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Preparation Zno Nanoparticles with Different Concentration by Laser Ablation in Liquid and Their Use in Anti Bacterial Activity

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Abstract

The study included the preparation of ZnO nanoparticles using (P.L.A.L.) technology, as their optical properties were improved. The effect of the number of pulses (400, 500, 600) made from distilled water (DW) as a growth medium was examined using an Nd: Yak laser. The wavelength used (1064 nm), the ablation energy (600 mj), the repetition rate (1 Hz), the diameter of the laser beam at the focal center (2 mm) of the target, and the distance between the target and the lens (8 cm). Tests were conducted and the results showed (FE-SEM) studied the surface morphology of the prepared material, and average size diameters ranging from (80.29 - 35.62 nm) were obtained for (ZnONPs).

The results of the FTIR examination showed that pure functional groups with different patterns and densities of absorption bands form zinc dioxide nanoparticles. It was shown that absorption appears for the beam near the wave number (478.3463 cm^{-1}) due to the coupling vibration (Zn-O), and the absorption band appears near the wave number (1660.71 cm-1) due to the vibration of the (H-O) beam.

UV spectroscopy measurements showed a clear absorption peak at the wavelength (375 - 380 nm). In biological application, nanoparticles have shown bacterial inhibition by acting as antibiotics against bacterial strains (S. epidemidis, P. aeruginosa, and E.coil).

Keywords: nanoparticles, ZnO, pulsed laser ablation in liquids, biological application.

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تحضير جسيمات أوكسيدالزنك النانوية بتراكيز مختلفة بواسطة الاسئصال بالليزر النبضي في السوائل واستخدامها في النشاط المضاد للبكتيريا

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الخلاصة

تضمنت الدراسة تحضير جسيمات (ZnO) النانوية باستخدام تقنية (P.L.A.L.) ، اذ تم تحسين الخصائص البصرية لها , حيث تم فحص تأثير عدد النبضات (400 ، 500 ، 600) نبضة, المصنوعة من الماء المقطر (DW) كوسط نمو باستخدام ليزر Nd: Yak ، الطول الموجي المستخدم (1064 nm) ، وطاقة الاستئصال (600 mJ) ، معدل التكرار (1 Hz) ، وقطر حزمة الليزر عند المركز البؤري(2mm) للهدف ، والمسافة بين الهدف والعدسة (8 cm). تم اجراء الفحوصات حيث اظهرت نتائج (FE-SEM) دراسة مورفولوجية السطح للمادة المحضرة وتم الحصول على معدل حجم اقطار تتراوح (80.29 – 35.62 nm) للـ (ZnONPs) . وبينت نتائج فحص (FTIR) أن المجموعات الوظيفية النقية ذات الأنماط والكثافات المختلفة لنطاقات الامتصاص تشكل جسيمات ثاني أكسيد الزنك النانوية. حيث تبين أن الامتصاص يظهر للشعاع بالقرب من رقم الموجة ($478.3463\ cm^{-1}$) بسبب اهتزاز اقتران (Zn-O) ، وظهور حزمة الامتصاص بالقرب من رقم الموجة ($1660.71\ cm^{-1}$) بسبب اهتزاز الحزمة (H - O) وأظهرت قياسات التحليل الطيفي للأشعة فوق البنفسجية ذروة امتصاص واضحة عند الطول الموجي (375 nm -380). وفي التطبيق البيولوجي لقد ظهر وجود تثبيط بكتيري في الجسيمات النانوية من خلال العمل كمضادات حيوية ضد سلالات البكتيريا (S. epidemidis، P. aeruginosa، وE.coil).

الكلمات المفتاحية: الجسيمات النانوية، أوكسيد الزنك، الليزر النبضي، السوائل، التطبيقات البيولوجية، المادة النانوية.

Introduction

Nanotechnology is the manipulation of matter on an atomic and molecular scale. Or it is processing a material with at least one dimension, with a size ranging from (1-100nm).

The term "nanoparticle" refers to a particle in which all three dimensions of nanometers in scale contain a sufficiently small number of constituent atoms or molecules that differ from the properties inherent in their bulk counterparts and exist in a variety of shapes such as spherical, triangular, cubic, pentagonal, rod, shells, ellipsoidal, etc. [1].

Because of their size-dependent features, nanoparticle preparations and their technological applications have promising areas in science and technology in terms of unexpected properties and behaviors [2]. Strange and wonderful changes in physical, chemical, optical, electrical and magnetic properties occur when the sizes of materials are reduced to the nanoscale [3- 4].

Properties that are not seen on the macroscopic scale are now important on the nanoscale such as quantum mechanics, optics, magnetism, surface interaction and thermodynamics. The nanoscale is that materials can have different properties on the nanoscale, some are better at conducting electricity or heat, some are stronger, some have different magnetic properties, and some reflect light better or change colors as they change size.

1-D nanostructures are confined in two spatial directions, for example, nanowires, nanotubes, etc., 0-D nanostructures are confined in all three spatial directions, for example, nanoparticles, quantum points, etc. [5].

The exciting and sometimes unexpected properties of nanoparticles are due in part to surface aspects of the material that dominate properties compared to mass properties. Nanoparticles exhibit several special properties related to bulk materials. Nanoparticles have a very high surface area to volume ratio [6].

This provides an enormous impetus for diffusion, especially at elevated temperatures. The large surface area to volume ratio also reduces the initial melting temperature of the nanoparticles [7].

In general, nanoparticles are designed with surface modifications tailored to the needs of the specific applications for which they will be used [8].

Pulsed Laser Ablation in Liquids (PLAL)

Laser ablation of a solid target in a liquid medium has been widely used in the preparation of nanomaterials and the fabrication of nanostructures. There is a large variety of nanomaterials such as metals, alloys, semiconductors, polymers, etc., which have been synthesized using laser ablation of solids in liquid. Therefore, laser ablation in liquids has been recognized as an effective and general route for nanocrystal synthesis and nanostructure fabrication [9].

Laser ablation is an important preparation method for producing materials with new properties. The excision of metal targets in solvents is used to prepare colloidal solutions of nanoparticles. Due to the unique photochemical and photophysical properties of the nanoparticles which differ from those in bulk [10].

This technique has attracted great interest due to the possibility of fabricating debris-free microstructures on various solids including metals [11] and diamond-like carbon [12]. On the other hand, laser ablation of solids immersed in a liquid medium is a simple method for generating nanoparticles from different materials. The ejected nanoparticles remain in the liquids, forming a colloidal solution [11]. As shown in Figure (1).

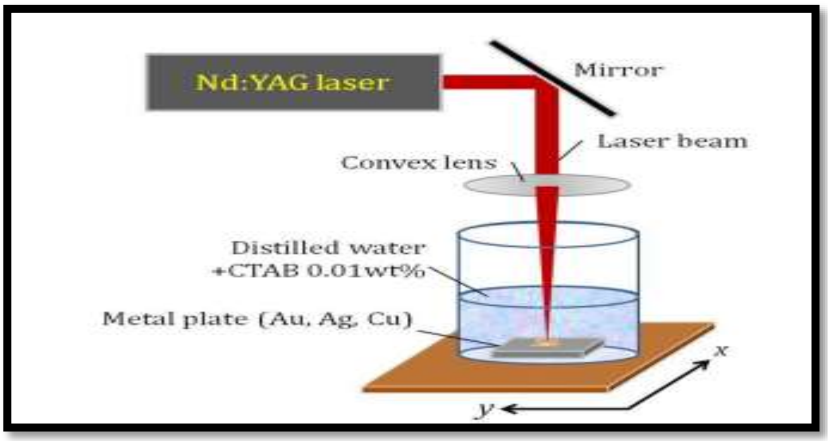

Fig.1: Pulsed laser ablation technique in liquids [11].

Advantages of Pulsed Laser Ablation In Liquid

Nanoparticles can be prepared using various techniques including chemical reduction, electrochemical reduction, laser ablation [13], pulsed laser deposition, photo reduction, and flame firing. Metal combustion, electrolysis, spark discharge, and chemical liquid deposition (CDF) [14]. Among the various methods, the pulsed laser ablation in liquids (PLAL) method is an important technique for the (top-down) fabrication of nanomaterials, which is a versatile technique for several different types of nanoparticles such as metals, alloys, oxides, and similar semiconductors [15]. and Noble Metal [16]. Advantages of the PLAL method :-

1. Inexpensive equipment to combat eradication cases [17].
2. Simple and clean preparation compared to the chemical process that pollutes the final product and the environment [18].
3. Fine nanoparticles can easily be obtained in a single step without subsequent heat treatment due to the high active state of the separated species [19].
4. Production of materials at very low temperatures and low pressure. [20].
5. Making two or more materials at the same time [20].

Physical properties of zinc oxide nanoparticles

Over the past few decades, nanotechnology has witnessed an amazing development in the fastest growing field of science and technology because metal oxide nanoparticles (NPs) are gradually being used in many industrial applications. Among the various metals, zinc attracts more attention due to its strong reductive ability, moderate reactivity, and having five stable isotopes. Among the various zinc-based nanostructures such as sulfide, ferrite, phosphide, selenide, and telluride, zinc oxide (ZnO) is most attractive due to its wide applicability, environmental friendliness, and diverse physicochemical properties. And it's also in one-dimensional, two-dimensional, and three-dimensional structures. One-dimensional structures make up the main group, which includes nanorods, tubes, wires, strips, belts, combs, spirals, screws, and rings. The two-dimensional structures of ZnO are found as nanosheets and nanospheres, while the flower, are examples of three-dimensional structures [21].

Figure (2) shows the crystal structure (ZnO) and the basic physical parameters of ZnO at room temperature in Table (2-1).

Table 1: Some physical properties of (ZnO) [22].

Atomic symbol	ZnO
Crystal system	Hexagonal a, b =3.249, c=5.2066) Å (
Atomic radius Zn Atomic radius O	0.74 Å 1.4 Å
Direct Energy Gap	3.37Ev

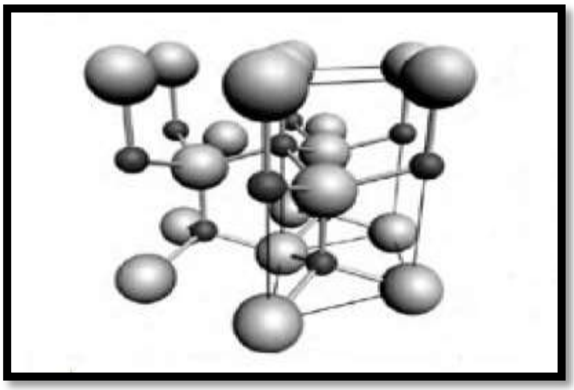

Fig. 2: The crystal structure of ZnO. The large spheres represent (Zn) and the small spheres represent (O) [22].

Methods

A pulsed laser was used to ablate ZnO nanoparticles and prepare them to study their structural and optical properties.

The system includes a Chinese Q-Switched N:YAG laser source from HUAFEI with two wavelengths (1064 nm, 532), with a maximum energy (1000 mJ) per pulse, pulse time (10 ns), repetition rate (6 Hz).) and the effective beam diameter (2 mm) used in ablation. The lens used has a focal length (20 cm) to achieve high laser flux.

The process of eradication of metal targets was carried out in the postgraduate laboratories of the Department of Physics, College of Science, University of Diyala.

Preparation of nanoparticles

Using in-liquid and room-temperature pulsed laser ablation (PLAL) technology, colloidal solutions of metal nanoparticles were generated using high-purity (9.99 percent) ZnO targets. As shown in Figure 2. Before and after each eradication stage, the metal target was polished and cleaned by washing it with ethanol and then purifying the water using an ultrasonic system, and then cleaning the targets to remove impurities. The target was then immersed in distilled water (DW) at the bottom of a glass vial with a volume of water used in all ablation operations (5) ml and a height of the liquid above the target surface (4) mm.

The energy used for each pulse is (600) millijoules. At this energy, three (400, 500, 600) laser pulses were used that bombarded the surface of the metal target. To produce a colored colloidal solution containing nano-metallic target particles. Which was then extracted with the color of the water changing after the eradication process.

Results and Discussions

X-ray Diffraction Results

The X-ray diffraction patterns of zinc were examined before work to ensure its purity. Figure 3 shows the results of the XRD of the target zinc before laser ablation. The results obtained are identical to a large extent with the results of the researcher Solati [23] and as shown in Table 2

Table 2: shows the experimental results of zinc metal before pulsed laser ablation.

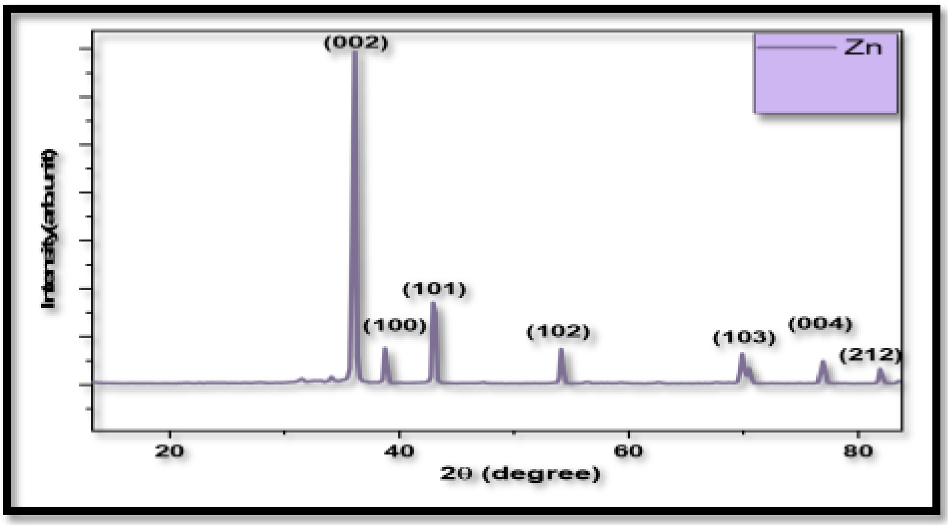

Fig.3: X-ray diffraction of zinc before excision.

Table 2: Experimental results of the target metal zinc before eradication.

2Θ (DEG)	D_{HKL} (Å)	HKL)(
36.1	**2.4809**	002)(
38.8	**2.315**	100)(
43.1	**2.0971**	101)(
54.2	**1.6905**	102)(

Field Emission Scanning Electron Microscopy Results (FESEM)

(FE-SEM) tests were conducted for the prepared sample at energy (600 mJ) and number of pulses (400, 500, and 600) pulse and frequency (1Hz). The surface topography of zinc was studied after it was deposited on glass. The sizes of the nanoparticles were calculated using the program (Image j), as well as the graph, size distribution and average size of the nanoparticles after they were drawn through the program (Origin Lab 2018).

Figure 4 represents (FE-SEM) images of zinc nanoparticles prepared with (400 pulses) and (600 mJ) energy. The figure shows microscope images at (200nm) scale and (20kx) magnification. Where the particles were obtained with a nanoscale and the particles obtained with almost spherical shapes.

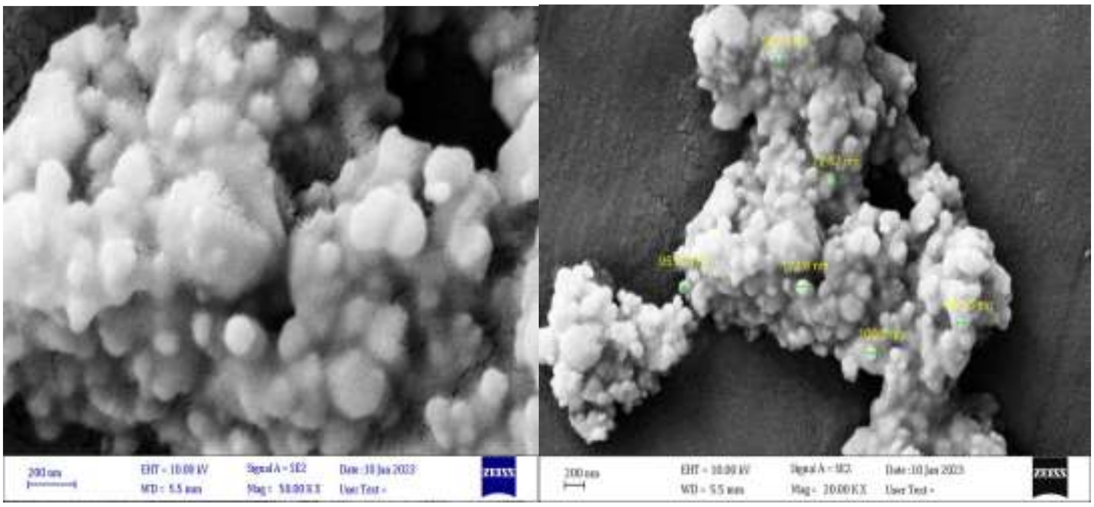

Fig.4: FE-SEM measurement results for zinc nanoparticles prepared with (400 pulses).

Figure (5) shows the EDX plot of the sample (ZnNPS) prepared with (400) pulses and the ablation energy (600 mJ) in a distilled water medium in which there are (Zn, O, Au) elements. Some elements were obtained because the atmosphere surrounding the device (EDX) contains these elements, while we notice the appearance of the highest peak of gold (Au) due to the surface of the (EDX) device, whose inner surface is coated with gold metal. The material has been coated with materials with high conductivity and small grain size to give a good image as it increases the signal ratio and reduces noise when shooting.

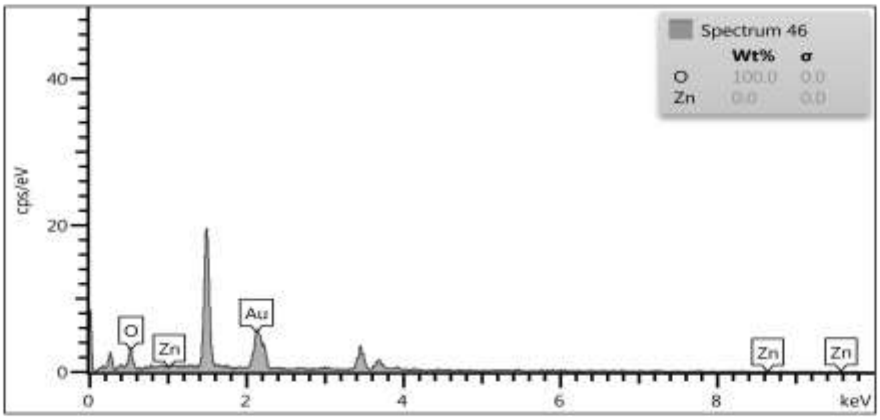

Fig.5: EDX diagram of zinc nanoparticles prepared with 400 pulses.

Figure (6) represents (FE-SEM) images of zinc nanoparticles prepared with (500 pulses) and (600 mJ) energy. The figure shows microscope images at (200nm) scale and (20kx) magnification. Where the particles were obtained with a nanoscale and the particles obtained with almost spherical shapes.

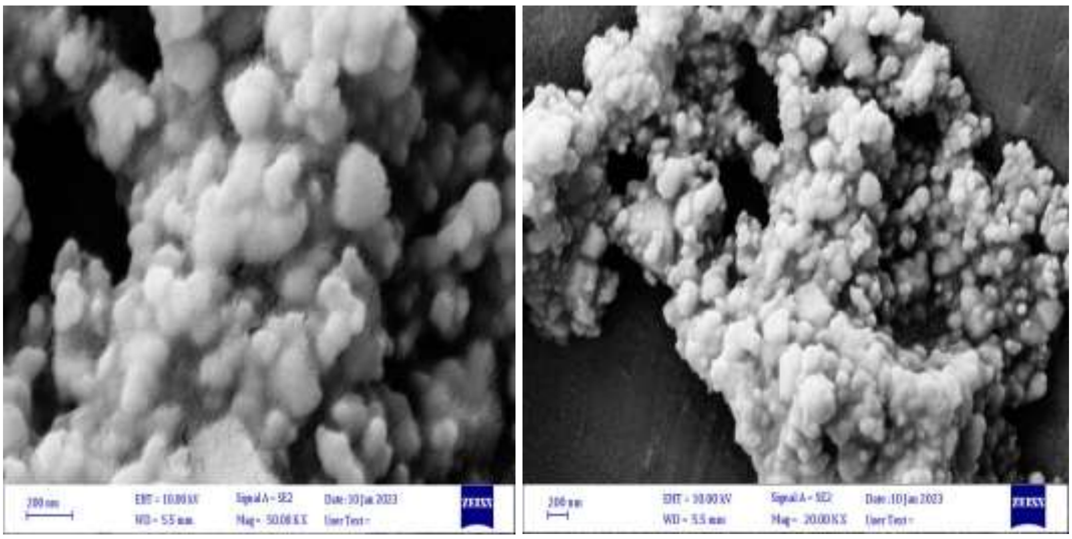

Fig. 6: FE-SEM results of zinc nanoparticles prepared with 500 pulses.

Figure (7) shows the EDX plot of the sample (ZnNPS) prepared with (500 pulse) and ablation energy (600 mJ) in a distilled water medium in which there are (Zn, O, Au) elements. Some elements were obtained because the atmosphere surrounding the device (EDX) contains these

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elements, while we notice the appearance of the highest peak of gold (Au) due to the surface of the (EDX) device, whose inner surface is coated with gold metal. The material has been coated with materials with high conductivity and small grain size to give a good image as it increases the signal ratio and reduces noise when shooting.

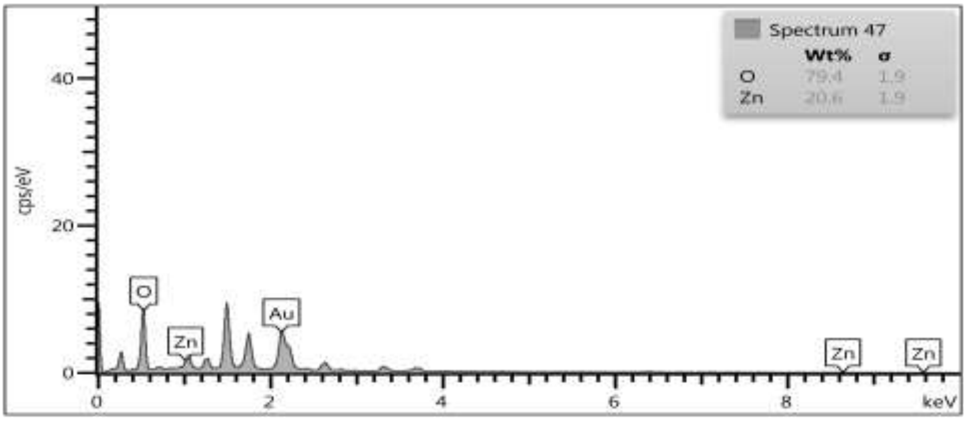

Fig.7: EDX diagram of zinc nanoparticles prepared with 500 pulses.

Figure (8) represents (FE-SEM) images of zinc nanoparticles prepared with (600 pulses) and (600 mJ) energy. The figure shows microscope images at (200nm) and (20kx) magnification. Where the particles were obtained with a nanoscale and the particles obtained with almost spherical shapes.

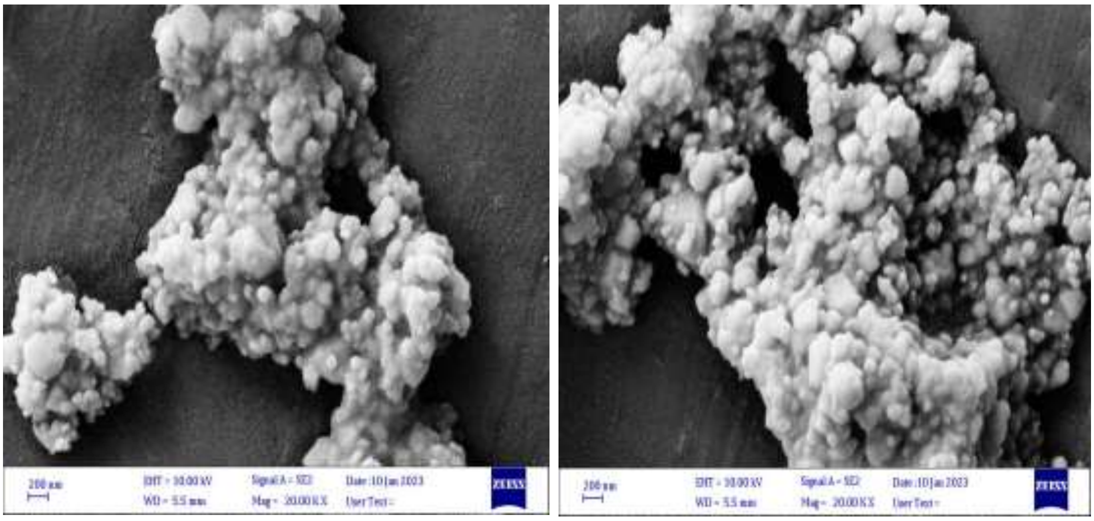

Fig. 8: FE-SEM results of zinc nanoparticles prepared with (600 pulses).

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Figure (9) shows the EDX plot of the sample (ZnNPS) prepared with (600 pulse) and ablation energy (600 mJ) in a distilled water medium in which there are (Zn, O, Au) elements. Some elements were obtained because the atmosphere surrounding the device (EDX) contains these elements, while we notice the appearance of the highest peak of gold (Au) due to the surface of the (EDX) device, whose inner surface is coated with gold metal. The material has been coated with materials with high conductivity and small grain size to give a good image as it increases the signal ratio and reduces noise when shooting.

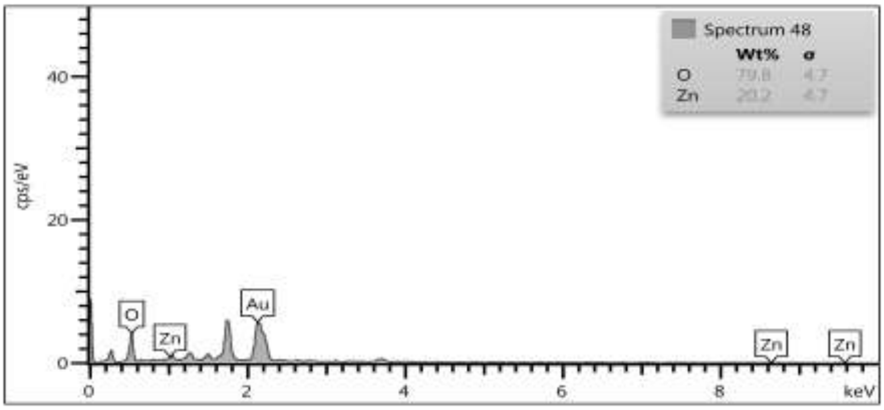

Fig. 9: EDX diagram of zinc nanoparticles prepared with 600 pulses.

Fourier Transform Infrared test results (FTIR)

An important laboratory test, infrared spectroscopy is an important tool for obtaining information about the position of ions in the crystal structure through oscillations [24, 25].

A measurement was also performed to identify the potential biomolecules responsible for the identification and reduction factor of the nanoparticles ablation by pulsed laser (PLAL) and a measurement of infrared spectroscopy (FTIR) is used to identify the active groups (chemical bonds) where the identity of the compound is determined based on how The absorption of chemical bonds ranges from (400-4000) cm-1 because each compound has its own absorption. The results of infrared spectroscopy of a solution of silver nanoparticles and titanium dioxide showed the presence of different bands, which indicates the multiplicity of active groups in these particles [25].

Figure (10) shows the infrared spectrum of (ZnNPs) prepared in the medium of distilled water with different number of pulses (400, 500, 600) pulses and constant ablation energy (600 mJ), as they consist of distinct functional groups of pure (ZnNPs). . The locations and intensity of the absorption bonds for (ZnNPs), we note that the absorption bond that appears near the wave number (420 cm-1) results from the vibration of the (Zn-O) bond, and the absorption appears near the wave number (1330-1420 cm-1) resulting from on the vibration of the (H-O) group [26]. This matches the results obtained by the researcher Nazima 2021[27].

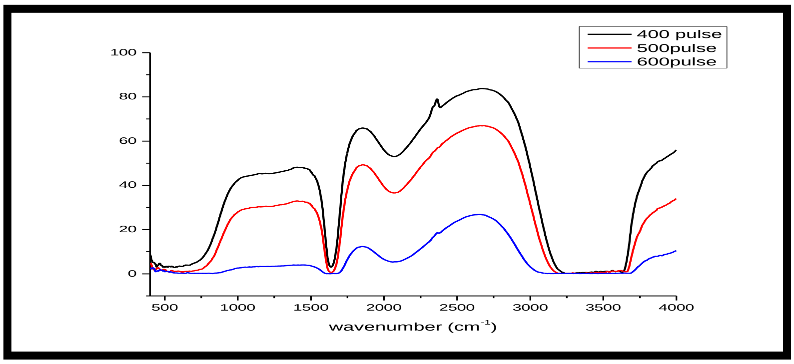

Fig. 10: Fourier Transforms Infrared (FTIR) of Zinc Nanoparticles Prepared at Energy (600mJ) and Number of Pulses (400, 500, and 600).

Effect of laser pulses on absorbance and surface plasmon resonance

Colloidal solutions containing target metal nanoparticles were prepared by pulsed laser ablation in liquid (PLAL) method to study the effect of laser pulses on the properties and size of nanoparticles at energy (600 mJ) and the different number of pulses (400, 500, 600) bpm. In distilled water and using the wavelength of the laser used (1064 nm), where the fall of the laser beam on the surface of the metal immersed in the liquid leads to the formation of a spark column with a strong shock wave that spreads and floats in all directions in the liquid, and it has been observed that the color of the solution has changed and its intensity has increased with Increasing the number of laser pulses, this indicates the formation of a solution containing metal nanoparticles from the extracted metal, and surface plasmon resonance (SPR) peaks in the UV-Vis region is a characteristic sign for the formation of metal nanoparticles from both silver and titanium dioxide [27]. This change is due to the oscillation and coherence of the electrons on the surface of the nanoparticles, in addition to the increase in the number and size of the excised particles, and the intensity of the absorption increases with the increase in the number of pulses [28].

Figure(11) shows the absorption peaks of the zinc particles prepared at (600mJ) energy and (1Hz) frequency. In high purity water, two types of electron transmission are generated, shown by the absorption peaks, which are caused by the transition between the band and within it, and the transition between the valence band and the conduction band, and this phenomenon is a result of the quantum confinement effect [29].

Increasing the number of laser pulses means saving more energy and means removing a larger amount of material. It has been observed that increasing the number of laser pulses produces plasma and the column becomes denser, and the colloidal Zn nanoparticles can become denser. This indicates that larger particles will be produced due to two facts. The first is due to a longer growth time and the second to a higher probability of mass aggregation that has a long tail towards the wavelength. is a characteristic of ZnO formation. The tail may be due to the range dispersion of the particle sizes [30].

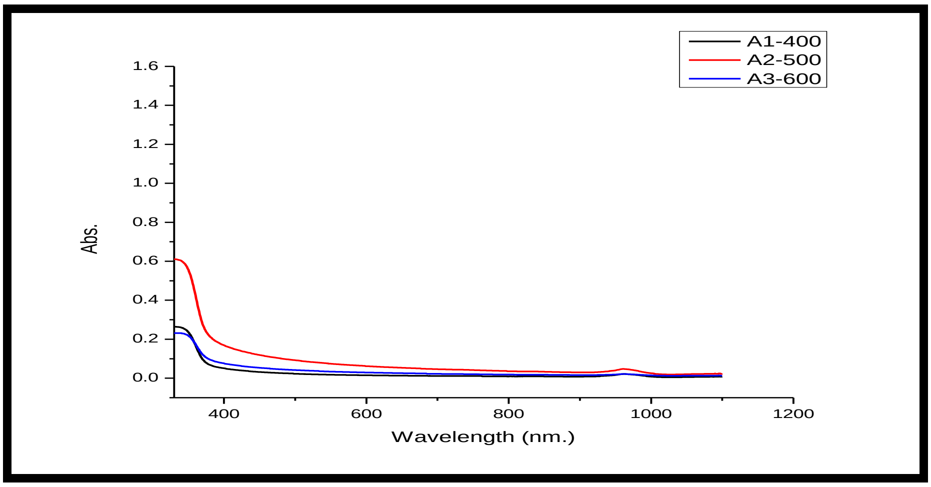

Fig. 11: Absorbance as a function of wavelength of zinc nanoparticles solution at energy (600 mJ) and number of pulses (400, 500, and 600).

Biological Applications

Results of Inhibition of Bacterial Isolates Using Nanomaterial's

Diverse nanomaterials are currently considered a viable alternative to antibiotics with significant efficacy towards antibacterial activities, and several studies have shown that the bactericidal properties of ZnNPs are strongly affected by their shape, size, concentration, and colloidal state [31].

It was found that reducing the size of ZnNPs enhances their stability and biocompatibility [32]. Thus, it is essential to design nanoparticles of suitable size and shape with desirable surface properties for use in a variety of clinical and therapeutic interventions.

The particle size of ZnNPs decreases, and the antibacterial activity increases. Interestingly, the attachment of ZnNPs to cell membranes and the resulting changes in the lipid bilayer lead to increased membrane permeability, cell damage and death, and the antibacterial effect appears to be more pronounced when using smaller nanoparticles [33, 34]. The area-to-volume ratio of ZnNPs and the crystal surface structures are important factors that determine the antibacterial activity of ZnNPs.

An inhibition zone test was performed to qualitatively verify the antibacterial property of zinc nanoparticles prepared with distilled water and the average size of different diameters obtained with different numbers of (600, 500, 400) pulses and ablation energy (600 mJ). Where these materials were used for three types of bacteria: (P. aeruginosa), (S.epidemidis) and (E.coil).

The results showed a high inhibitory effect against both types of bacteria, as shown in Figures 12 , 13 and 14 . It was found that the highest bacterial inhibition appeared in the number of (600) pulses.

The antibacterial activity was detected by the sample that was without laser ablation, that is, distilled water only (0 mJ). The inhibition was very small compared to the laser-prepared samples. On the other hand, Zn has long been proven to be a good antibacterial material as well. [35].

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Fig. 12: Inhibition Area of S.epidemidis

Fig. 13: Inhibition Area of P.aeruginosa

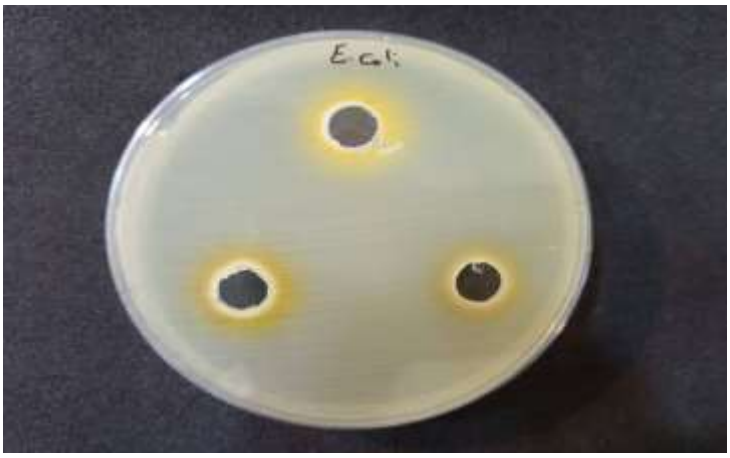

Fig. 14: Inhibition Area of E.coli

Conclusions

Zinc nanoparticles were prepared using the surface plasmon resonance pulsed laser ablation method. Ablation was performed at a wavelength of 1064 nm under ideal experimental conditions, with different numbers of pulses (600, 500, and 400) and constant energy (600 mj). The optical and structural properties of zinc nanoparticles were studied. UV-visible, X-ray, FE-SEM, and FTIR analyzes confirmed the antibacterial effects. The results of (FE-SEM) showed that we can obtain nearly spherical nanoparticles. As for the optical properties of the hybrid, the UV results indicated distinct peaks at appropriate wavelengths confirming the composition of the prepared materials. The prepared nanoparticles were used in biological applications to inhibit bacterial species, and three types of bacteria were inhibited.

It can be concluded that the work integrates both nanotechnology and bacteriology leading to new future long-term studies of bactericides.

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