

Article

Fabrication and Characterization of Al₂O₃ Nanoparticles Prepared via Pulse Laser Ablation Method

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Abstract

Aluminum oxide (Al₂O₃) nanoparticles were synthesized using the pulse laser ablation method in liquid with a laser energy of 900 mJ . Aluminum (Al) plate was immersed in deionized water and then ablated by an Nd-Yag laser with wavelength 1064 nm. The size, shape, and structural properties of the Al₂O₃ nanoparticles were analyzed using scanning electron microscopy (SEM) imaging and X-ray diffraction (XRD). UV-Vis spectroscopy was employed to determine the optical properties of the prepared nanomaterials. SEM images revealed that the produced nanoparticles had a spherical shape, and the particle size was under 100 nm. XRD analysis showed that the crystallinity of the nanoparticles improved with time. UV-Vis spectroscopy measurements indicated a clear absorption peak at a wavelength of 211 nm, with an energy gap (E_g) value of 5.3 eV. These results suggest that the pulse laser ablation in water technique is a simple, cost-effective, scalable, and environmentally friendly method for synthesizing nanoparticles, particularly for applications in optoelectronics and photocatalysis.

Keywords: Al₂O₃, nanoparticles, pulsed laser ablation method, optical properties.

1. Introduction

Metal oxides have become much more attractive from both scientific and technological perspectives because of their unique properties, which covers nearly every topic of materials science and solid-state physics [1]. A wide range of nanostructured metal oxide materials in one, two, and three dimensions have been used in potential industrial applications such as sensors, biomedical devices, catalysis, piezoelectric devices, energy conversion and storage systems [2]. The small size of metal oxide nanomaterials provides high stability, a high surface-to-volume ratio, a high carrier capacity, high chemical reactivity, and biological activity, which can be beneficial for many applications [3]. Aluminum oxide material is a promising material in different fields of nanotechnology, including electronics, nanomedicine, fuel cells, and corrosion-resistant surface coatings. This is because Al₂O₃ semiconductor nanomaterials exhibit a wide transparency window, excellent mechanical properties, thermal stability, and attractive electrical properties [4]. Nanostructured materials can be formed through many different approaches, such as the hydrothermal method, chemical vapor deposition, the sol-gel technique, sputtering, pulsed laser deposition (PLD) method, and atomic layer deposition. Recently, the laser ablation technique has become the most commonly utilized method in the production of nanoparticles (NPs), which can be made in liquid, vacuum, or gas. Laser ablation in liquid offers various advantages over traditional approaches; it is simple and can be carried out without using chemicals [5-8]. In the present study, pulsed laser ablation in liquids (PLAL) technique was used to deposit Al₂O₃ NPs and study their morphological, structural and optical properties.

2. Experimental

An aluminum plate with a purity 99.99% (2cm x 2cm) was used for Al₂O₃ nanoparticles growth in deionized water (DI) by laser ablation technique. The aluminum metal foils were polished using 4000-grit sandpapers; the samples were then

cleaned in an ultrasonic bath with acetone, ethanol, and deionized water for 10 minutes and finally dried with an air gun. To generate Al_2O_3 nanoparticles, the Al metal targets were immersed in a glass beaker filled with 5 ml of DI water at room temperature, and the liquid medium's height over the Al target surface was 5 mm. As shown in Figure 1, the PLAL process contains a Q-switched N:YAG RG190 laser source with two wavelengths (1064 nm, 532). The experimental operating settings and parameters for the laser ablation in liquid technique were a 1064 nm wavelength, 10 ns pulse duration, 6 Hz repetition rate, and 900 mJ output energy per pulse. A convex lens was employed to achieve high laser flux. In addition, 500 laser pulses were used to bombard the metal target surface. After the laser ablation process, the deionized water solution containing nanoparticles was collected for analysis.

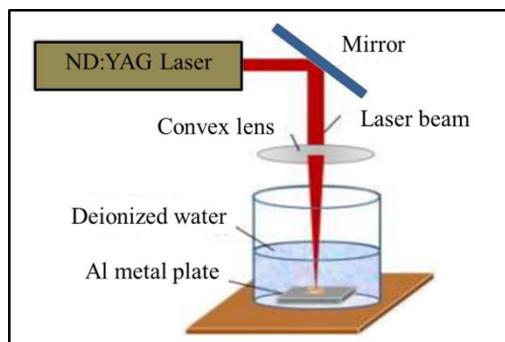


Figure 1: Pulsed laser ablation in liquid system set up.

3. Results and Discussion

3.1 Structural and Morphological Analysis

Figure 1 represents the X-Ray diffraction patterns of the (a) ablated particles and (b) aluminum metal plate. The five observed diffraction peaks can be indexed to (104), (113), (024), (116), and (214) reflections. These observations are match well the same structure reported in [4, 9-13]. The XRD peaks indicate that the $\alpha\text{-Al}_2\text{O}_3$ nanoparticles synthesized by laser ablation in water have a polycrystalline structure. The XRD pattern showed no notable peaks for pure Al metal or different alumina phases, indicating that Al was completely transformed into $\alpha\text{-Al}_2\text{O}_3$ during the laser ablation process [11]. The

XRD pattern for the Al metal substrate in Figure 1(b) shows intense peaks located at $2\theta = 38.70^\circ, 44.94^\circ, 65.25^\circ, 78.41^\circ$, which are attributed to the Al (111), (200), (220), and (311) plans, respectively. These results are similar to previous studies [10, 14, 15].

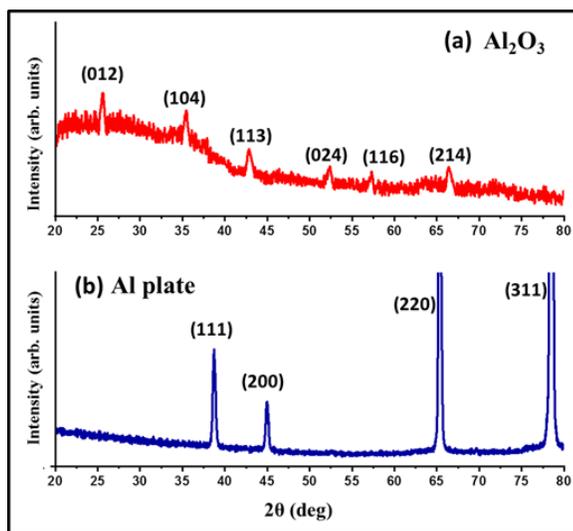


Figure 1. XRD spectra of (a) α - Al_2O_3 nanoparticles and prepared by laser ablation in water method (b) Al metal substrate.

The mean crystallite size of α - Al_2O_3 is calculated using Scherrer equation ($D = K\lambda/\beta \cos \theta$) [13], where β (in rad) is the full width at half-maximum (FWHM) of the XRD peak, ($\lambda = 1.54060 \text{ \AA}$) is the used X-ray radiation wavelength, 0.9 is Scherrer's constant, and θ the Bragg diffraction angle. The estimated average crystallite sizes of α - Al_2O_3 nanoparticles are summarized in the table 1.

Table 1: Average crystal size of α - Al_2O_3 nanoparticles prepared by laser ablation in water method

$2\theta^\circ$ (113)	FWHM (β) deg	Average crystal size (nm)
25.57	0.39	21.8
35.40	0.34	20.3
52.30	0.54	17.1
57.30	0.39	24.2
66.45	0.57	17.4

The morphological characteristics of the ablated particles of Al₂O₃ nanoparticles from Al plate by the laser ablation in water process were investigated using scanning electron microscopy (Thermo Scientific™ Quattro SEM). Figure 2 shows SEM images of (a) the aluminum plate and (b) Al₂O₃ nanoparticles.

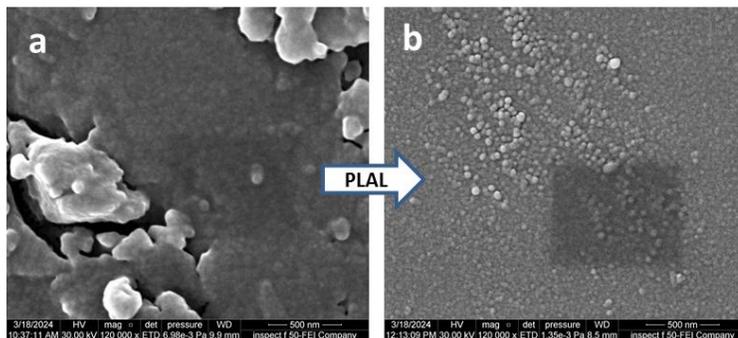


Figure 2. Surface morphology images of the as-grown Al₂O₃ NPs by laser ablation in water technique .

It is obvious that the manufactured Al₂O₃ nanoparticles have a spherical form. From SEM image in figure 1 (b) The particle size distribution ranged from 20 to 65 nm, these results are in good agreement with previous reported studies [12, 16-18].

The Dispersive Spectroscopy (EDS) spectra of Al₂O₃ nanoparticles synthesized using the laser ablation method were represented by Figure 3. EDX analyses confirmed the atomic composition of the prepared structures. The results indicate that the spectra consisted of aluminum (Al) and oxygen (O) peaks. This suggests that the synthesis of pure Al₂O₃ nanoparticles was successful. The spectrum of atomic compositions analysis reveals that the weight percentages of aluminum and oxygen elements were 26.6 and 73.4 atomic respectively.

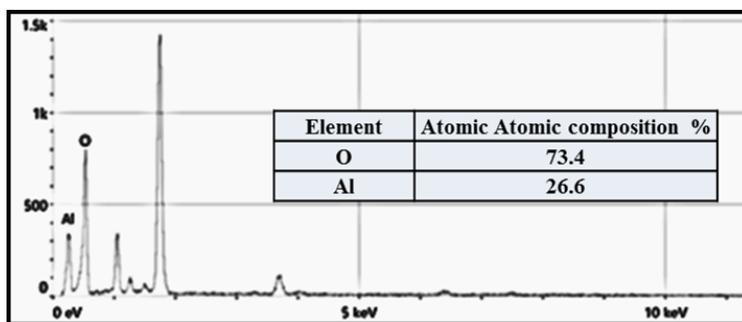


Figure 3. The atomic compositions analyzed by EDS study of Al₂O₃ prepared by laser ablation in water technique .

3.2 Optical Characterization

The UV-Vis spectrometer was used to investigate the optical properties of the Al_2O_3 nanoparticles synthesized using laser ablation in water method. Figure 4 shows the UV-Vis absorbance spectra for Al_2O_3 NPs over the wavelength range of 300 to 700 nm. The results revealed that the Al_2O_3 sample exhibited an absorbance peak at 211 nm [19, 20]. The transmittance spectrum of Al_2O_3 sample in the spectral range 300-700 nm are shown in Figures (5). It is clear that the sample was transparent in the range of 250-700 nm.

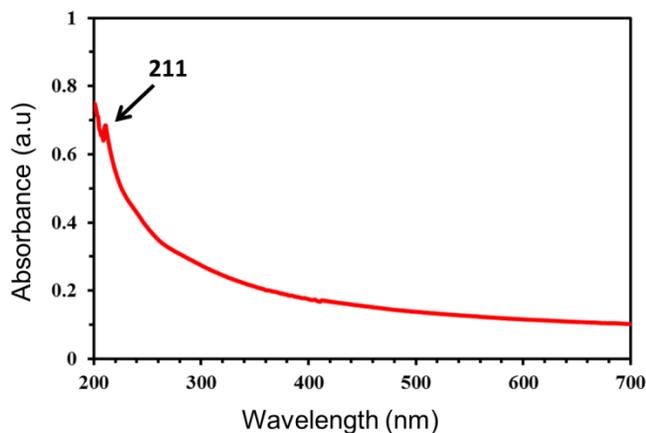


Figure 4. UV-Vis spectra of as-prepared Al_2O_3 nanoparticles

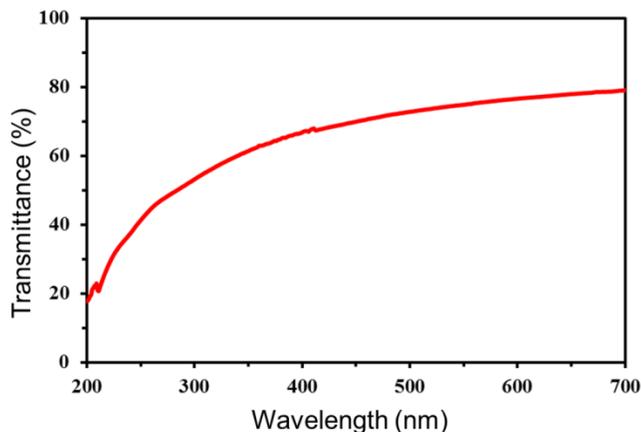


Figure 5. Transmittance spectra of the Al_2O_3 NPs prepared by laser ablation in water technique .

A graphs in Figure 4 shows the optical energy band gap for Al₂O₃ NPs formed by the laser ablation in water process. The linear portion of the $(\alpha h\nu)^2$ vs photon energy ($h\nu$) curves is used to evaluate the Al₂O₃ band gap. The optical band gap E_g was computed using Tauc's equation, as shown in the equation below [21, 22]:

$$\alpha h\nu = A(h\nu - E_g)^n \quad \dots\dots (1)$$

where α is the absorption coefficient, h is Plank's constant, ν is the frequency, and A is a proportionality constant, and n is the exponent factor that characterizes the nature of the electronic transition generating the absorption (1/2 and 2, 3/2 or 3) for direct allowed or indirect allowed, direct forbidden and indirect forbidden transitions, respectively [23]. As shown in figure 4, the band gap. The band gap of the alumina nanoparticles was found to be approximately 5.3 eV, which is in good agreement with previously reported studies by other researchers [24-26].

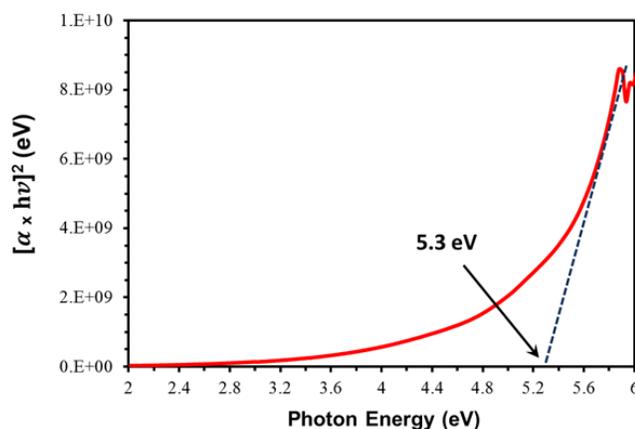


Figure 6. The $[\alpha h\nu]^2$ versus $h\nu$ plots Al₂O₃ NSs.

4. Conclusion

Aluminum oxide nanoparticles were successfully synthesized from an aluminum plate using the laser ablation in water technique. The XRD pattern of the as-formed Al₂O₃ NPs displayed major characteristic peaks of the α -phase. The mean crystallite size of α -Al₂O₃ was found to be in the range of 17- 22 nm. The SEM images revealed the that the as-synthesized products had a spherical shape. The EDX elemental composition analyses confirmed the presence of Al and O as the major elements. The

UV-Vis spectrum indicated that the band gap of the Al₂O₃ sample is approximately 5.3 eV. Given the ease fabrication and the excellent performances of green laser ablation technique, it holds promise for production of other nanomaterials. Additionally, the deposited Al₂O₃ structures produced by PLAL technique are expected to be highly promising in for a range of application, particularly in optoelectronics and photocatalysis.

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5. References

1. Cox, P.A., Transition metal oxides: an introduction to their electronic structure and properties. Vol. 27. 2010: Oxford university press.
2. Jeevanandam, J., et al., Review on nanoparticles and nanostructured materials: history, sources, toxicity and regulations. Beilstein journal of nanotechnology, 2018. **9**(1): p. 1050-1074.
3. Soltaninejad, H., et al., Evaluating the Toxicity and Histological Effects of Al₂O₃ Nanoparticles on Bone Tissue in Animal Model: A Case-Control Study. Journal of toxicology, 2020. **2020**.

4. Oleiwi, H.F., et al. Structural and optical properties of Al₂O₃ nanocrystalline: Effect of deposition time. in AIP conference proceedings. 2019. AIP Publishing.
5. Shi, S., et al., Structural and optical properties of amorphous Al₂O₃ thin film deposited by atomic layer deposition. *Advances in Condensed Matter Physics*, 2018. **2018**(1): p. 7598978.
6. Avci, N., et al., Optical and structural properties of aluminium oxide thin films prepared by a non-aqueous sol-gel technique. *Journal of sol-gel science and technology*, 2011. **59**: p. 327-333.
7. Ahmed, M.I., et al., Synthesis and characterization of γ -Al₂O₃ and α -Al₂O₃ nanoparticles using a facile, inexpensive auto-combustion approach. *Egyptian Journal of Chemistry*, 2021. **64**(5): p. 2509-2515.
8. Ismail, R.A., S.A. Zaidan, and R.M. Kadhim, Preparation and characterization of aluminum oxide nanoparticles by laser ablation in liquid as passivating and anti-reflection coating for silicon photodiodes. *Applied Nanoscience*, 2017. **7**(7): p. 477-487.
9. Bharthasaradhi, R. and L. Nehru. Preparation and characterisation of nanoscale α -Al₂O₃ by precipitation method. in *AIP Conference Proceedings*. 2015. AIP Publishing.
10. López-Juárez, R., et al., Synthesis of α -Al₂O₃ from aluminum cans by wet-chemical methods. *Results in Physics*, 2018. **11**: p. 1075-1079.
11. Bharthasaradhi, R. and L. Nehru, Structural and phase transition of α -Al₂O₃ powders obtained by co-precipitation method. *Phase Transitions*, 2016. **89**(1): p. 77-83.
12. Su, X., S. Chen, and Z. Zhou, Synthesis and characterization of monodisperse porous α -Al₂O₃ nanoparticles. *Applied surface science*, 2012. **258**(15): p. 5712-5715.

13. Kim, H.S., et al., Preparation of Nanosized γ - Al_2O_3 Particles Using a Microwave Pretreatment at Mild Temperature. *Advances in Materials Science and Engineering*, 2012. **2012**.
14. Ayieko, C.O., et al., Controlled texturing of aluminum sheet for solar energy applications. *Advances in Materials Physics and Chemistry*, 2015. **5**(11): p. 458-466.
15. Rashad, M., et al., Effect of graphene nanoplatelets addition on mechanical properties of pure aluminum using a semi-powder method. *Progress in Natural Science: Materials International*, 2014. **24**(2): p. 101-108.
16. Ma, P., et al., Effect of Al_2O_3 nanoparticles as reinforcement on the tensile behavior of Al-12Si composites. *Metals*, 2017. **7**(9): p. 359.
17. Riahi, A., et al., Study of thermal conductivity of synthesized Al_2O_3 -water nanofluid by pulsed laser ablation in liquid. *Journal of Molecular Liquids*, 2020. **304**: p. 112694.
18. Piriya Wong, V., et al., Preparation and characterization of alumina nanoparticles in deionized water using laser ablation technique. *Journal of Nanomaterials*, 2012. **2012**(1): p. 819403.
19. Saadi, N.S., L.B. Hassan, and T. Karabacak, Metal oxide nanostructures by a simple hot water treatment. *Scientific reports*, 2017. **7**(1): p. 7158.
20. Khedir, K.R., et al., Growth of zinc oxide nanorod and nanoflower structures by facile treatment of zinc thin films in boiling de-ionized water. *Journal of Nanoscience and Nanotechnology*, 2017. **17**(7): p. 4842-4850.
21. Mallick, P. and S. Sahu, Structure, microstructure and optical absorption analysis of CuO nanoparticles synthesized by sol-gel route. *Nanoscience and Nanotechnology*, 2012. **2**(3): p. 71-74.

22. Tran, T.H. and V.T. Nguyen, Copper oxide nanomaterials prepared by solution methods, some properties, and potential applications: A brief review. International Scholarly Research Notices, 2014. **2014**.
23. Sabbaghan, M., A.S. Shahvelayati, and K. Madankar, CuO nanostructures: Optical properties and morphology control by pyridinium-based ionic liquids. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 2015. **135**: p. 662-668.
24. Johan, M.R., et al., Annealing effects on the properties of copper oxide thin films prepared by chemical deposition. Int. J. Electrochem. Sci, 2011. **6**: p. 6094-6104.
25. Alqadasy, S.S., et al., Synthesis and Study the Structure, Morphological and Optical Properties For TiO₂-Al₂O₃-La₂O₃ Prepared by Chemical Bath Deposition.
26. Prashanth, P., et al., Synthesis, characterizations, antibacterial and photoluminescence studies of solution combustion-derived α -Al₂O₃ nanoparticles. Journal of Asian Ceramic Societies, 2015. **3**(3): p. 345-351.