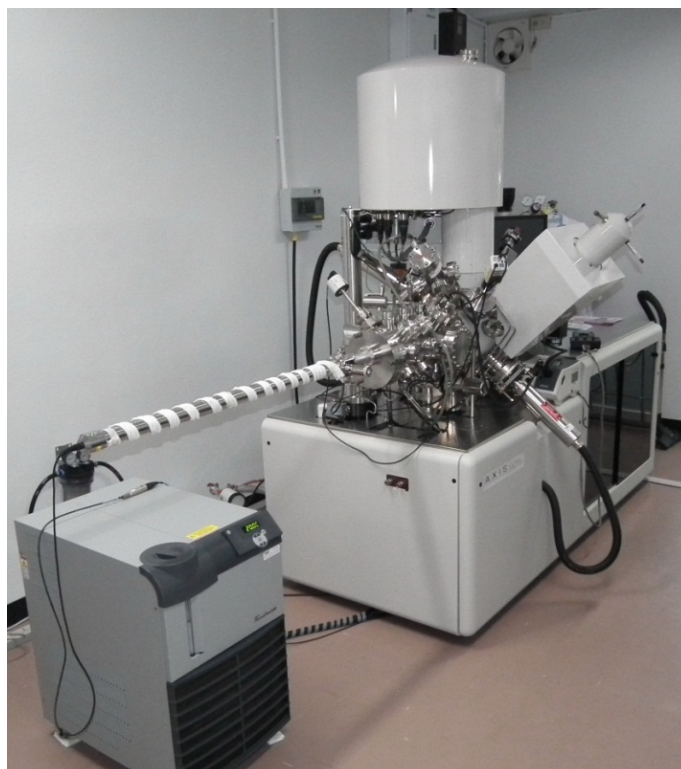


Figure 3.7 X-ray Photoelectron Spectroscopy