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Synthesis, Antibacterial Activity of Tio_2 Nanoparticles Suspension Induced by Laser Ablation in Liquid

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ABSTRACT

Colloidal solutions of Titanium dioxide nanoparticles were synthesized by laser ablation of Titanium plate in deionized water. Studies of synthesized nanoparticles suspensions were characterized by UV-VIS absorption, Fourier transform infrared spectroscopy (FTIR) and transmission electron microscope (TEM). FTIR characterization confirms the formation of Titanium dioxide nanoparticles. TEM shows that the nanoparticles with sizes from (3 - 30) nm. The antimicrobial activity was carried out against Staphylococcus aureus and Escherichia coli. The Titanium dioxide nanoparticles showed inhibitory activity in bacteria.

Keyword: Tio_2 Nanoparticles, Metal Oxide, Laser Ablation In Liquid, Antibacterial Activity.

تحضير وفعالية المضاد الحيوي لثاني أوكسيد التيتانيوم النانوي المحضر بواسطة الاستئصال بالليزر في سائل

الخلاصة

تم تحضير السائل الغروي من جسيمات ثاني اوكسيد التيتانيوم النانوية بواسطة الاستئصال بالليزر لمعدن التيتانيوم المغمور في ماء لا ايوني . درست الخصائص للجسيمات النانوية العالقة باستخدام طيف الامتصاص الأشعة فوق البنفسجية (UV-VIS) وتحويل فورير لأشعة تحت الحمراء (FTIR) والمجهر الالكتروني (TEM). وضح ال FTIR تشكل جزيئات أوكسيد التيتانيوم. ويشيرTEM الى جسيمات نانوية مع حجم يتراوح nm(3 -30). تم استخدام المحلول النانوي كمضاد بكتيري ضد Staphylococcus aureusو Escherichia coli. أظهرت جسيمات أوكسيد التيتانيوم النانوية فعالية تثبيط في البكتريا.

INTRODUCTION

Metal oxide nanoparticles NPs have unique physical, chemical and biological properties. This attributed to their small particle size and high surface-to-volume ratio [1]. Because of these facts, wide varieties of techniques for the synthesis of nanostructured materials were developed such as: hydrothermal [2], photo-reduction [3], electrochemical [4], and sol-gel [5]. Among these unique properties, Nano-sized organic and inorganic particles are being producing for use in medical properties. Titanium dioxide (TiO_2) nanoparticles have interest attention due to have special physicochemical properties. TiO_2 nanoparticles is a large band gap semiconductor and is used in a wide range of application such as photocatalytic, solar cells , gas sensor , and nanomedicine [6]. Highly ionic nanoparticles metal oxides may be particularly valuable antimicrobial agents as they can be prepared with extremely high surface areas and unusual crystal morphologies [7]. The bactericidal property of such nanoparticles depends on their size, stability, and concentration added to the growth medium, since this provides greater retention time for bacterium nanoparticles interaction [8.9].

Pulsed laser ablation in liquid media (PLAL) is a technique for fabricating metal and metal-oxide nanoparticles. It produces sized controlled nanoparticles in colloidal as well as powder phases, usually in a one-step top–down procedure [10]. The main advantages of this method are simplicity, does not require costly chambers and high vacuum pumping systems hence it is considered as a green method. In PLAL, the shape and size of the produced nanoparticles can be controlled by using a variety types of liquid and a range of laser parameters such as wavelength, ablation time, pulse energy, etc. [11].

In the present work, studying the characterization of colloid TiO_2 NPs were synthesis by PLAL and investigated the antibacterial activity of colloidal TiO_2NPs against Gram-negative bacteria (E. coli) and Gram-positive bacteria (Staph. aureus.)

EXPERIMENT PROCEDURE

Preparation of colloidal NPs

Titanium targets in the form of plate was placed at the bottom of glass vessel containing 1 ml of deionized water of height 2mm from the target surface as shown in experimental setup in Figure (1). The titanium was irradiated with pulsed Q-switched Nd:YAG laser (1064 nm, pulse duration=9 ns, repetition rate 1Hz) operating at different energies (80 and 200 mJ) for different ablation time (10, 20) min. Each sample was weighed before and after the ablation by a digital weighter to determine the mass concentration.

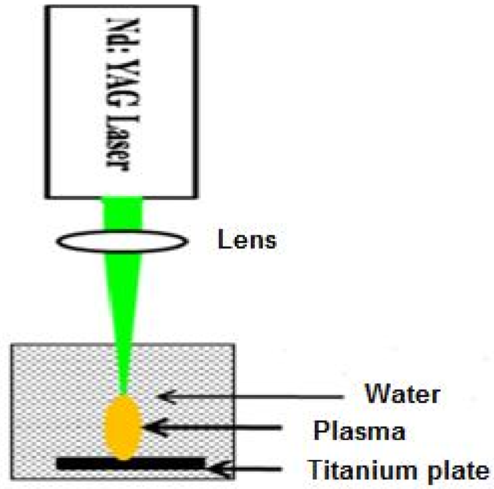

Figure (1) The schematic of the experimental setup.

Characterization of Nps

The colloids of synthesized nanoparticles were characterized by using optical and structural techniques. For recording the FTIR spectrum, the (Testscan Shimadzu FTIR 8000 series) are performed over range between (400-4000) cm^{-1} for suspension to confirm the formation of Titanium oxide nanoparticles. The optical absorption spectrum of TiO_2 nanoparticles was recorded by spectrophotometer (type UIR-210A SHIMADZU) within the wavelength ranges (200-500 nm).TEM analysis was used to determine the particles size and morphology of NPs.

Antimicrobial activity of NPs

Two bacterial strains were used: *Staphylococcus aureus* and *Escherichia coli*. The division of biotechnology, department of applied sciences; university of technology, Baghdad, Iraq kindly supplied these bacteria .The bacterial suspension was prepared and adjusted by comparison against 0.5 Mc-Farland turbidity standard (5x 10^7 cell ml^{-1}) tubes. It was further diluted to obtain a final of 5 x 10^6 cell ml^{-1}. All bacteria strains were sub- culture on nutrient broth. The broth was inoculated by the 0.2 ml/5ml broth with either the bacteria strains, then added 0.5 ml of TiO_2 nanoparticles at concentration 400, 600 or 1000µg ml^{-1}. The tubes were incubated at 37 °C for 24 h. The bacterial growth was measured by optical density at 600 nm wavelength. The mean values of inhibition were calculated from triple reading in each test.

RESULTS AND DISCUSSION

Figure (2) shows the FTIR spectrum TiO_2nanopaicles colloidal solution prepared using laser ablation of Titanium target with Nd:YAG at two energy (80 and 200) mJ in deionized water for 10 min. The spectrum was recorded in the range (400 -4000cm^{-1}). This shows the peaks at 3417.6cm^{-1}, 1645.2cm^{-1}, 434cm^{-1} and 450cm^{-1}. The intense and wide peak centered at 3417.6 cm^{-1} assigned to O-H stretching bond, and the peak at 1645.2cm^{-1} corresponds to the H-O-H bending mode bond. The band at around 434cm^{-1} and 450cm^{-1}were attributed to the O-Ti-O vibration. These results agree with other researches [12].

The UV-Vis absorption spectra taken for two samples of colloid TiO_2 nanoparticles prepared at 10 min ablation time with different laser energy (80-200) mJ of are shown in figure (3). UV -Vis absorption spectra were carried out in order to characterize the optical absorbance of colloidal solutions. The results collected immediately after the production of nano-colloids in deionized water. A strong absorption due to the TiO_2 nanoparticles dominates the spectrum below 350nm while a smooth decay towards longer wavelengths is observed. Slightly high laser energy causes a considerably higher absorbance, which refers to high concentration of the NPs.

The morphologies of colloids TiO_2 nanoparticles were observed by TEM of sample prepared at 200 mJ and for 10 min ablation time, from Figure (5a), nanoparticles exhibited a spherical shape with little aggregations observed in the as-prepared sample. While Figure (5b) shows the particle size distribution histogram for TiO_2 nanoparticles. It was seen that the size particles with ranging s between 3 and 30 nm and the average size of particles is 16 nm.

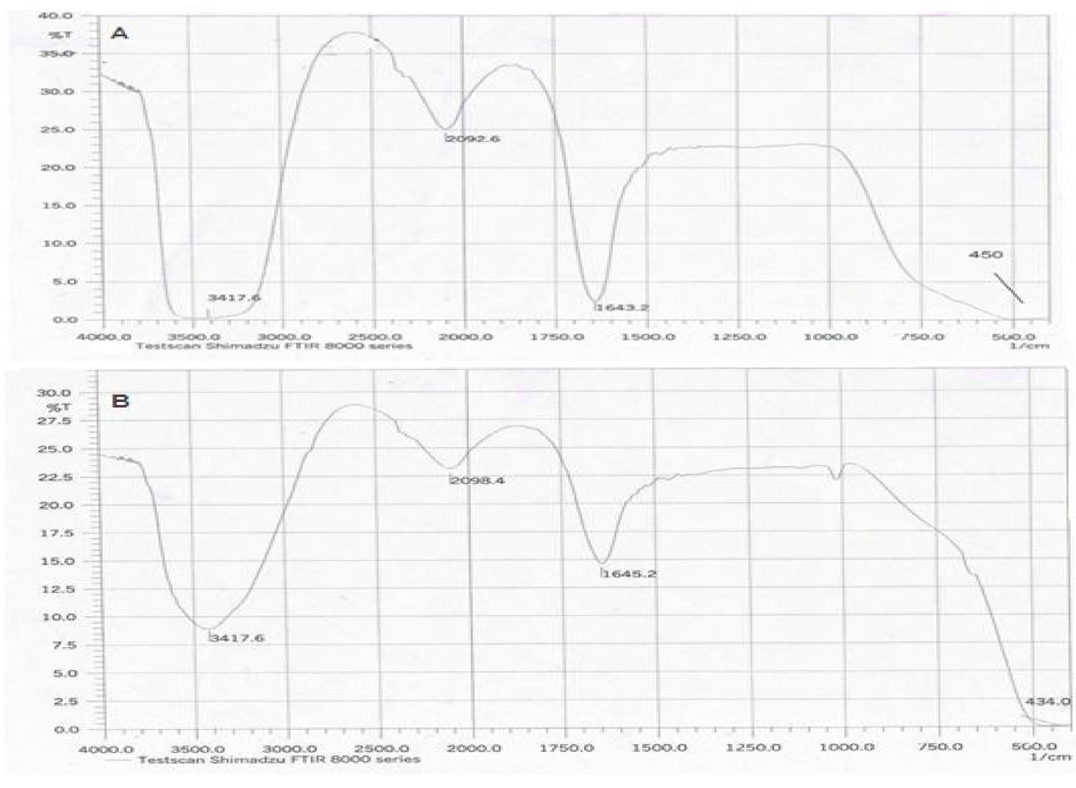

Figure (2) FTIR spectrum of Colloidal TiO_2 nanoparticles prepared for 10min at two energy: A) 200mJ. B) 80mJ.

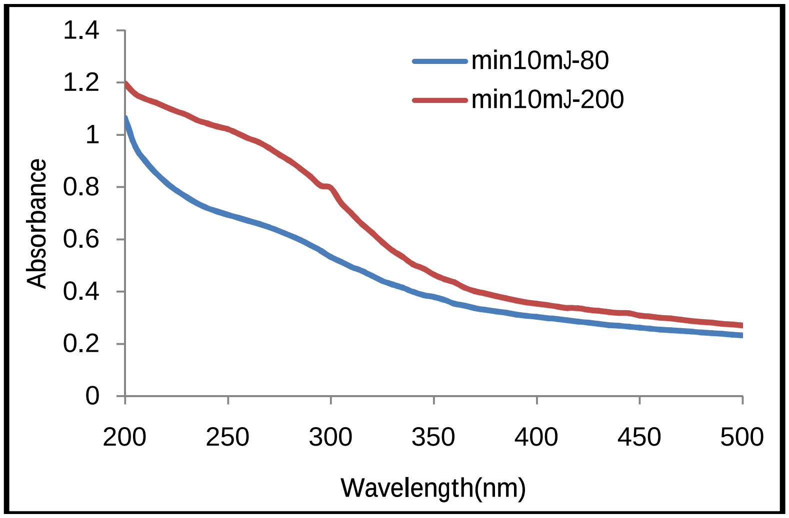

Figure (3) UV-Vis absorption spectrum of TiO_2 nanoparticles colloids prepared by Nd:YAGlaser ablation of nickel in deionized water.

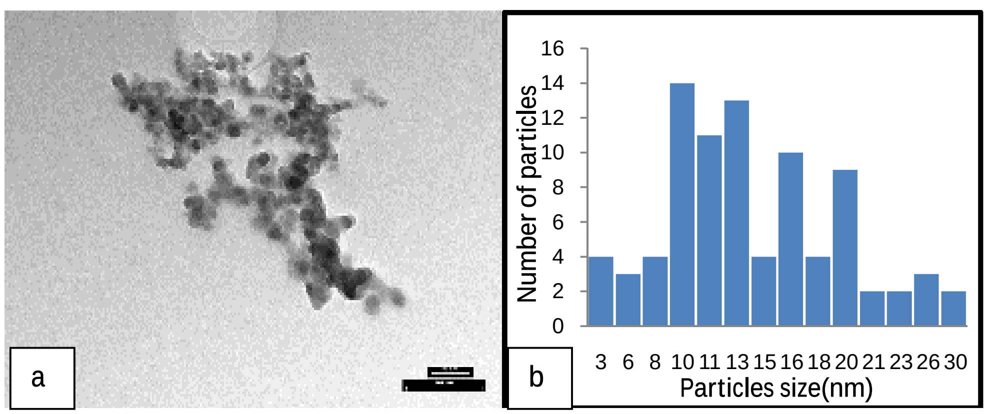

Figure (4 a) TEM image of sample, (b) Particle size distribution of sample.

The antimicrobial activity of titanium nanoparticles against two pathogens *S.* aureus and E.coli was investigatedby broth medium methods. UV-VIS spectrophotometer was used in the measurements of the optical density of the bacterial cultures in liquid nutrient medium, with the measuring wavelength set at 600 nm. Based on the data derived from the liquid medium tests, we were able to see that the bacterial cell number was dropped in both types of pathogens and the inhibition was concentration dependant manner (Figures5).

Bacteria have different membrane structures, so they are classified as Gram negative and Gram positive. The structural difference lies in the organization of peptidoglycan, which is the key component of membrane structure. Gram-negative bacterium exhibit a thin layer of peptidoglycan (about 2–3 nm) between the cytoplasmic membrane and the outer cell wall. Outer membrane of Gram-negative is predominantly constructed from tightly packed lipopolysaccharide (LPS) molecules, which increase the negative charge of cell membranes and are essential for structural integrity and viability of the bacteria. The outer membrane of Gram-negative bacteriam often confers resistance to hydrophobic compounds [7]. The wall of Gram-positive bacteria contains a thick layer (about 20–50 nm) of peptidoglycan, which is attached to teichoic acids that are unique to the Gram-positive bacteria cell wall [7]. The overall charge of bacterial cells at biological PH values is negative because of excess number of carboxylic groups, which upon dissociation makes the cell surface negative. The opposite charges of bacteria and nanoparticles are attributed to their adhesion and bioactivity due to electrostatic forces. Nanoparticles have larger surface area available for interactions, which enhances bactericidal effect than the large size particles; hence, they impart cytotoxicity to the microorganisms [13].

The mechanism of inhibitory action of titanium nanoparticles induced by laser ablation on microorganisms, though not very clearly understood, could be by their adhesion to the cell membrane and further penetration inside or by interaction with phosphorus containing compounds like DNA disturbing the replication process or preferably by their attack on the respiratory chain. The merit of this explanation depends much on the molecular organization of these particles. The particles may effect on cell division by modifying the cellular environment but induce damage through a direct action on the cell wall and plasma membrane, which become weaker regions which suspected that of dividing cell for both gram-positive and gram- negative bacteria.

Another studies suggest that when E. coli bacteria were treated with highly reactive metal oxide nanoparticles. A bacterial membrane with this morphology exhibits a significant increase in permeability, leaving the bacterial cells incapable of properly regulating transport through the plasma membrane and, finally, causing cell death [14].

Studies on the mechanism of bactericidal activity of TiO_2 suggest that oxidative damage first takes place on the cell wall when the TiO_2 makes contact with the cell. The results of the antimicrobial study complies with the earlier findings by Matsunaga et al. [15] and other researchers that irradiated TiO_2 exhibits bactericidal activity [16, 17] and the efficacy of this disinfection process is proportionally correlated with the TiO_2 dose and the time of exposure. The potential of this multifaceted compound for disinfection in environmental and medical fields is immense and further research needs to be done to harness its exceptional properties.

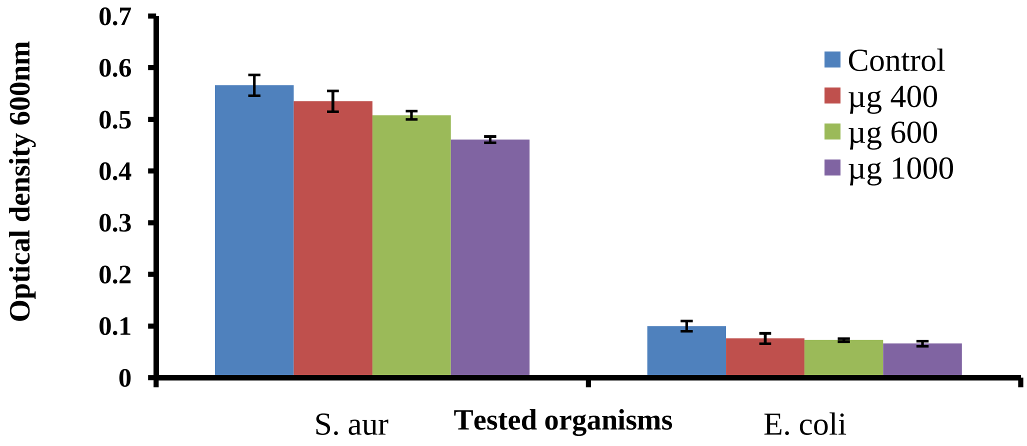

Figure (5) Optical density of tested bacteriatreated with titanium nanoparticles. (Control bacteria without NPs), (, and bacteria with different concentration of NPs 400,600 and 1000 µg μl^{-1} respectively).

CONCLUSIONS

The present work shows that titanium dioxide nanoparticles can be easily produced by laser ablation of titanium metal in water. Variation of size and shape of nanoparticles has be found to depend on laser energy. FTIR spectra confirmthe bond between O-Ti-O at around (434) cm^{-1}. Antibacterial activity experiments performed on various microorganisms clearly demonstrated the effectiveness of TiO_2 nanoparticles against bacterial growth due to smaller particle size and high concentration of this sample.

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