Journal of Wasit for Science and Medicine 2024: 17(2), 23-30

Optical, Structural, and Morphological Characterization of Titanium Oxide (TiO_2) Nanoparticles from Laser Ablation in Deionized Water

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Abstract

In this research, we focus on the synthesis of titanium oxide (TiO_2) nanoparticles using the Nd:Utilization of YAG laser pulse ablation technique in liquid environments, with pulse energies from 100 to 500 millijoules. The process employs a Nd:YAG laser operating at a frequency of 6 Hz and a wavelength of 1064 nm. (The TiO_2 nanoparticles synthesized in this study could be used in different high-temperature environments, as they are characterized by favorable physical, chemical, and mechanical properties). The synthesized product was further treated by calcination for four hours at temperatures higher than 600°C which promoted crystalline structure in the lattice. Comprehensive analyses were carried out to determine the nanoparticles' features. Optical features were studied through UV-visible spectroscopy to examine the absorption spectrum and band gap. The detailed examination of the crystal and surface structures was done using X-ray diffraction and field emission scanning electron microscopy (FE-SEM), in order to obtain insight into the structural integrity and the morphology of the titanium oxide nanoparticles. The study provides a deeper insight into TiO_2 nano particle production and their characteristics, thereby emphasizing their suitability for advanced material applications.

Keywords: Titanium oxide (TiO_2), Nd: YAG laser, PLAL pulse ablation technique in liquid.

1 Introduction

Nanomaterials are increasingly being acknowledged as the key ingredient in most cutting-edge technologies and industries. This is because of the peculiarity in properties that can differ greatly from those of displayed by bulk materials[1]. Properties such as size, shape, and surface-to-volume ratio supported by atomic structural differences influence directly the physical and chemical behaviours of nanoparticles, comprising electrical conductivity and energy band characteristics [2].

Some of the nanoparticles that have received great interest are titanium oxide (TiO_2) nanoparticles, especially in their use in many fields right from environmental remediation up to photocatalysis, medical applications, and energy solutions [3]. One of the areas of research focus for TiO_2 nanoparticles has thus been their synthesis, since they have been environmentally friendly, low in toxicity, and show potential for application in green energy technologies [4].

This process of obtaining these NPs usually involves the physical interaction of laser pulses with the titanium target to vaporize material, and then the nucleation and growth of NPs in a liquid medium [5]. This highlights pulsed laser ablation in liquid (PLAL) as an attractive technique, since both energy input and environmental conditions are precisely controlled, giving rise to high-purity nanoparticles with the exact morphological and structural properties required for their application [6].

This will set a stage in due course to discuss in detail the methodologies adopted for the synthesis of titanium oxide nanoparticles using PLAL, their resulting structural, optical, and morphological characteristics across the fields of many applications [7]. It will put an emphasis on the benefits derived from using LA, such as being energy-efficient and low-cost, in order to produce nanoparticles which can suffice the harsh requirements for materials technology[8].

2 Experimental

The experimental setup began with pristine metal sheets of titanium (Ti), measuring 1 cm x 1 cm, characterized by a high purity level of 99.99%. The laboratory configuration, including the use of a high-purity titanium target and its specific composition, intended for pulsed beam irradiation. This apparatus comprises a Nd:YAG laser and a titanium (Ti) target assembly, submerged in deionized water. The titanium target, with a thickness of 2 mm, was mounted on a rotating device at the base of a glass container to prevent continuous ablation at a single point. The sample rotation facilitates even distribution of the laser influence. During the process, the target received 500 pulses from a

Nd:YAG laser, emitting at a wavelength of 1064 nm. These pulses varied in energy from 100 to 500 mill joules and were delivered at a frequency of 6 Hz. The laser was positioned 12 cm away from the target to the focusing lens. The experimental protocol used a typical fluid volume of 3 mL of deionized water (DW). Subsequently, a colloidal solution of titanium oxide nanoparticles was synthesized. The resultant titanium oxide nanoparticles were preserved in the DW.

3. Result and Discussion

3.1 Optical properties

Absorbance for colloidal nanoparticles was assessed using UV-Vis Spectroscopy. These nanoparticles were synthesized via Pulsed Laser Ablation in Liquids (PLAL) at a wavelength of 1064 nm, utilizing energies from 100 to 500 mJ in a medium of deionized distilled water, and subsequently analyzed in a quartz cuvette with deionized water as the reference. It's important to highlight that the particle size of nanomaterials significantly influences their electronic structure, particularly affecting the energy levels of electrons and holes in the conduction and valence bands. Consequently, the absorption and optical emissions of the nanomaterials are dependent on these energy levels. Specifically, absorbance was measured for five TiO_2 nanomaterial samples, prepared using an Nd:YAG laser ablation at varied energies (100, 200, 300, 400, and 500 mJ) operating at a frequency of 6 Hz. The absorbance range for these samples was diagnosed using the UV-Vis Spectroscopy. Figure 1 shows the absorbance of titanium oxide, which is within the range of low wavelengths with high energy a blue colour from 200 nm to 350 nm. This practical result comes in agreement with the size of nanoparticles of titanium oxide in previous studies and the phenomenon in electron microscopy examinations [9].

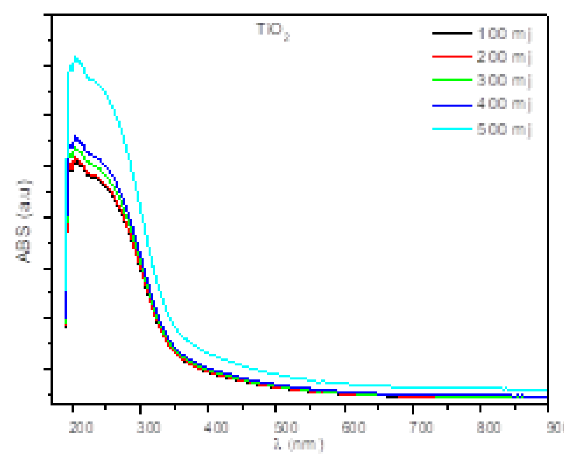

Figure 1: UV-Visible absorbance of synthesiz TiO_2 NPs using different laser energies.

Moreover, figure 2 illustrates the optical energy gaps for titanium oxide, starting at

3.5 eV and decreasing with laser beam injection energy until 3 eV.

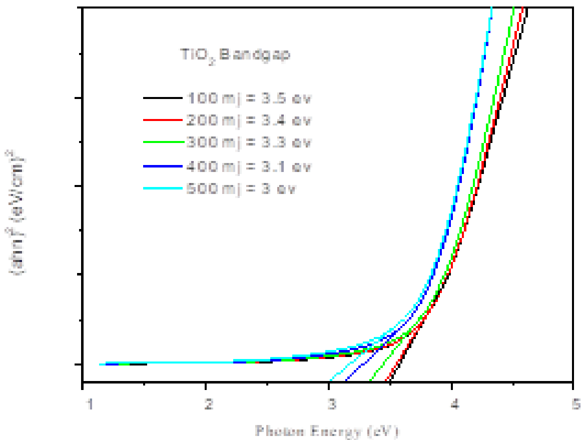

Figure 2: Optical energy gap of TiO_2 NPs for different laser energies.

3.2 X-Ray Diffraction Analysis

The powder X-ray diffraction (XRD) analysis of TiO_2 nanoparticles was performed using a Philips X-ray diffractometer equipped with CuKα radiation (wavelength = 0.15406 nm) to determine the crystal structure. The crystallite size, an important parameter in nanoparticle characterization, was calculated using the Scherrer formula from the full width at half maximum (FWHM) of the most intense peak. This calculation yielded an average crystallite size of 29.35 nm. The crystallite size is crucial for understanding the material's properties, as it can influence the chemical reactivity, mechanical properties, and electronic behaviour of the nanoparticles.

The XRD patterns are shown in Figure 3 that verifies the presence of the tetragonal crystal phase of titanium dioxide, specifically the Anatase form, as it lacks any peaks indicative of impurities. Distinct reflection peaks were observed and labeled according to their corresponding Miller indices: (222), (400), (440), and (622), which are typical for TiO_2 diffraction planes. The average crystallite size calculated from the most intense peak was determined to be 29.35 nm [10].

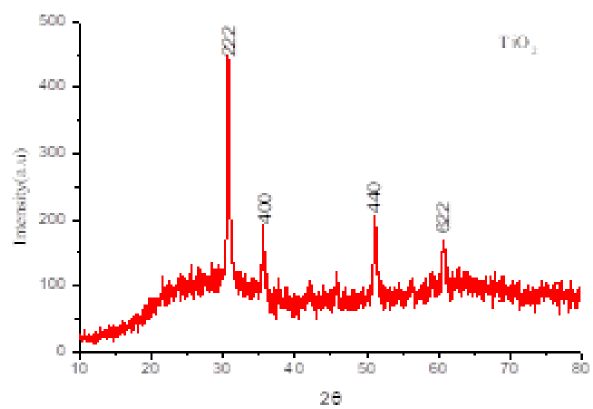

Figure 3: XRD pattern of TiO_2 nanoparticles.

3.2 Surface Morphology and EDX Analysis

Field Emission Scanning Electron Microscope (FE-SEM), Figure (4) shows titanium oxide nanoparticles. These images are particularly useful in analysing the morphology (shape and size) and surface characteristics of the nanoparticles. The titanium oxide nanoparticles appear mostly cubic in shape, which is evident from the clearly visible edges and flat faces in several of the particles, especially noticeable in the high magnification images.

Cubic structures are significant in materials science due to their symmetrical properties and often uniform packing capabilities in crystal lattices. The shape of the particles influences the surface area significantly. For any given volume, a sphere has the lowest surface area, whereas other shapes like cubes have higher surface areas due to their flat faces. Higher surface areas result from non-spherical shapes such as these cubic nanoparticles, which is crucial for applications where interaction with the environment (such as catalysis, sensor functions, or drug delivery) is key.

The cubic shape can expose more active sites on the surface, enhancing the effectiveness in catalytic or adsorption processes. From the FE-SEM images, it appears the titanium oxide nanoparticles are relatively uniform in size, which suggests controlled growth conditions during synthesis.

The image with a marker (e.g., "DI = 24.73 nm") helps in understanding the size distribution and scale. Consistent size and shape are important for reproducible performance in their applications. Heel Growth: In materials science, "heel growth" often refers to the way material layers build up over each other, which doesn't seem directly referenced here. However, if referring to the aggregation or secondary growth on the primary particles, we can observe that there are clusters of cubic nanoparticles. These aggregations can affect the effective surface area available, as clustered particles may not expose as much surface area as individually dispersed particles. Catalysis: For catalysts, high surface areas provided by the cubic.

morphology and surface characteristics (as observed in the FE-SEM images) enhance the reaction rates. Titanium oxide is often used in photocatalysis; thus, maximizing surface area is beneficial. Sensors: In sensor applications, the surface interaction with analytes is crucial, and the increased surface area can improve sensitivity and response times. Biomedical Applications: For drug delivery, higher surface areas allow for greater drug loading capacities and potentially more controlled release profiles. These FE-SEM images provide a clear visual confirmation of the growth and structure of the titanium oxide nanoparticles, which is fundamental for tailoring their properties for specific applications.

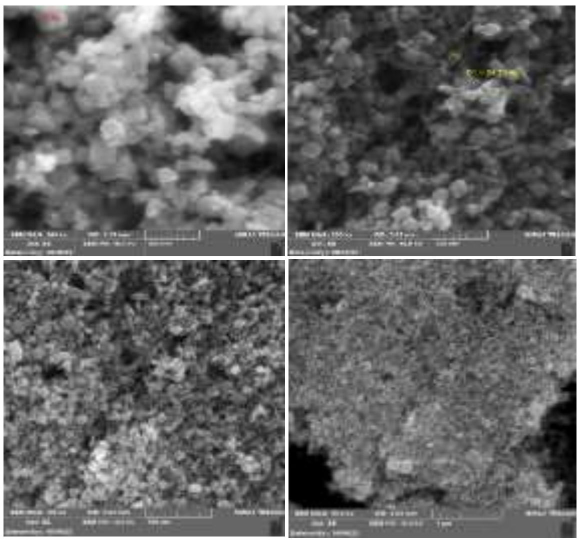

Figure 4: FE-SEM of TiO2 nanoparticles laser pulse ablation technique in liquid.

Furthermore, figure 5 shows the EDX spectra of TiO_2 nanoparticles levels appeared clearly Titanium and oxygen, as well as the appearance which is consistent with the previous characterization and the results have listed in table (1)

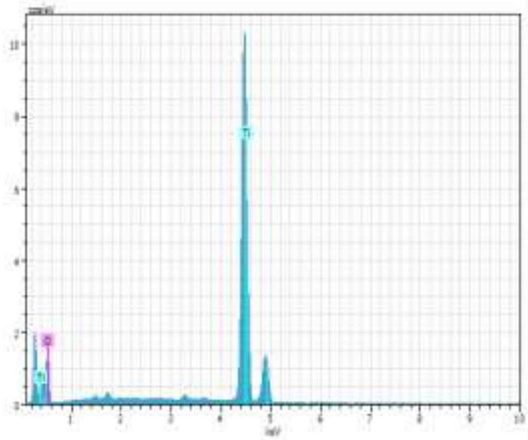

Figure 5: EDX spectra of TiO2 Nanoparticles.

Table 1: Quantitative results from EDX analysis for pure TiO_2 NPs prepared by PLA and calcinated at 500ºC for 2 hours.

Element	Weight %	Atom %
Ti	70.32	87.64
O	29.68	12.36
Total	100	100

4 Conclusions

This study has successfully demonstrated the robustness and versatility of the pulsed laser ablation in liquid (PLAL) technique for synthesizing titanium oxide (TiO_2) nanoparticles using a Nd: YAG-laser. These results, however, show that in this case the structural, optical, and morphological properties of nanoparticles can be controlled through the adjustment of energy level of pulse, which in turn allows changing the properties of the nanoparticles.

The adjustability of the laser throughout the pulse permits the controllable production of TiO_2 nanoparticles, which can be utilized to fine-tune their optical absorption spectra and band gaps. This dial-ability is crucial for the optical devices and photocatalysis, which have specific optical properties of their own. Structural and Morphological Excellence: Most nanoparticles were identified as anatase crystal phase, with the biggest advantage that this phase has the highest chemical reactivity and mechanical stability. The uniformity of the crystallite size revealed by XRD and FE-SEM verifies the reproducibility and thus control of the synthesis.

The mostly cube faces of the nanoparticles, which are demonstrated by the FE-SEM images, improve the applicability of these materials for the processes that require high surface area and active sites, e.g. catalysis and sensors. It has the advantage that the growth process can be optimized, and the desired result is achieved in a high efficiency. Elemental Purity and Composition: EDX (energy-dispersive X-ray spectroscopy) certified titanium and oxygen as the main components of the nanoparticles, which need to meet the requirement of purity to ensure the performance of TiO_2 in the various applications. Broad Application Potential: The synthesized TiO_2 nanoparticles can be credited with facilitating the cross-cutting applications in the environmental remediation, energy solutions, and advanced medical technologies. The fascinating characteristics of these nanoparticles conform to the rigorous standards of the given applications; the potential of PLAL for the development of nanoparticle technology is highlighting.

5 References

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