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RESEARCH ARTICLE - PHYSICS

Effect of Nanoparticle Size on the Optical Properties and Morphology of Lead Oxide Prepared by Laser Ablation Method

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Abstract

The aim of this study is to prepare lead oxide nanoparticles using a laser ablation method. Nanoparticles are prepared with different pulse numbers, ranging from 100 to 400 pulses. This study focuses on the difference in nanoparticle production with varying pulse numbers. The optical properties were studied using UV-Visible examination, and the results showed that increasing the number of laser pulses leads to an increase in both the optical absorption and the energy gap of lead oxide. Increasing the number of pulses leads to an increase in the average surface roughness of lead oxide. The presence of lead and oxide was confirmed in the FT-spectrum. IR of lead oxide nanoparticles, resulting from laser ablation of a lead target.

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Keywords: Laser ablation, surface morphology, UV-visible, FT-IR, energy gap

1. Introduction

Nanoparticles have a great future and interest in many applications due to their surface area and density. The physical and chemical properties are determined by its shape and size[1]. Nanoparticles have deep overlap and interest in the fields of medicine, energy, and technology [2]. Moreover, the nanoparticles have novel physical properties that differ from both molecular matter and solid matter due to the presence of a large proportion of atoms on their surface. Appropriate preparation techniques can lead to the formation of diverse types of nanoparticles that can be applied in energy and environment. One of the difficulties facing the chemical preparation of nanomaterial's is the emergence of other materials due to side reactions. Which requires processing it in special ways to extract the required distribution[2], [3]. One of these nanoparticles is a lead oxide which an important material that has a unique properties that make it useful for a wide range of applications in various industries such as production of batteries, radiation protection, ammunition, welding materials, outdoor paints, and electronics [4], [5].

There are various methods used to synthesis lead oxide nanoparticles such as chemical, green Synthesis and laser ablation methods [5], [6]. The pulsed laser deposition method provides pollution-free and by-product-free conditions in the preparation of nanoparticles. It provides the greatest possible purity and also allows precise control of particle properties by treating several factors such as the type of material, laser properties, and type of liquid medium [4], [7]. Using the pulsed laser deposition method, many nanoparticles were prepared, such as copper oxide, silver oxide, and many others. Among them is lead oxide, which appears in dark brown color or in the form of a black powder and has an energy gap range between 1.4 to 2.8 eV[6], [8].

Numerous studies have searched at the synthesis and analysis of lead oxide using the laser ablation technique. such as, in 2015, Sharma et al, [9],Investigated the effects of laser pulse duration and energy density on the size and optical properties of PbO nanoparticles. They found that shorter pulse durations and higher energy densities produced smaller nanoparticles with larger band gaps. While in 2016, Krishnan et al[10],studied the effects of laser pulse duration on the properties of PbO nanoparticles synthesized by laser ablation. They found that shorter pulse durations resulted in smaller, more uniform nanoparticles with enhanced optical properties. In 2017, Halah H. Rashed et al [6], reported that the final surface morphology characteristics indicated that the particle size of the nanoparticles decreased with increasing the number of laser pulses, the reason was due to excised particles absorbed the incident laser energy. Thus, it was divided into smaller particles. The optical properties characteristics showed that the absorbance increased with the increase of laser pulses due to an increase in the concentration while the energy gap decreases with increasing the number of laser pulses.

In this study, lead oxide is prepared using the laser ablation technique, and its structural, morphological, and optical properties are investigated by subjecting a sheet of lead to varying numbers of pulses from an Nd:YAG laser after immersing it in an 2-Methoxyethanol solution.

2. Experimental

To synthesize Lead oxide nanoparticles, the pulsed laser ablation technique was used. A lead substrate measuring 6x10 mm with a thickness of 5 mm underwent cleaning using ethanol and distilled water through ultrasonic cleaning methods to eliminate unwanted pollutants from the surface of the lead plate. Subsequently, it was positioned at the bottom of the inner base of a cylindrical glass beaker and filled with an organic solution (2-Methoxyethanol). Moreover, the prepared lead target was then irradiated by an Nd:YAG laser (1064 nm, 4 Hz, 6 ns, 700 mJ) with a different numbers of pulses (100, 200, 300, 400 pulses) to generate colloidal solutions of Lead oxide nanoparticles. The experimental setup for the ablation process is depicted schematically in Figure 1. Furthermore, the characterization of the produced nanoparticles was conducted using AFM, UV-VIS, FT-IR Spectra and electrical conductivity analyses.

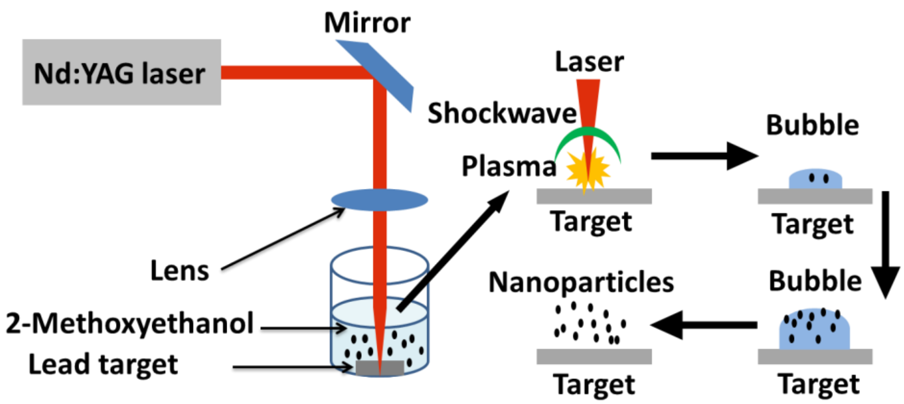

Fig. 1. Setup of the ablation process[11], [12]

3. Results and discussions

3.1. FT-IR studies

Fourier transform infrared can be used to analyze the chemical bonding of lead oxide. The presence of lead and oxide in the FT-IR spectrum of lead oxide nanoparticles, produced by laser extraction of a lead target submerged in 2-Methoxyethanol, is indicated by two distinct and highly pronounced peaks at 833 and 856. The spectrum region of 1000-500 cm-1 exhibited distinct peaks for 100, 200, 300, and 400 pulses, which corresponded to the distinctive vibrations of PbO. The presence of the transmission peak at 538 cm-1 shows the occurrence of the (PbO) stretching vibration mode. The peaks at 1120 and 1192 cm-1 can be attributable to the –CH3 rocking modes of the methoxy group1.The band observed at a wavenumber of 1235 cm-1 can be attributed to methyl deformation, as indicated by references [5] and [13]. The detection of pb-OH was confirmed by the absorption peak at 1659 cm-1, which corresponds to the bending vibration of pb-OH. The peaks seen at 2930, 2877, and 2820 cm-1 corresponded to the stretching vibration of the –CH3 group. The prominent peak at around 3377 cm-1 corresponds to the stretching vibrations of the (O-H) bonds, which arise from the presence of a minor quantity of ethanol utilized during the sample washing process [5]. The presence of metal-oxygen (M-O) stretching vibrations was detected by the appearance of peaks in the 500-900 cm-1 range [14].

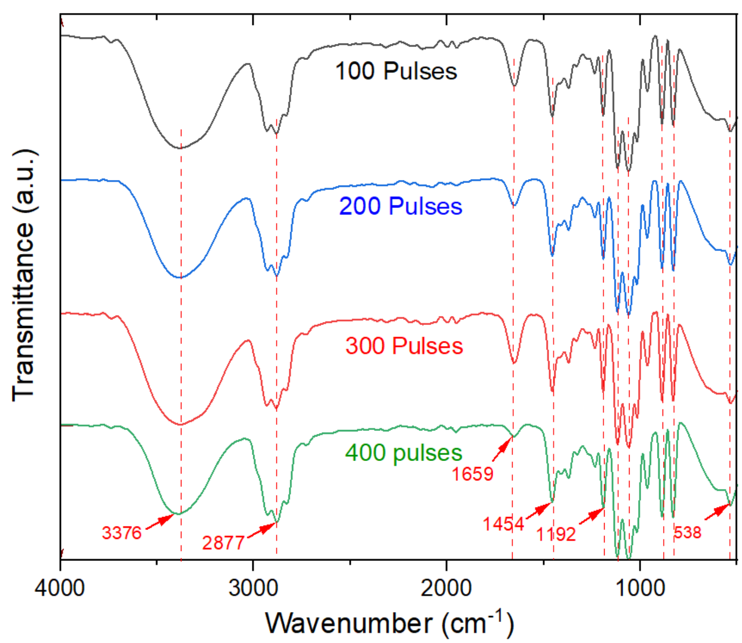

Fig.2. FT –IR spectra of prepared lead oxide nanoparticles.

3.2. Optical properties

The optical characteristics of PbO are typically analyzed using a UV-visible (UV-vis) spectrophotometer. Several scientific articles demonstrate the existence of absorption peaks in the visible range of the UV-vis absorption spectra of PbO. These peaks are associated with the bandgap of PbO in its orthorhombic phase [15]. The characteristic spectra are depicted in Figure , after subtracting the background of 2-Methoxyethanol . Lead oxide compounds, depending on their specific composition and structure, exhibit electronic transitions that lead to absorption of light in the UV-Vis range [6]. Fig.3-a shows absorption spectra of lead oxide samples prepared with different pulses of 100, 200, 300, and 400 pulses. Preparing lead oxide by laser ablation with different pulses greatly affects the optical properties of this material. Using ultraviolet and visible spectroscopy, information will be provided about the electronic structure of these samples. Spectroscopic analysis provides information about the absorption spectra of samples. These absorption spectra reveal the wavelengths that are absorbed by lead oxide. Differences in laser pulse parameters, such as pulse duration, number of pulses and energy density, can lead to differences in the absorption spectrum. For example, shorter pulse durations may result in PbO nanoparticles with narrower band gaps, especially nanosecond pulses, offer unique advantages in targeting narrow-band PbO nanoparticles by providing precise control over nanoparticle size, morphology, and optical properties through minimized thermal effects and enhanced ablation efficiency. These capabilities make nanosecond pulses laser ablation a promising technique for the synthesis of nanoparticles with tailored optical characteristics suited for advanced technological applications [16].resulting in different absorption properties compared to Longer pulse[6], [8]. As can be seen in the absorption spectrum of the sample with 100 pulses, which has less exposure in the number of pulses, this leads to a smaller energy gap. As for the absorption spectrum of samples with 200 and 300 pulses, the absorption is higher, which leads to a larger energy gap. As for the absorption spectrum of the sample with 400 pulses, it has the highest absorption due to the higher number of pulses. This led to the energy gap being larger than the previous one as show fig.3-b.

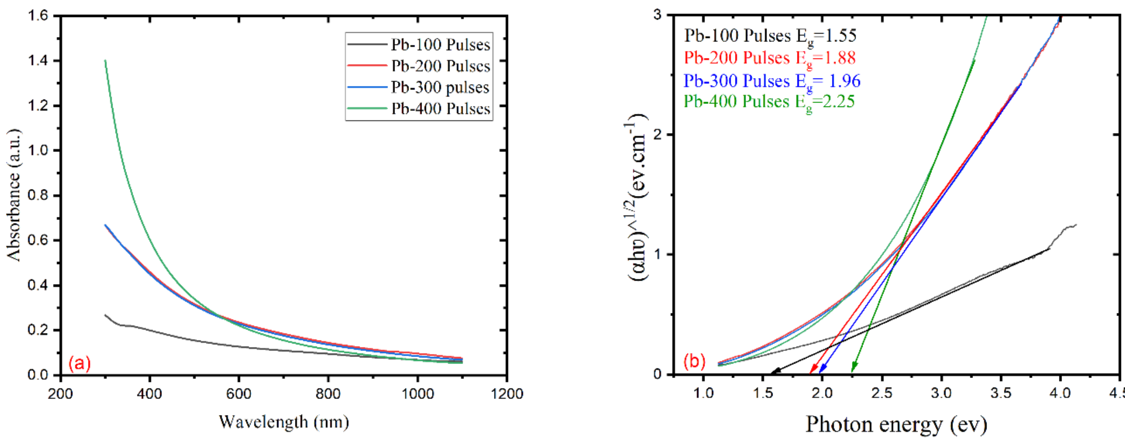

Fig.3. UV-Visible spectra of prepared nanoparticles of Lead oxide at different number of laser pulses. (a) absorbance spectra, (b) Plot of (αhν)1/2 versus hν to determine the energy band gap corresponding to indirect transitions.

3.3. Morphological and structural analysis

Studying the topographical structure of materials and characterizing the surface morphology and topography of materials is crucial for studying various problems, including friction, contact deformation, grain size, etc. Atomic force microscopy (AFM) is a powerful technique to describe these parameters in this research. The morphology parameters were studied by depositing a thin film of lead oxide nanoparticles prepared on a glass substrate using the casting method. The deposited films were annealed at 80 °C for 3 hours inside an oven. Subsequently, the scanning process in contact mode was employed. In this study, the standard software Gwyddion was used to visualize and analyze scanning probe microscopy (SPM) data. The 3D AFM images and the calculated distribution of grain size diameters are presented in Figure 4. The surface roughness, root mean square, and grain size diameters are listed in Table 1.

With an increase in the number of pulses, it was observed that the rate of average roughness and root mean square changes with the size of the resulting particles, explaining that increasing the diameter of grain size leads to greater roughness. This finding aligns with most previous studies on various nanomaterials prepared using the laser ablation method [6]. However, this study observed variations in the size of resulting particles with the number of pulses

In these samples of lead oxide with different number of pulses, when the pulses were increased from 100 to 200 pulses, the diameter of the grain size increased from 71.63 to 72.61 nm. Also, when the number of pulses was increased from 200 to 300 pulses, the diameter of the grain size increased from 72.61 to 78.41 nm. But when the number of pulses increases from 300 to 400 pulses, the diameter of the grain size decreases from 78.41 to 69.92 nm. Its change in size can be attributed to the increased interaction of the nanoparticles with the laser beam as the number of pulses increases, resulting in the production of more nanoparticles that tend to aggregate near the laser spot [17], [18].

Atomic force microscopy (AFM) analysis can provide values about the surface roughness of films for nanoparticles prepared by laser ablation with different pulse parameters. By analyzing the height of the peaks obtained from AFM images, surface roughness such as root mean square (RMS) roughness and mean roughness can be measured. Also, varying pulse parameters can affect the surface roughness of lead oxide, which in turn affects the optical and electrical properties.[19]

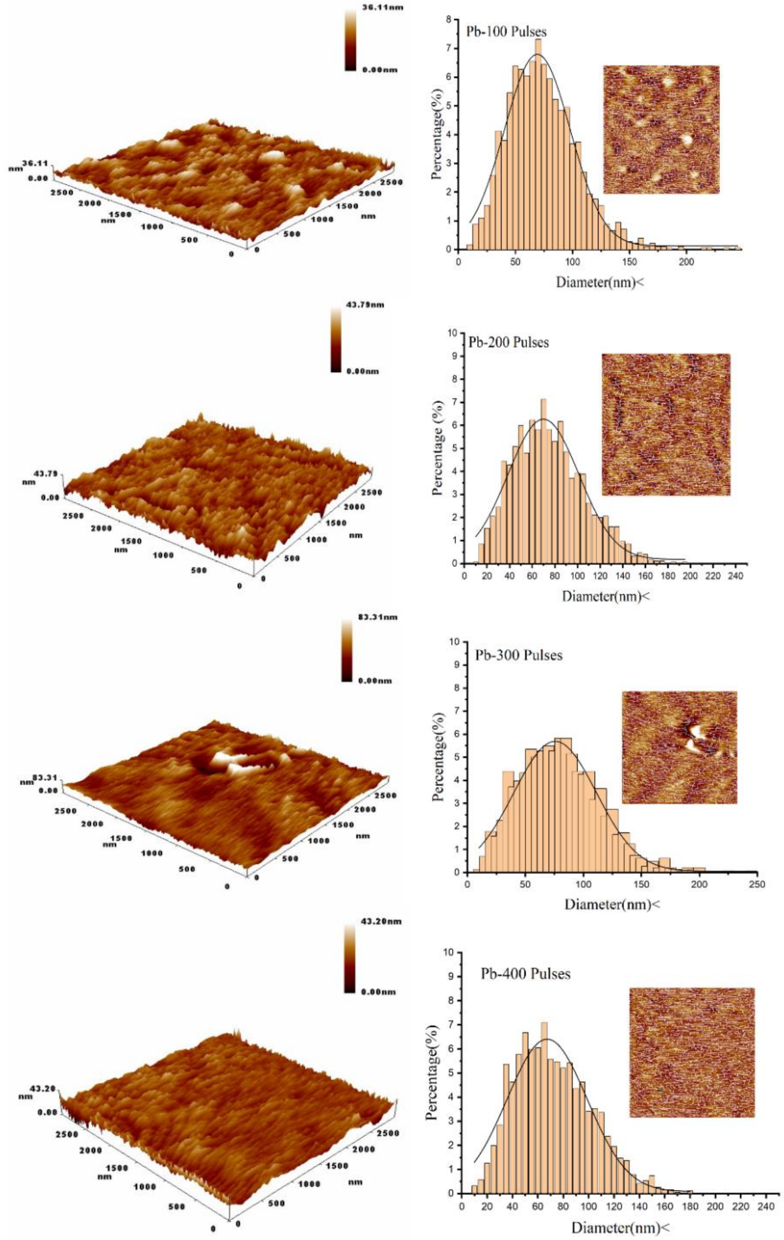

Fig. 4. AFM images of lead oxide nanoparticles

Table 1 Morphological parameters of lead oxide nanoparticles

Sample	Average Diameter (nm)	Root Mean Square RMS (nm)	Roughness Average (nm)
Pb-100 Pulses	71.63	5.09	3.95
Pb-200 Pulses	72.61	5.17	3.96
Pb-300 Pulses	78.41	10.2	7.34
Pb-400 Pulses	69.92	3.32	2.52

4. Conclusion

This study successfully prepared lead oxide nanoparticles using laser ablation technology with pulse counts ranging from 100 to 400. The effect of varying the number of pulses on surface morphology and optical properties was analyzed using different techniques, including AFM, UV-Vis, and FT-IR. The study concluded that the laser ablation method is effective in preparing nanoparticles. The variation in pulse numbers resulted in significant changes in surface morphology, the absorption edge, and consequently, the energy gap.

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References

[1] N. J. G. A. H. Mohammed and A. M. AbdulMajeed, "MgO nanoparticles synthesized using chemical method for Skin cancer cell line (A375) cytotoxic assay," *Mustansiriyah J. Pure Appl. Sci.*, vol. 2, no. 2, pp. 88–95, 2024.

[2] M. H. Mahdieh and B. Fattahi, "Effects of water depth and laser pulse numbers on size properties of colloidal nanoparticles prepared by nanosecond pulsed laser ablation in liquid," *Opt. Laser Technol.*, vol. 75, pp. 188–196, 2015, doi: https://doi.org/10.1016/j.optlastec.2015.07.006.

[3] S. Kamble, K. Bhosale, M. Mohite, and S. Navale, "Methods of Preparation of Nanoparticles," *Int. J. Adv. Res. Sci. Commun. Technol.*, pp. 640–646, Aug. 2022, doi: 10.48175/IJARSCT-7029.

[4] M. Dimitrova and S. Vassilev, "Synthesis of Nanosized Powders for Lead-Acid Battery Pastes by Recycling of Used Batteries †," Oct. 2021.

[5] K. Arulmozhi and M. Narayanan, "Studies on the chemical synthesis and characterization of lead oxide nanoparticles with different organic capping agents," *AIP Adv.*, vol. 3, p. 122122, Dec. 2013, doi: 10.1063/1.4858419.

[6] H. H.Rashed, F. Fadhil, and I. Hadi, "Preparation and Characterization of Lead Oxide Nanoparticles by Laser Ablation as Antibacterial Agent," *Baghdad Sci. J.*, vol. 14, pp. 801–807, Dec. 2017, doi: 10.21123/bsj.2017.14.4.0801.

[7] K. Bin Masood, P. Kumar, M. A. Malik, and J. Singh, "A comprehensive tutorial on the pulsed laser deposition technique and developments in the fabrication of low dimensional systems and nanostructures," *Emergent Mater.*, vol. 4, no. 3, pp. 737–754, 2021, doi: 10.1007/s42247-020-00155-5.

[8] T. Özdemir, A. Güngör, I. K. Akbay, H. Uzun, and Y. Babucçuoglu, "Nano lead oxide and epdm composite for development of polymer based radiation shielding material: Gamma irradiation and attenuation tests," *Radiat. Phys. Chem.*, vol. 144, pp. 248–255, 2018, doi: https://doi.org/10.1016/j.radphyschem.2017.08.021.

[9] A. Sharma *et al.*, "Methods of preparation of metal-doped and hybrid tungsten oxide nanoparticles for anticancer, antibacterial, and biosensing applications," *Surfaces and Interfaces*, vol. 28, p. 101641, 2022, doi: https://doi.org/10.1016/j.surfin.2021.101641.

[10] M. I. Mendivil Palma, B. Krishnan, G. A. C. Rodriguez, T. K. Das Roy, D. A. Avellaneda, and S. Shaji, "Synthesis and properties of platinum nanoparticles by pulsed laser ablation in liquid," *J. Nanomater.*, vol. 2016, no. 1, p. 9651637, 2016.

[11] A. A. Bayyoodh, R. K. F. Alfahed, and F. S. Zainulabdeen, "The effect of nanoparticle size on the structural, optical, and electrical properties of tungsten oxide prepared by laser ablation method," *J. Mater. Sci. Mater. Electron.*, vol. 35, no. 16, p. 1098, 2024, doi: 10.1007/s10854-024-12821-z.

[12] S. M. Siftain, T. Hussain, and S. Khalid, "Preparation of molybdenum oxide and tungsten oxide nanoparticles by pulsed laser ablation," *Indian J. Phys.*, vol. 98, no. 9, pp. 3237–3242, 2024, doi: 10.1007/s12648-024-03098-3.

[13] A. Omidtorshiz, M. R. Benam, M. Momennezhad, Z. Sabouri, and M. Darroudi, "Green synthesis of lead oxide nanoparticles using Ocimum basilicum extract: Photocatalytic assessment and cytotoxicity effects," *Inorg. Chem. Commun.*, vol. 158, p. 111575, 2023, doi: https://doi.org/10.1016/j.inoche.2023.111575.

[14] A. Miri, M. Sarani, A. Hashemzadeh, Z. Mardani, and M. Darroudi, "Biosynthesis and cytotoxic activity of lead oxide nanoparticles," *Green Chem. Lett. Rev.*, vol. 11, pp. 567–572, Nov. 2018, doi: 10.1080/17518253.2018.1547926.

[15] D. L. Perry and T. J. Wilkinson, "Synthesis of high-purity α-and β-PbO and possible applications to synthesis and processing of other lead oxide materials," *Appl. Phys. A*, vol. 89, pp. 77–80, 2007.

[16] A. H. Hamad, *Effects of different laser pulse regimes (nanosecond, picosecond and femtosecond) on the ablation of materials for production of nanoparticles in liquid solution*. IntechOpen London, UK, 2016.

[17] M. Alagar, T. Thirugnanasambandan, and A. Raja, "Chemical Synthesis of Nano-Sized particles of Lead Oxide and their Characterization Studies," *J. Appl. Sci.*, vol. 12, Apr. 2012, doi: 10.3923/jas.2012.398.401.

[18] R. Mahfouz, F. J. Cadete Santos Aires, A. Brenier, B. Jacquier, and J. C. Bertolini, "Synthesis and physico-chemical characteristics of nanosized particles produced by laser ablation of a nickel target in water," *Appl. Surf. Sci.*, vol. 254, no. 16, pp. 5181–5190, 2008, doi: https://doi.org/10.1016/j.apsusc.2008.02.022.

[19] S. Meshram, R. Rupnarayan, S. Jagtap, V. Mete, V. Sangawar, and K. Badnera, "Synthesis and Characterization of Lead Oxide Nanoparticles," vol. 4, Feb. 2015.