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Studied Structural, Morphological and Antibacterial properties of TiO₂

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ABSTRACT

In this study, TiO₂ nanoparticles (NPs) were prepared by a laser ablation approach. The synthesized nanoparticles were systematically characterized structurally, morphologically and optically. The structural and compositional studies were performed by employing (XRD) X-ray diffraction and energy-dispersive X-ray spectroscopy EDX, respectively; while FESEM field-emission scanning electron microscopy was the technique used to study surface morphology. The synthesized TiO₂ NPs showed distinct antibacterial activity with significant inhibition rate against both *Staphylococcus aureus* and *Escherichia coli*. In summary, the results show that laser ablation is a simple, an effective and low-cost method for preparing metal oxide nanoparticles.

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INTRODUCTION

Nanostructured materials with unique physical and chemical properties and their substantial fields of application in various branches of science and technology attracted researchers attention in the last decades. Among these, inorganic NPs of metals (e.g., copper, silver, titanium and zinc) It has been widely studied because they act as an essential-driven functional versatility for controlling human pathogens [1-4].

Among the metal oxide nanomaterials, Titanium dioxide TiO₂ nanoparticle has been extensively studied on account of its unique optical, chemical, electrical and photocatalytic properties. TiO₂ has been known as the most widely used photocatalytic material and an active subject for bactericidal applications due to its high

photosensitivity, good photocatalytic activity, low toxicity, high oxidative ability, favorable chemical stability and relatively low cost [5]. Consequently, TiO₂-based materials have been widely studied to improve their photocatalytic activity in interdisciplinary studies [6,7]. The photocatalytic activity of TiO₂ nanoparticles is influenced by numerous physical and chemical parameters including crystallite structure [7], dopant elements [8], surface area specific to their particle size [9] and surface hydroxyl layers [27].

This has been achieved by exploring various approaches to enhance the photocatalytic activity of TiO₂ (metal doping). Among them, silver (Ag) has attracted special attention thanks to its high stability and good electrical and thermal Conductivity level. The introduction of metals, for example Ag onto

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metal–semiconductor heterojunctions was found to improve photocatalytic activity by retarding the fast recombination of photogenerated electron–hole (e[−]–h⁺) pairs. In addition to promoting photocatalytic performance, this mechanism maybe accountable for the enhanced antibacterial activity. Copper ions (Cu²⁺) have also drawn much attention as antibacterial agents due to their broad-spectrum antimicrobial [10–12].

Therefore, one of the strongest strategies for obtaining novel antimicrobial materials with enhanced activity is the incorporation of antibacterial components (Cu, Ag and Zn) into metallic or metal-oxide systems. Thus, one compound with high interest in this sense is copper due to its direction toward being a low-toxicity and resident-compatible [13] metal for biomedical/clinical applications. Currently, numerous researchers have reported the antibacterial activity of copper containing titanium alloys. For example, Ti alloys with 1 and 5 wt. Increasing Cu replacement results in almost 30% better antibacterial performances than commercial pure ti (cp-Ti) bulk samples [14].

On the other hand, in this current study TiO₂ nanoparticles were prepared by using pulsed-laser-ablating-in-liquid (PLAL) technique. Subsequently, the physical and chemical properties of synthesized nanoparticles were analyzed for size, morphology, particle purification phase composition, elements present in synthesized products and use of spectroscopic methods to determine optical properties. It was determined that these potential antibacterial nanostructures are effective against some pathogenic bacteria.

MATERIALS AND METHODS

Synthesis Pulsed Laser Ablation in Liquid (PLAL)

(PLAL) Pulsed laser ablation in liquid is an elegant and eco-conscious place to prepare nanomaterials [2,3]. This technique has been widely studied for synthesis of metallic and metal oxide nanoparticles since it is a pure physicochemical method which allows to obtain nanoparticles in LGA without using complex chemical reduction or stabilizers. Here Laser ablation of titanium (Ti; the target material) in an aqueous solution was employed, using a neodymium-doped yttrium aluminum garnet (Nd:YAG) laser, to generate titanium dioxide (TiO₂) nanoparticles.

Nd:YAG lasers are prevalent, reliable and efficient. PLAL is a process in which the laser beam

concentrates just on the titanium target that is submerged, causing localized heating, melting and vaporization of the titanium target. The species are rapidly cooled and condensed from the hot gas into the surrounding liquid, forming nanoparticles suspended in solution.

Laser Parameters

The operating parameters of the laser (energy, wavelength, fluence, pulse repetition rate and irradiation time) control the key features of nanoparticles generated by PLAL. Besides system parameters, it also affects ablation efficiency and the particle size, morphology, structural features and particle-size distribution of the generated nanoparticles [17]. Consequently, precise control over laser parameters is thus needed to achieve reproducible nanoparticle suspensions.

THz Refl ectivity of Titanium Pellet Irradiation In this study, a titanium pellet was added to 3 mL deionized water and was irradiated with Nd:YAG laser. We further modified the energy in 700 mJ with 1065-nm λ , pulse repetition rate: 1 Hz. Experimental focusing of pulse laser beam on the surface of titanium target in liquid medium. Next, the colloids that were obtained were collected for further characterization and analysis.

Characterization of TiO₂ Nanoparticles

The crystalline structure of synthetic TiO₂ nanoparticles was characterized by X-ray diffraction (XRD) in a SHIMADZU XRD-6000 diffractometer (Shimadzu, Japan). They were obtained with Cu-K α radiation $\lambda = 1.54 \text{ \AA}$ and from the XRD patterns taken as a function of Bragg diffraction angle, we can identify all the crystalline phases that existed in produced material.

The nanoparticle morphology was characterized by Field-emission scanning electron microscopy (FE-SEM; TESCAN Mira-3, Czech Republic). Energy-dispersive X-ray spectroscopy (EDX; Bruker Nano GmbH, Germany) was performed to investigate elemental and compositional features of the synthesized samples.

RESULTS AND DISCUSSION

X-Ray Diffraction Analysis

The characterization of the synthesized TiO₂ nanoparticles included X-ray diffraction (XRD) in order to determine their basic crystalline structure and long-range ordering, as depicted by Fig. 1. XRD patterns of the synthesized sample

and standard PDF Journal—standard powder diffraction file (No. 21-1272). As shown in Fig. 1, The synthesized sample gave clearly indexed diffracted peaks to tetragonal TiO₂ (Huang2020), the known phase for anatase.

It produces, in the x-ray diffraction (XRD) pattern, very broad diffraction peaks that proves to

be quantifiable of a nanoscale crystalline material and smaller crystallite domains. The broadening of these peaks [150 a1fia64] may also be attributed to structural disorder and the conditions used during synthesis (for laser-ablation) in which lithography-compatible parameters are pursued. Table 1 also provides the average crystallite size

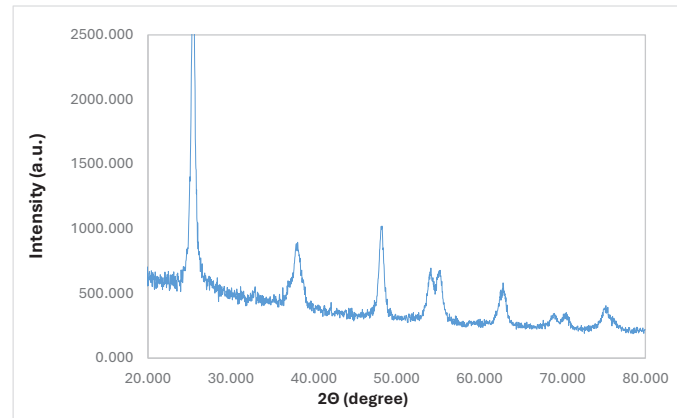


Fig. 1. XRD pattern of TiO₂ Ps.

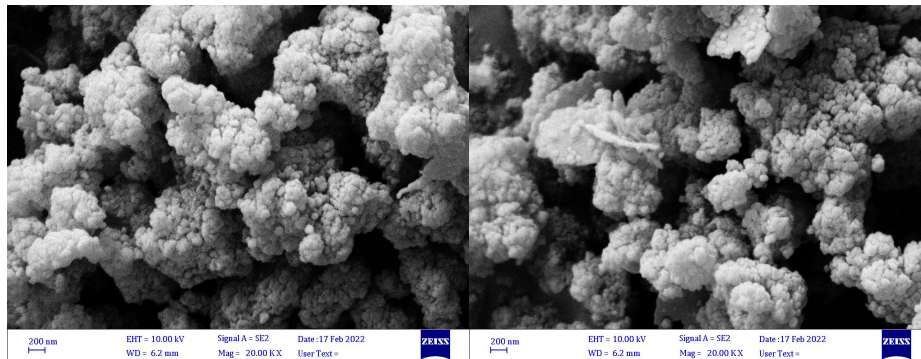


Fig. 2. FESEM of TiO₂.

Table 1. The average of the grain size of TiO₂ NPs.

Peak position (2θ)	Grain size (nm)	Average of grain size (nm)
25.42	15.07	13.37
32.92	27.60	
36.9	13.95	
37.89	11.99	
48.14	12.79	
54.05	10.24	
55.17	8.14	
62.84	7.15	
68.95	8.67	
75.36	10.03	

of prepared TiO₂ nanoparticles as calculated using data obtained from XRD.

We reported in Table1 the size of crystallite that we calculated for the TiO₂ nanoparticles obtained using above-mentioned PLAL method.

The morphology of TiO₂NPs sample is introduced in Fig. 2. The FESEM image with higher resolution showed that the TiO₂ particles with diameters from 40 to 100 nm are discretely dispersed. shows spherical morphology and show small nanoparticles on the surface of TiO₂ spheres.

Antibacterial Activity

The prepared TiO₂ nanoparticles were examined

for their antibacterial activity against two bacterial strains (ie, Gram-negative *Escherichia coli* (*E. coli*) and Gram-positive *Staphylococcus aureus* (*S. aureus*)) using agar well diffusion method [19,20]. Sterile Petri dishes were aseptically filled with 20 mL of Mueller–Hinton (MH) agar medium. With a sterile inoculation loop, transfer of bacterial strains from stock cultures [21]. With a sterile tip, 6 mm diameter wells were prepared in inoculated agar plates. Aliquots of the TiO₂ nanoparticle suspensions (with selective particle types included or excluded from an all-or-nothing screening) at differing concentrations were then added to well-defined locations in these selected wells.

To assess its antimicrobial activity, the

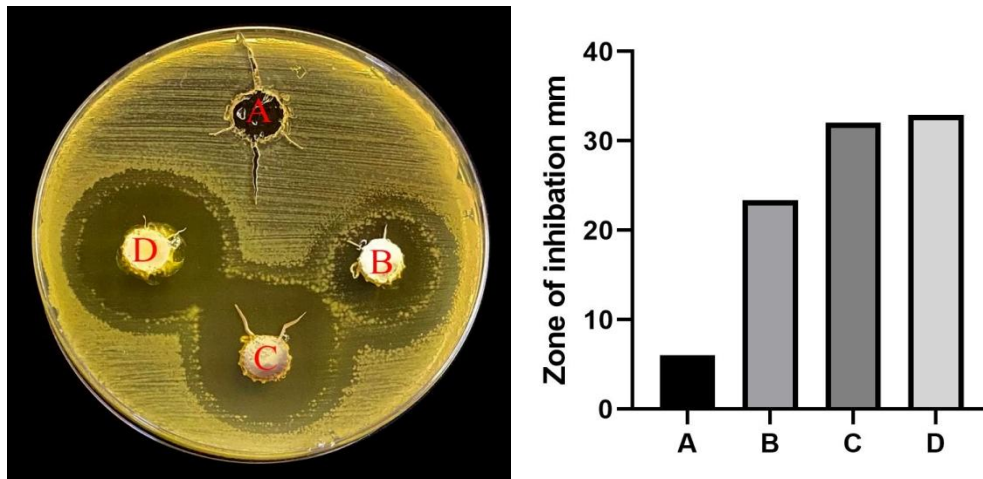


Fig. 3. Antibacterial activity of (TiO₂) against *E.Coli*. A, control. B, TiO₂.

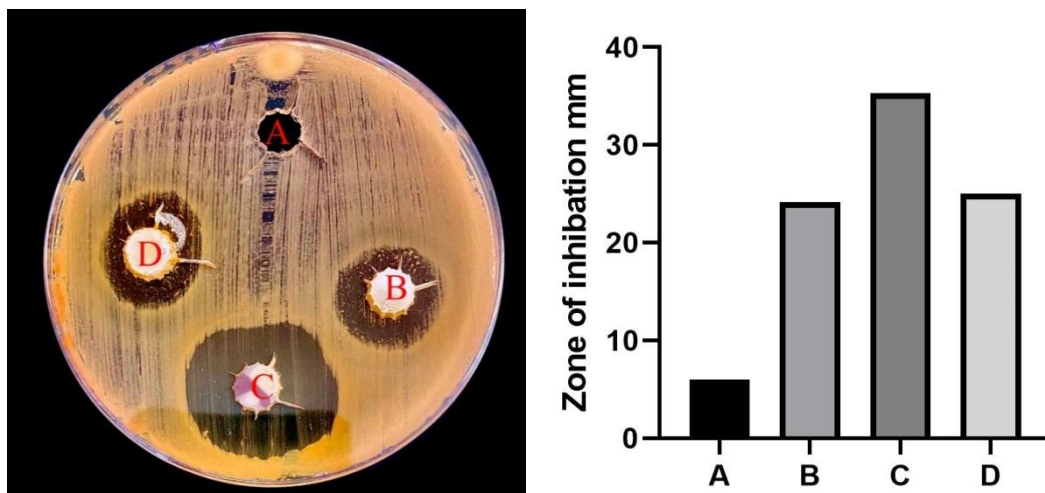


Fig. 4. Antibacterial activity of (TiO₂ NPs) against *S.aureus*. A, control. B, *E-coli*.

inoculated plates were incubated for 24 h at 37 °C and inhibition zones around wells containing TiO₂ nanoparticles were measured. Diameters of the inhibition-zones were recorded for further analysis [22,23].

Data were analyzed using Graphpad Prism software [24]. Each of the measurements was done in duplicate on three independent experiments and reported as mean ± SD. Statistical significance was defined as $p < 0.05$ (25).

The TiO₂ nanoparticles that were synthesized exhibited detectable antibacterial activity for both of *E. coli* and *S. aureus*. In contrast, the inhibition by this treatment was strain-specific wherein greater *E. coli* relative to *S. aureus* inhibition responses were detected in three different experiments conducted across time-points

Potential differential susceptibilities of bacteria to the TiO₂ nanoparticles may be explained by structural differences in the cell envelope. Gram-positive bacteria have thick peptidoglycan and gram-negative have thinner peptidoglycan, an outer membrane as well as lipopolysaccharide. The structural differences can also lead to the interaction of nanoparticles between nanomaterials and costory bacterial cells eventually affecting the antibacterial response. Nonetheless, the variation in susceptibility might alternatively depend on other factors such as particle size, surface characteristics and physicochemical properties of the paromomycin plan cell envelope or concentration of the nanoparticles.

CONCLUSION

TiO₂ nanoparticles were prepared by pulsed laser ablation in liquid method and characterized XRD, focus ion beam and UV-Vis spectroscopy. X-ray diffraction (XRD), scanning electron microscope (SEM) and energy-dispersive spectroscopic (EDS) analyses confirmed the structure and morphology of the synthesized TiO₂ nanoparticles. In antibacterial evaluation, the TiO₂ nanoparticles were found to inhibit growth of *Escherichia coli* and *Staphylococcus aureus*, however the bactericidal action rather differed with bacterial strain. In conclusion, these results highlight the potential of PLAL to be a more convenient and greener way for producing TiO₂ nanoparticles with antibacterial activity. These results obtained here may also be useful for the synthesis of various types TiO₂ based

nanomaterials and their potential utility where antimicrobial activity is more important.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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