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Study the Effect of Different Liquid Media on the Synthesis of Alumina (Al_2O_3) Nanoparticle by Pulsed Laser Ablation Technique

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Abstract This work was devoted for production of alumina (Al_2O_3) nanoparticles via PLAL technique from a solid alumina target immersed in different solutions Distilled Water (D.W) as well as in ethanol in order to study the effect of these different solutions on the optical properties and structure of Al_2O_3 nanoparticles. The controllability of particle size and size distribution is shown in this paper to be dependent upon the type of media and it proved that the peak absorption spectrum of samples produced in ethanol is lower than that produced in water. Thus it implies that the ablation efficiency in ethanol is lower, but in the same time it ensures that its oxides are not formed in ethanol due to prohibition of ethanol surrounding media form oxidation. The produced NPs were characterized via many tests such as UV-visible (UV-Vis.) and Atomic Force Microscope (AFM).

Keywords Alumina Nanoparticles, Pulsed Laser Ablation, Different Liquid Media

1. Introduction

The properties and behavior of materials at nano levels have special properties, 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 [1].

Pulsed Laser Ablation in Liquid (PLAL) has become an increasingly important technique for production metals and metal oxides nanoparticles (NPs) and others. This technique has its many advantages compared with other conventional techniques (physical and chemical) such as 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[2-5].

The mechanisms of producing NPs can be summarized in 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 cool of the plume region then form nanoparticles clusters. Finally after plasma extinguishing the formed nanoparticles clusters encounter and interact with the solvent and surfactant molecules in the surrounding solution, typically all these steps take place and nanoparticles are synthesized about a few microseconds [6,7].

Using femtosecond laser in this technique can be considered the best methods to generate free nanoparticles with unique properties such narrower size distribution and with reduced porosity [3].

Alumina or aluminum oxide is one of important metal oxides, it 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. drug delivery, catalyst, insulator, surface coating, composite materials, thermal protections etc [1, 8].

The type of liquid media plays an important role in the nature of produced nanoparticles, the effect of the solvent on nanoparticle formation is further shown by comparison of the size distribution of various solvents, The solvent molecules form a shell around produced NPs which prevents these nanoparticle from undergoing further reaction. The presence of this solvent shell also serves to solvate the nanoparticles, allowing them to remain suspended in solution for an indefinite period of time therefore many researchers direct their researches towards studying the effect of a surfactant type on reducing particles sizes and collides stability [9, 10]. Ogale and co-workers [11] opened a new route in synthesizing metastable structures using

PLAL technique in various confining liquids.

The work was focused on the study the effect of different liquid media in controlling the size and stability of generated NPs as well as agglomerations reduction. D.W and ethanol were used as ablation environment to capture and store the synthesized NPs. both ethanol and D.W media play a crucial role in the nature of produced Al_2O_3 NPs, ethanol is proposed to be an optimal substitute of D.W. for fabricating pure materials colloid because of the ethanol prohibition to form oxidation. While that the ablation efficiency in ethanol is lower than D.W because of lower absorption spectra of ethanol indicates lower abundance of NPs in solution. Size and morphology of the nanoparticles were investigated by AFM, Their optical properties were examined using by UV-visible spectroscopy.

2. Experimental Procedure

Figure 1 shows the schematic diagram of PLAL experiment setup for synthesis colloidal solution of alumina NPs in different aqueous media, these nanoparticles were 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 ≤130 fs at 2.5 mJ/pulse maximum laser beam output, 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 rpm to ensure uniform irradiation on target and the movement of water that can enhance ablated particle 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. The alumina target was cleaned by ultrasonic cleaning device type EMAG 50 HC then wiped with acetone and ethanol solvents before starting the experiment. D,w and ethanol were used as a wet environments. A number of tests were done to characterize the produced alumina NPs at different liquid media, before doing these 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.

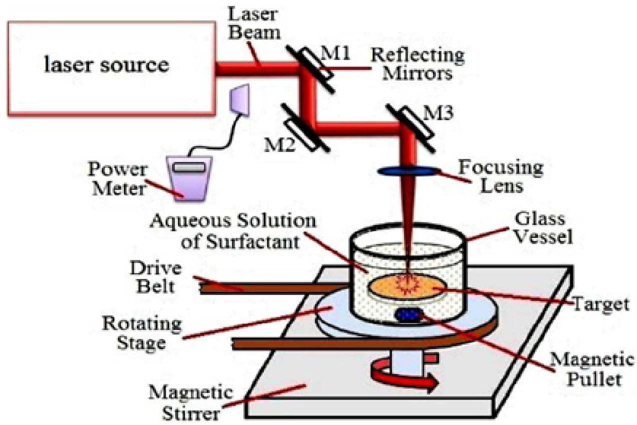


Figure 1. Mapping Experimental set up of femtosecond laser ablation method [12]

3. Results and Discussions

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

During laser ablation of alumina in ethanol small bubbles can be observed in the solution, while in water no bubbles were observed. At high pressure and high temperature, ethanol can decompose to form permanent gases. The formed permanent gases in ethanol solution aggregate to bubbles that can be seen during laser ablation. The gases bubbles in the path of laser beam in combination with ablated plasma and formed nanoparticles in earlier pulses weaken the laser light that couples to the target. It suggests that no oxidation occurs under the protection of ethanol solution. Therefore, ethanol is proposed to be an optimal substitute of water for fabricating pure materials colloid due to prohibition of ethanol surrounding media form oxidation.

Table 1. shows the effect of ablation medium on domain size and UV-Visible absorption peak wavelength(nm) by using femtosecond laser (λ=800 nm mJ/pulse, τ =130 fs ,P.R.R =1KHz) for Al_2O_3 NPs

Sample Code	Ablation medium	Fluences J/cm^2	Domain size of NPs nm	UV-Visible Absorption peak wavelength (nm)
L1	D.W.	0.83	75	225
L2	Ethanol	0.83	80	213

Figure 2 shows uv-visible absorption spectra of these samples, the absorption peak of sample synthesized in water is 225nm, but the one synthesized in ethanol is 210 nm. It is found that the absorption spectrum of samples produced in ethanol is lower than that produced in water. The lower of absorption spectra indicates lower abundance of particles in solution. Thus, it implies that the ablation efficiency in ethanol is lower. Absorption peak of colloidal solutions on around 210 nm wavelength discloses that products are alumina particles and its oxides are not formed due to prohibition of ethanol surrounding media form oxidation.

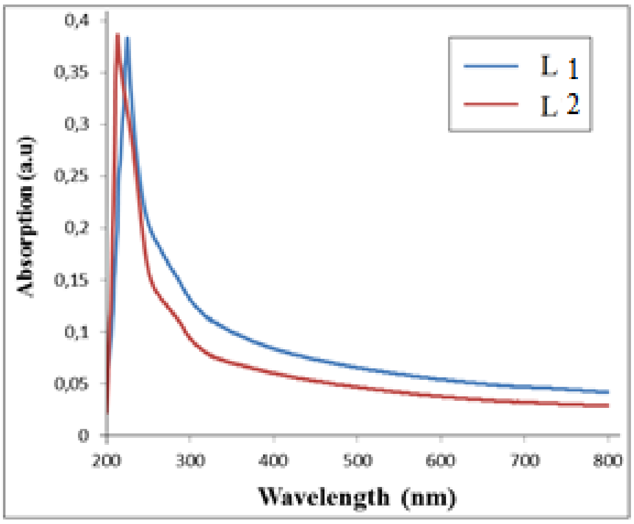
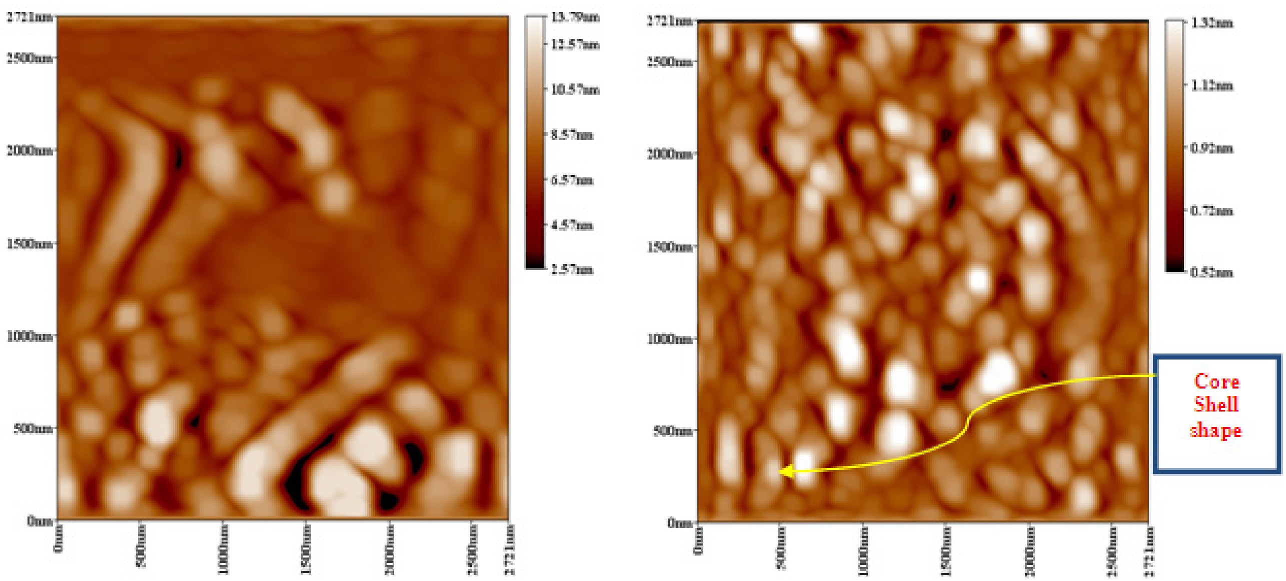


Figure 2. UV-Visible absorption spectra of Al_2O_3 NPs prepared in two media Water (L1)r and Ethanol (L2)

AFM photograph of nanoparticles of sample L1 and L2 is presented in **Figure 3**, which certifies the presence of some particles in L2 sample with core–shell structure, which are mostly larger than other nanoparticles corresponding in sample L1. In general, there are two different opinions about characterization of this structure. According to Kazakevich et al. [13], this particular structure can be assigned to carbon coated nanoparticles, which consisted of metallic nanoparticles as the core and layer of amorphous carbon as the shell, the carbon atoms of which have been released from decomposition of ethanol. On the other hand, Stratakis et al. [14] reported the presence of core– shell structure belong to that most of alumina nanoparticles are amorphous and the core may be crystallized Al_2O_3 if it is dark area or may be pore if it is bright area. These might bring about dark grayish color of colloidal solutions synthesized in ethanol and could lead to rapid bonding between carbonic surfaces of the nanoparticles, which precipitate as grayish sediments.



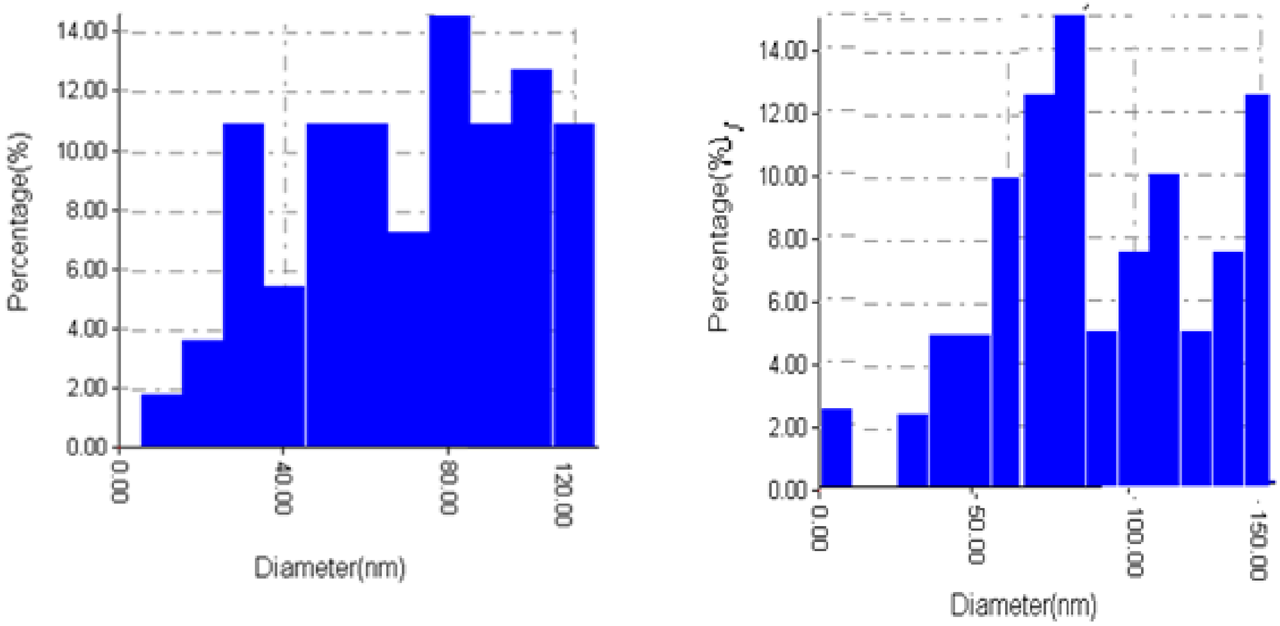
(a) (b)

Figure 3. AFM images of Al_2O_3 NPs produced in a) water and b) ethanol.

Figure 4 shows a histogram of nanoparticles produced by ablation at water and ethanol media respectively. The histogram was normalized to represent the particles have a size ranging from 10-150 nm depending on the nature of liquid. From Figure 4 the dominant sizes of the particles obtained from water and ethanol are 75and 80 nm, respectively. However, the size of the particles in sample L2 is not uniformed and most of them were aggregated.

As mentioned above during laser ablation of alumina in ethanol small bubbles can be observed in the solution. A larger bubble would have a larger volume for interaction, then allowing for longer times of nucleation and easy to form larger nanoparticle a superheated bubble expands until it reaches a critical volume, and after that collapses. The ablation event causes many bubbles to be ejected normal to the surface of the rod but the collapse is not proficient by all bubbles, as there are some that reach the surface. Based upon these results, the bubbles that survive to reach the surface contain a mixture of volatile gases such as H_2, CO, CO_2, as well as small hydrocarbon species. The gases are formed as a direct result of the high energy from the laser irradiation, which is sufficient to break the carbon-hydrogen bonds. During collapse, confined species are subjected to extremely high pressures, which may contribute to the formation of the nanoparticles. The collapse of the bubble also effectively binds solvent molecules to the nanoparticles.

These ethanol molecules form a shell which prevents the nanoparticle from undergoing further reaction. Therefore the solvent molecules surrounding the particles serve to protect the formed nanoparticles from oxidation [15-17].



(a) (b)

Figure 4. Histogram of nanoparticles formed by ablation Al_2O_3 in: a) Water and b) Ethanol.

4. Conclusions

In summary, we have successfully demonstrated that alumina nanoparticles with a size of less than 100 nm can be easily synthesized via laser ablation in water and ethanol with less oxide cladding and different nanostructure formation. The results indicate both ethanol and D.W media play a crucial role in the nature of produced Al_2O_3 NPs, ethanol is proposed to be an optimal substitute of D.W. for fabricating pure materials colloid because of the ethanol prohibition to form oxidation. While that the ablation efficiency in ethanol is lower than D.W because of lower absorption spectra of ethanol indicates lower abundance of NPs in solution.

This convenient synthesis strategy can be applied as a general approach that Al_2O_3 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 such industrial applications.

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