

## Research Article

## Production of Alumina Nanoparticles ( $\text{Al}_2\text{O}_3$ ) using Pulsed Laser Ablation Technique in Distilled Water Solution

Adel K. Mahmoud<sup>A\*</sup><sup>A</sup>Mechanical Engineering Department, College of Engineering, University of Diyala , IraqAccepted 10 January 2014, Available online 01 February 2014, **Special Issue-2, (February 2014)**

### Abstract

*Pulsed Laser Ablation in Liquid (PLAL) has become an increasingly important technique for metals production and metal oxides nanoparticles (NPs). This technique has its many advantages compared with other conventional techniques (physical and chemical). This work was devoted for production of alumina ( $\text{Al}_2\text{O}_3$ ) nanoparticles via PLAL technique from a solid alumina target immersed in Distilled Water (D.W.) at different values of laser fluences in order to study the effect of laser fluences on the optical properties and structure of  $\text{Al}_2\text{O}_3$  nanoparticles. The controllability of particle size and size distribution is shown in this paper to be dependent upon laser fluences and it proved that the ablation at lower fluence led to the creation of smaller nanoparticles, smaller aggregates of nanoparticles, and a lower concentration of nanoparticles in contrast an increase of fluence leads to the formation of larger nanoparticles and most of these NPs were aggregated. The produced NPs were characterized by mean of many tests such as UV-visible (UV-Vis.), Atomic Force Microscope (AFM) and Scanning Electron Microscope (SEM).*

**Keywords:** Alumina nanoparticles, Pulsed laser ablation technique, Distilled water solution.

### 1. Introduction

The properties and behavior of materials at nano levels differ greatly when compared to micro levels. Also these nanoparticles show great differences of their outstanding properties such as physical, chemical, optical and electronic properties from the bulk material of which they are made (Piriyawong, *et al*, 2012).

PLAL represents one of the most important, effective and simple technique for preparing metal, metal oxide nanoparticles. PLAL has many advantages compared with other conventional physical and chemical methods like purity, stability of the fabricated nanoparticle colloids, and do not require a vacuum chamber. It is the most flexible and promising technique because of its ability to control NPs size by optimizing the laser parameters, Also this technique provides the possibility of generating a large variety of NPs those are free of both surface-active substances and counter ions (Liu, *et al*, 2009, Krishna, *et al*, 2012, Viau, *et al*, 2011).

In general, the produced NPs through PLAL technique pass through three fundamental steps. Firstly plasma generates due to extreme heating during the interaction of laser with matter. Secondly the plasma, containing vapor of target atoms expands adiabatically, this leads to quick cooling of the plume region and hence to the formation of nanoparticles clusters. Finally after plasma extinguishing the formed nanoparticles clusters encounter and interact with the solvent and surfactant molecules in the

surrounding solution, typically about a few microseconds, all these steps take place and nanoparticles are synthesized (Baladi, 2012, Arash, *et al*, 2010).

Laser ablation in liquid media has been occurred either in nanosecond (ns) or femtosecond (fs) laser pulses. Femtosecond laser ablation (FLA) is one of the best methods to generate free nanoparticles with unique properties such narrower size distribution and with reduced porosity (Arash, *et al*, 2010). FLA can be considered as isochoric process since the irradiation of laser pulse causes local heating in short time and it lasts before expansion of metal takes place (Krishna, *et al*, 2012).

Alumina or aluminum oxide one of important metal oxides has many interesting properties such as high hardness, high stability, high insulation, and transparency. Thus, because of these properties, it can be used in various applications e.g. fire retard, catalyst, insulator, surface coating, composite materials, thermal protections etc (Lukić, *et al*, 2009).

Many researchers direct their researches towards studying the effect of laser parameters on reducing particles sizes and collides stabilities and co-workers and later Tsuji and co-workers have shown that the formation efficiencies and size of particles were changed with power of laser pulses. Recently, Sajti and co-workers found that laser ablation in water yields a greater material removal rate than in air (Al-Mamun, *et al*, 2009).

In this work, alumina nanoparticles were produced by using pulsed laser ablation technique and study the effect

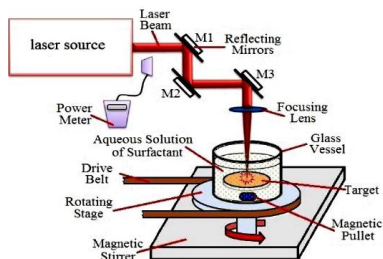
\*Corresponding author: **Adel K. Mahmoud**

of laser fluences on the size and size distribution of NPs in D.W., size and morphology of the nanoparticles were investigated by AFM and SEM. Their optical properties were examined by using UV-visible spectroscopy.

**2. Experimental work**

The experiment arrangement for the synthesis of colloidal solution of nanoparticles by using pulsed laser ablation in aqueous media was carried out with a pulsed Ti/Sapphire laser beam (Quadronix IntenC laser) Kocaeli University-Laser Technologies Research and Application Center. The laser operates at 1 kHz repetition rate with a pulse width of  $\leq 130$  fs at 2.5 mJ/pulse maximum laser beam output.

Figure 1 shows the schematic diagram of PLAL experiment setup for synthesis colloidal solution of alumina NPs. The experiments of producing alumina NPs were done at 90 min exposure time at 800 nm wavelength, 10 Hz pulse repetition rate and 130 fs pulse duration at different values of fluences. This laser beam is focused via a 100 mm focal length focusing lens to a minimum spot size at a solid alumina target (purity 99.99%). The alumina target was fixed by a fixture inside the flask and immersed at 10 mm depth in the solution inside the flask. A well designed and fabricated rotator mechanism used to rotate the flask in order to maintain continuous changing of the focused laser spot position at the target. A magnetic stirrer rotator was placed in the solution rotates at 600 r.p.m. to ensure uniform irradiation on target and the movement of water that can enhance ablated particles diffusion also to disperse the produced NPs. Laser power was measured via a power meter type Newport 841-PE, the measurement was obtained at two locations very near to the final stage of the laser apparatus and before the focusing lens to evaluate the losses of the power in the beam delivery unit. Before starting the experiment the alumina target was cleaned by ultrasonic cleaning device type EMAG 50 HC then wiped with acetone and ethanol solvents. D.W. and ethanol were used as a wet environments. A number of tests were done to characterize the produced alumina NPs at different values of laser fluences, before doing the tests the sample was placed in the ultrasonic cleaner to ensure the homogeneity of the NPs solution. UV-visible extinction spectrum of the colloidal solutions was recorded using a spectrophotometer type Varian Cary-50 UV-Visible. NPs Size, morphology and distribution were examined by SEM imaging device type Tescan VEGA series and AFM test type AA3000 Scanning Probe Microscope is most popular model.



**Fig.1** Mapping Experimental set up of femtosecond laser ablation method (Adel, et al, 2013).

**3. Results and discussion**

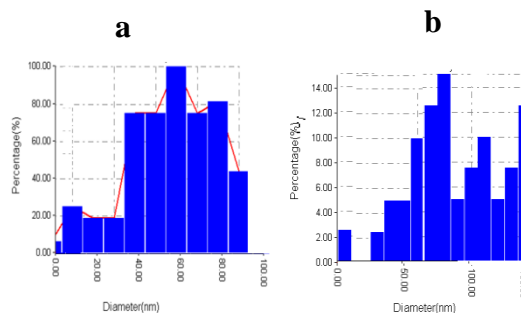
Table 1 shows the effect of laser fluences and the effect of ablation medium on domain particle size and UV-Visible absorption peak wavelength(nm) in D.W. and ethanol by using femtosecond laser ( $\lambda=800$  nm mJ/pulse,  $\tau =130$  fs ,P.R.R =1KHz) for  $Al_2O_3$  NPs.

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Sample Code	Ablation medium	Fluences J/cm <sup>2</sup>	Domain size of NPs	UV-Visible Absorption peak wavelength (nm)
L1	D.W	0.462	60	210
L2	D.W	0.83	75	225

Laser fluence, plays an important role in ablated particles shape, size, structure, phase, and ablation efficiency, the laser ablation of the alumina rod was performed in water at fluences of 0.462 J/cm<sup>2</sup> and 0.83 J/cm<sup>2</sup> corresponding in samples (L1 & L2).

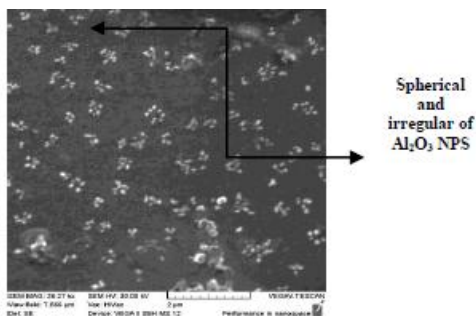
Figure 2 shows a histogram of nanoparticles produced by ablation at 0.462 J/cm<sup>2</sup> and 0.83 J/cm<sup>2</sup> laser fluences respectively. The histogram was normalized to represent the particles have a size ranging from 10-150 nm depending on the laser fluences. From figure 2 the dominant sizes of the particles obtained from 0.462 J/cm<sup>2</sup> and 0.83 J/cm<sup>2</sup> are 60 and 75 nm, respectively. At 0.462 J/cm<sup>2</sup> of laser fluences offered the smallest dominant particle size, whereas that of the 0.83 J/cm<sup>2</sup> yielded the largest size, this evidence that the ablation at lower fluence led to the creation of smaller nanoparticles, smaller aggregates of nanoparticles, and a lower concentration of nanoparticles in contrast an increase of fluence to 0.83 J/cm<sup>2</sup> lead to the formation of larger nanoparticles. However, the size of the particles in sample L2 is not uniformed and most of them were aggregated.



**Fig. 2** Histogram of nanoparticles formed by ablation  $Al_2O_3$  in water at a) 0.462 and b) 0.83 J/cm<sup>2</sup> laser fluences

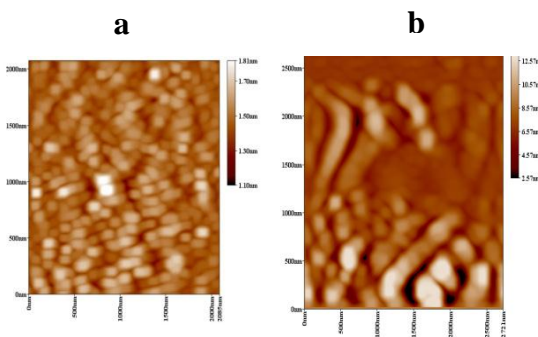
More details information about the size distribution of  $Al_2O_3$  NPs were obtained by SEM and AFM images as shown in figure 3 and figure 4. Figure 3 shows the SEM images of NPs at low fluence 0.462 J/cm<sup>2</sup>, both nanoparticles and submicron particles present with both

spherical and irregular shapes, the mechanical defragmentation may be played an important role in this regime.



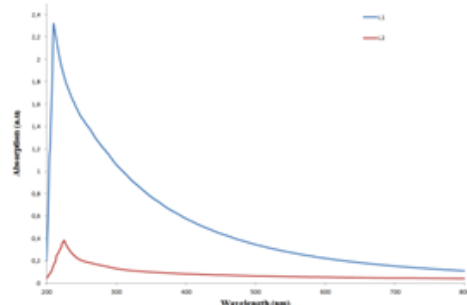
**Fig.3** SEM image of nanoparticles formed from ablation of Al<sub>2</sub>O<sub>3</sub> in D.W. at 0.42 J/cm<sup>2</sup> laser fluence

Figure 4 shows AFM images of an area at a distance of ~ 50 nm from the edge of an ablated crater (for samples L1 & L2), It can be seen the average size and the shape of nanoparticles differ between these two samples.



**Fig. 4** AFM images of Al<sub>2</sub>O<sub>3</sub> NPs produced in water solution at a) 0.462 and b) 0.83 J/cm<sup>2</sup> laser fluences.

Figure 5 shows the absorption spectra of alumina nanoparticles in water at different laser fluences. The difference in the absorption spectra between two samples may be due to the difference in the concentration of nanoparticles in suspension. According to Beer-Lambert law, the absorbance relies upon concentration of the suspension, thus higher concentration of nanoparticles leads to higher value of the absorbance (Piriyawong, et al, 2012), therefore it showed a strong absorption in the UV range with the maximum absorbance at around 210 nm at 0.462 J/cm<sup>2</sup> laser fluences but in 0.83 J/cm<sup>2</sup> laser fluences is 225 nm.



**Fig. 5** shows the absorption spectra of alumina nanoparticles in water at different laser fluences.

**4. Conclusions**

In summary, we have successfully demonstrated that alumina nanoparticles with a size of less than 100 nm can be easily produced via laser ablation in distilled water and different nanostructure formation at different values of laser fluences.

- 1) The results indicate that lower laser fluences results in higher ablation efficiency and finer spherical nanoparticles in contrast an increase of fluence leads to the formation of larger nanoparticles and most of these NPs were aggregated.
- 2) Too high fluences, may cause a coalescence or melt of the adjacent particles, which may be not suitable for some applications requiring individual particles and too low energy may have problems with a large fragment of alumina that did not ionized by the laser beam.
- 3) This convenient synthesis strategy can be applied as a general approach that Al<sub>2</sub>O<sub>3</sub> NPs have attracted significant interest of materials scientists and physicists due to their special properties and have attained a great importance in several technological applications.

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