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Preparation of Cadmium Nanoparticles by Laser Ablation Method in Water at Different Energies and Their Biological Application

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Abstract

This work includes preparation of CdO nanoparticles by laser ablation in water at two energies (360 mJ and 560 mJ). Various techniques (XRD, SEM, and AFM) were employed to ascertain the structural and morphological properties. X-ray diffraction of CdO films at different energies (360 mJ and 560 mJ) demonstrated their polycrystalline nature with a cubic phase. The crystalline size was 28.30 and 40.16 nm at energies 360 mJ and 560 mJ respectively. AFM measurements indicate that the average diameter has risen from 82.99 nm to 117 nm, while the surface roughness has escalated from 1.18 nm to 2.68 nm as laser energy rose from 360 mJ to 560 mJ. The SEM scans indicate that the substrate surfaces are uniformly coated with interconnected particles, exhibiting smallest diameter 13.94 nm and 21.78 nm at energies of 360 mJ and 560 mJ, respectively. (UV, and FTIR) techniques were employed to ascertain the optical and chemical properties, where the energy bandgap for CdO is documented as 3.8 eV and 3.6 eV at energies 360 mJ and 560 mJ respectively. FTIR spectra identified the range 400-1000 cm^{-1} as the characteristic bands for the CdO mode at 400-1000 cm^{-1}. Increased inhibition of bacteria and fungi was observed with increasing laser energy concentration, where the inhibition zone of Staphylococcus aureus and candida albicans was 12 mm and 19 mm at 360 mJ, while it records 14 mm and 20 mm at 560 mJ.

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1. Introduction

Pathogenic microorganisms represent a major health issue, as they cause numerous hospital admissions and deaths annually. Bacteria that are resistant to antibiotics are developing at an alarming rate [1]. Generally, bacteria are divided into two categories: Gram-positive and Gram-negative. Nanoparticles (NPs) can impede the growth of both types of bacteria by penetrating their nanometer-sized pores and diminishing their activities [2] Smaller NPs usually have a greater number of surface atoms, resulting in a larger surface area for contact with bacteria. Consequently, smaller NPs exhibit greater activity than bulk. Thus, a key focus of research should be the development of new anti-microbial substances or the alteration of those already in existence. It is thus of great interest to create anti-microbial materials and surfaces for use in healthcare, food, biomedicine, and personal care applications. As bacteria develop resistance to conventional antibiotics nanoparticles have arisen as potential drugs for treating bacterial infections [3]. The nanoparticles produced through various methods can be adjusted at the atomic or molecular level according to the properties sought. CdO nanoparticles, which are n-type semiconductors, possess unique optical and electronic characteristics including a wide band gap, high visible-region transmission, and low electrical resistivity. CdO nanoparticles are utilized in various biomedical domains, such as bioimaging, biosensors, and drug delivery systems. It is worth mentioning that their lower cytotoxicity relative to that of Cd (NO3)2 improves their safety profile for medical applications. Moreover, due to their exceptional electrochemical characteristics, CdO nanoparticles are appropriate for research purposes [4]. Various methods have been suggested for synthesizing cadmium nanoparticles, such as evaporation, spray pyrolysis, chemical techniques, metal-organic chemical vapor deposition, sputtering, sol-gel spin coating, and laser ablation.

Laser ablation in deionized water has been widely used for the synthesis of metallic, bi-metallic, and tri-metallic nanoparticles, as well as metal oxides. A significant advantage of using laser technology for synthesizing nanoparticles is the production of nanoparticles that have a greatly enhanced purity level, distinct surface charge characteristics, and more straightforward preparation methods in comparison to traditional chemical methods [5]. Several laser parameters, including laser power, can be used to manipulate the size distribution and shape of nanoparticles; energy density (fluence), size of the beam spot, speed of scanning, medium for ablation, and wavelength [6]. According to previous studies, CdO prepared laser ablation has good antimicrobial activity, as Othman A. F. conducted an investigation in 2024 into the effectiveness of (CdO nanoparticles) synthesized through the pulsed laser ablation method (PLA) at various laser power settings (200, 300, 400, and 500 mJ) for inhibiting biofilm formation by two types of bacteria: Staph bacteria and E. coli. The cadmium nanoparticles showed growth inhibition effects on all bacterial pathogens, with a maximum inhibition of 16 mm for Staph bacteria and 19 mm for E. coli at 300mJ [7]. In 2025, Abdalameer, N. K and Khaleefa T. A. utilized the pulsed laser ablation in liquid (PLAL) method to produce CdO nanoparticles (NPs) with a laser energy of 250 mJ. The study's findings show that the synthesized CdO NPs have anti-bacterial properties, making them effective in eliminating pathogenic bacteria and fungi, with maximum values of 21 mm and 15 for Staphylococcus aureus and Candida, respectively [8]. In this work. In this work, we investigate how varying the pulse energy in laser ablation in liquid affects the properties and antimicrobial activity of CdO thin films.

2. Materials and Methods:

CdO powder (99%) was sourced from "BDH Chemicals Ltd in Poole, England". 3 grams of CdO powder were compressed into a 1.6cm diameter round pellet by a hydraulic compressor and 11 tons of pressure. we used 100 pulses at a frequency of 5 Hz to shine the fundamental radiation of a "Q-switched Nd:YAG laser (1064 nm) with energies 360 and 560 mJ at a glass jar that had 6 ml of distilled water and CdO pellet. With the help of a 110 mm lens, the laser beam was focused on the surface of the pellet. During the laser irradiation, the vessel was turned so that the nanoparticles that were made wouldn't get in the way of the laser radiation hitting the target surface (figure (1) shows the preparation of CdO solutions). Drop casting method has been used to deposit a CdO film on glass sample, as figure (2). The films were deposited by drop-casting the colloid onto glass substrates. Three glass slides (2 * 2 cm^2) were subjected to a 30-minute ultrasonic cleaning with deionized water, and then immersed in CH_3OH for an additional ten min. They were finally air-dried to avoid impurities and surface contaminations. Using the drop casting technique, the CdO solution (roughly 10 drops) was applied to glass slides at a temperature of about 80° C [4]. The characteristics of CdO produced by laser ablation in water were examined through various methods, including X-ray diffraction "D2 phaser, Bruker, Karlsruhe, Germany" using CuKa1 radiation at a wavelength of 0.154060 nm, generated at 30 KV and 10 mA for 2θ values ranging from (-3° to 160°), SEM "JSM-7600F, JEOL, Tokyo, Japan", AFM morphologies "CSPM-Scanning probe microscope",UV-VIS analysis "UV-1601, Shimadzu Corporation Ltd., Tokyo, Japan", FTIR "FT-IR 8400S spectrophotometer, Shimadzu Corporation Ltd., Kyoto, Japan" over the wavenumber range of (4000–400 cm^{-1}), and biological assessment of CdO via the Agar method.

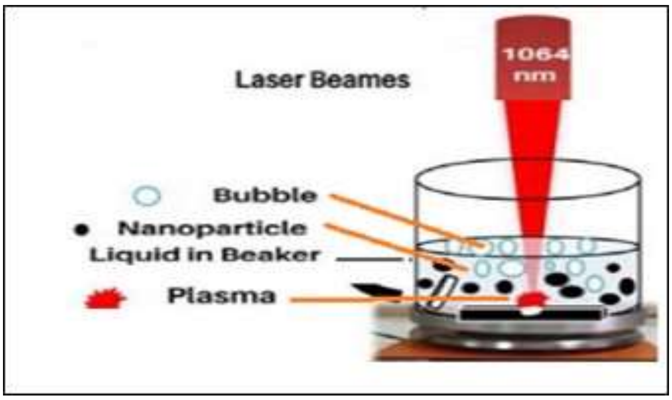

Figure.1: preparation of CdO solutions by laser ablation at different energies (360mJ and 560mJ), and the resulting solutions.

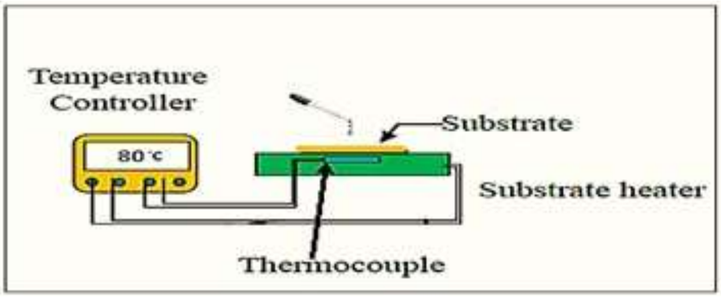

Figure 2: Experimental set up the method of drop casting.

3. Anti-microbial activity of CdO NPs

Urine samples from "patients with urinary tract infection (UTI) at Al-Imamian Al Kadhimiyain Medical City in Baghdad" were used to isolate harmful bacteria like Staphylococcus aureus and the fungus Candida albicans [9]. The fungus Candida albicans and the bacteria Staphylococcus aureus were used to test the anti-microbial qualities of CdO NPs. Bacterial and yeast isolates were cultivated for 24 hours at 37 °C in nutrient broth and yeast broth, respectively, to produce working cultures of 10^8 CFU/ml. 100 µl of CdO NPs were dispensed into each well using a sterile cork borer (diameter = 6 mm). After that, the plates were incubated for twenty-four hours at thirty degrees Celsius for Candida albicans and thirty-seven degrees for bacteria. To evaluate the inhibitory effect of CdO nanoparticles on bacteria and fungi, the diameters (in mm) of the inhibition zones in the wells were measured using a ruler [10-11].

4. Results and Discussion

The XRD patterns of CdO prepared at 360 mJ and 560 mJ and deposited on glass substrates are displayed in Figure (3). According to "(JCPDS No. 05-0640)," the XRD patterns show peaks with planes (111), (200), (220), and (311), which were in close agreement with the results of earlier investigations [12,13]. Peaks clearly show that the CdO prepared by two different energies has an FCC structure and is polycrystalline; furthermore, higher laser energy clearly results in higher crystallinity of the CdO films that are deposited. The crystallite size values shown in Table 1 can then be used to further explain this. Additionally, the absence of any additional impurity peaks suggests that the produced nanoparticles are pure across the board. As laser energy increases, there are few variances in the positions of the peaks due to the difference in lattice uniform strain with the variation of crystalline size. It is also clear that as the laser energy increases, the intensity of the peak rises and its width decreases, indicating an increase in crystallinity and crystalline size. The crystalline size increases with laser energy, allowing for the removal of larger masses of the target material. Additionally, the higher energy promotes the merging of small particles [14]. The Scherrer equation (1) was applied to determine the crystallite size (D) of CdO [15]. The size was determined to be 28.30 and 40.16 nm for 360 mJ and 560 mJ respectively.

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \quad \dots (1)$$

Where λ is the laser wavelength, β is the full width at half maximum and θ represents the diffraction angle.

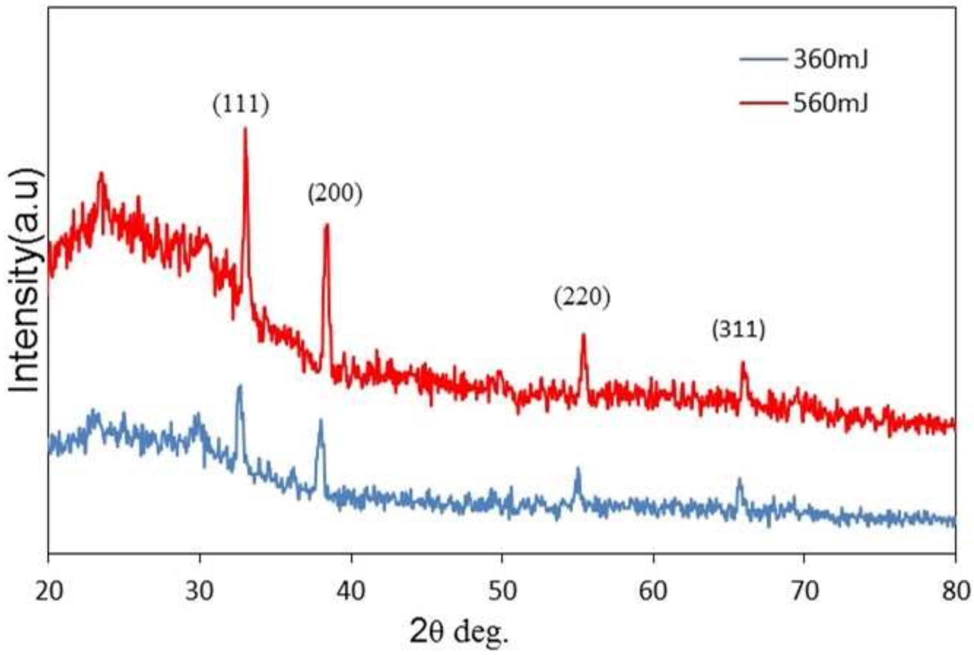

Figure.3: XRD analysis of CdO films prepared at (360mJ and560mJ).

Table 1: The XRD results for CdO films prepared at different laser energies.

Sample	2Theta (deg)	FWHM (deg)	Crystalline size (nm)
CdO at 360 mJ	32.69 38.94 55.04 65.69	0.33 0.38 0.34 0.24	28.30
CdO at 560 mJ	32.99 38.39 55.39 65.89	0.41 0.27 0.18 0.16	40.16

Figure (4) shows the surface morphology and size distribution of the produced CdO NPs at two distinct energies, 360 mJ and 560 mJ. The average diameter increased from 82.99 nm to 360 mJ and 117 nm for 560 mJ, indicating that the film is homogenous and adherent, according to the AFM image. According to Table (2), the surface roughness for 360mJ and 560mJ were 1.18 nm and 2.68 nm, respectively. The average particle diameter increases with the increase of laser energy. AFM indicates larger grain growth and higher surface roughness at 560 mJ not higher homogeneity. Of the produced films. This outcome aligns with the findings from XRD examination regarding grain size calculation. It noted that the morphology and size of CdO nanoparticles change with increasing laser energy. The surface of a target immersed in water is affected by the interaction with a laser pulse, which influences the characteristics of the plasma produced in the area where the laser pulse meets the target's surface. The characteristics of the induced plasma have a significant influence on the size and morphology of NPs. As the laser power increases, more thermal energy is transferred to the material. This can cause particles to fuse together and create larger particles, resulting in bumps or irregular structures on the surface.

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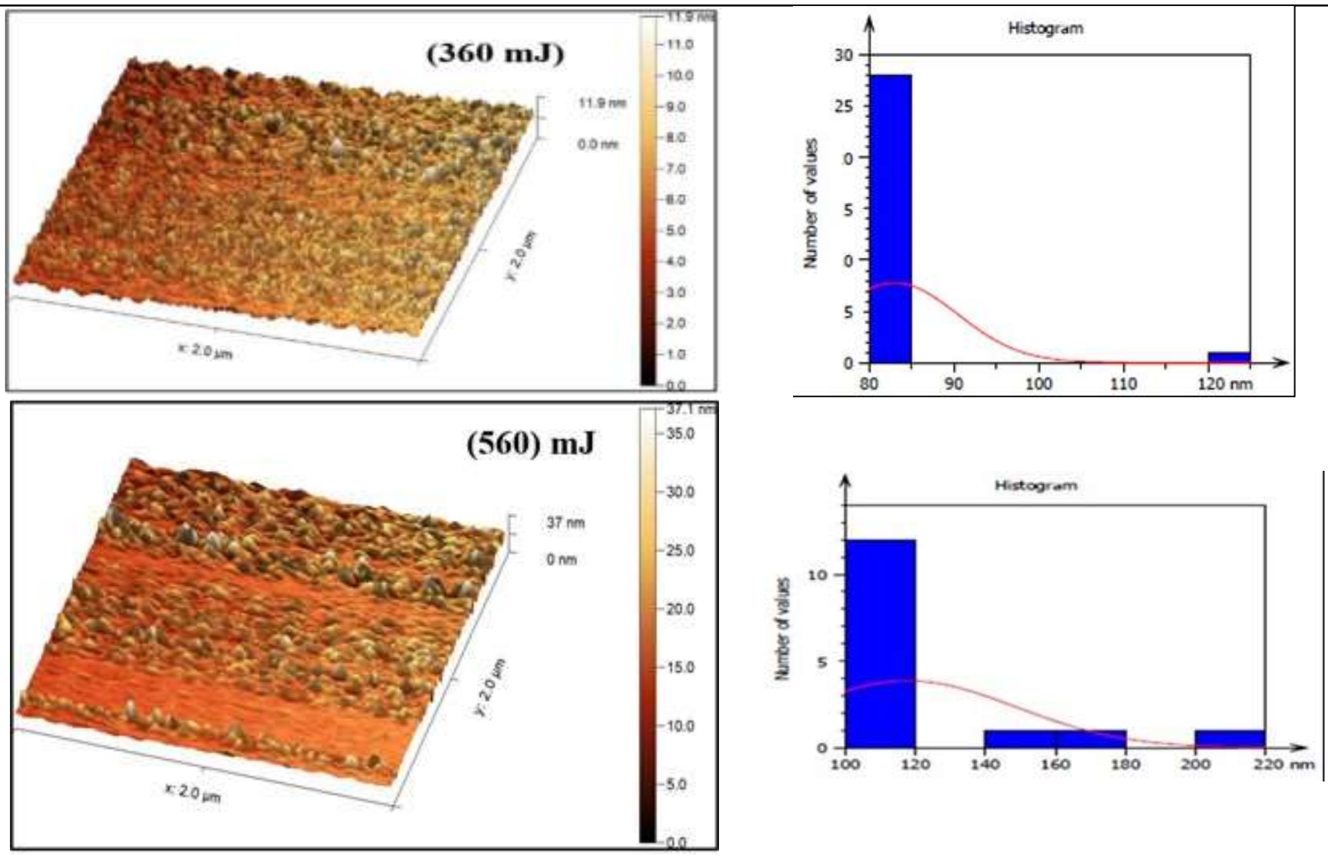

Figure. 4: AFM image of the prepared CdO films at 360mJ and 530mJ

Table 2: The AFM results for CdO films at different laser energies

CdO	Mean roughness	RMS grain size	Mean grain size
360mJ	1.18 nm	1.47 nm	82.99 nm
560mJ	2.68 nm	3.58nm	117nm

In Figure (5) FESEM images of the cadmium oxide that was made by laser ablation with energies of 360 and 560 mJ in water and deposited by drop casting. The whole structure is made up of a network of very small spherical particles that are strongly connected to each other at energy 360 mJ, while at 560 mJ the particles are of different sizes and shapes. The pulsed laser's higher energy caused the particles' size to gradually increase, where the smallest diameter particle was 13.94 for 360mJ and 21.78 nm for 560mJ with some of the aggregate resulting from the accumulation of small particles. Aggregates can diminish the anti-microbial efficacy of cadmium oxide by reducing the particles' surface area and altering their interaction with microbes. This aggregates increase of the particle size to a few micrometers, which may influence the particles' capacity to penetrate the cell wall.

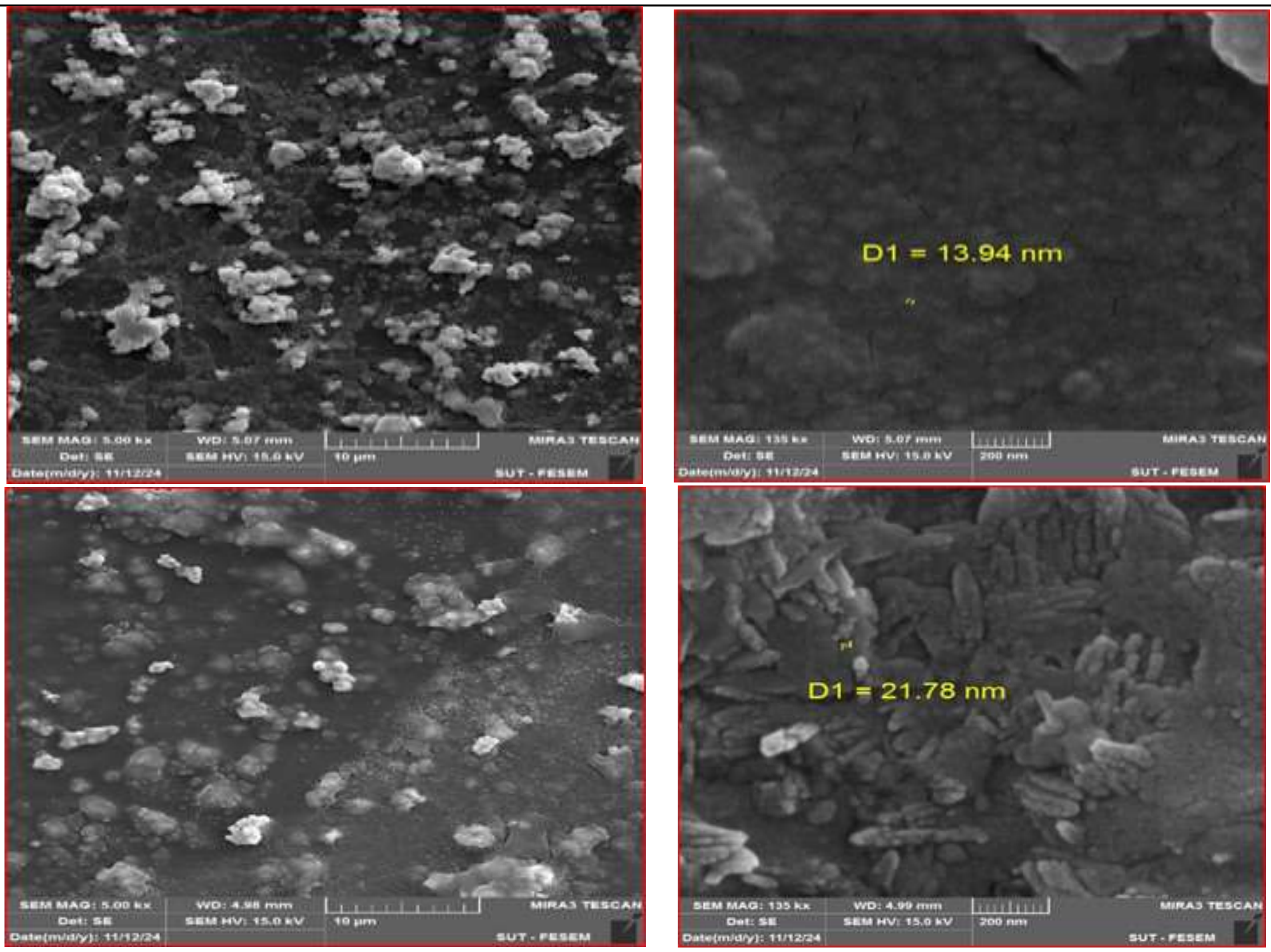

Figure 5: SEM images of the prepared of CdO films at 360mJ and 560mJ.

Figure (6) shows the absorption curves for CdO that was made with 360 mJ and 560 mJ of laser energy. Because the ablated matter from the target rises when the laser energy is increased, the absorbance of CdO generally increases as well. Longer wavelengths result in higher transmittance. The CdO nanoparticles produced by laser ablation at 560 mJ had an absorption peak at 294 nm, according to UV-Vis-NIR spectroscopy. In both the VIS and NIR spectrums, CdO samples show excellent transparency. The highest optical transmittance value, approximately 94%, was observed for CdO at a lower energy of 360 mJ. The Tauc relation (equation 2) used to find the straight band gap energy [16].

$$\alpha h\nu = A\left(h\nu - E_g\right)^{0.5} \quad \dots(2)$$

where, α is the coefficient of optical absorbance, $h\nu$ is the photon energy, Eg is the direct band gap and A is a constant. The band gap energy of the CdO NPs is reported as 3.8 and 3.6 eV as shown in figure (7). It illustrates how the energy gap decreases as the source energy increases for samples, a phenomenon resulting from the increase in crystal size or changes in the lattice constant, along with grain development and a reduction of defect states near the bands. This lowering of Eg occurs due to an inverse relationship between grain size and nanoscale dimensions [17]. In our study, the energy gap values of CdO NPs exceeded those of bulk, with the high values attributed to the quantum size effect. The quantum size effect describes the occurrence whereby, when the size of a particle decreases to a specific value (equal to or less than the Bohr radius), the electron energy levels near the Fermi level transition from a quasi-continuous state to a discrete one. This results in energy level splitting or an expansion of the energy gap [18].

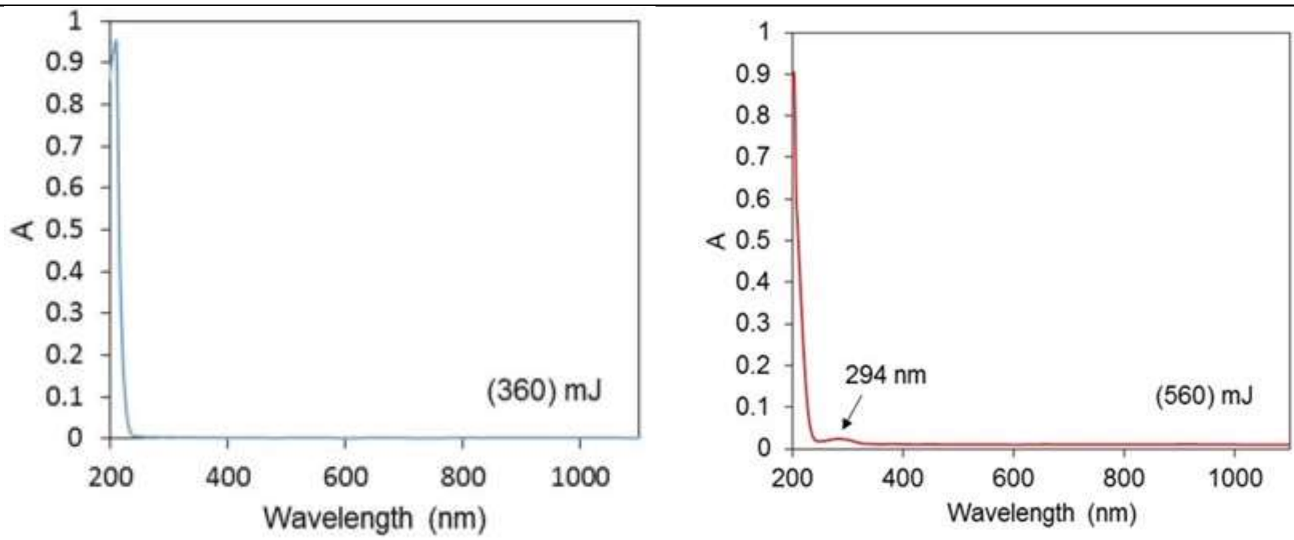

Figure 6: UV-VIS absorption spectrum of the prepared CdO films at 360mJ and 560mJ

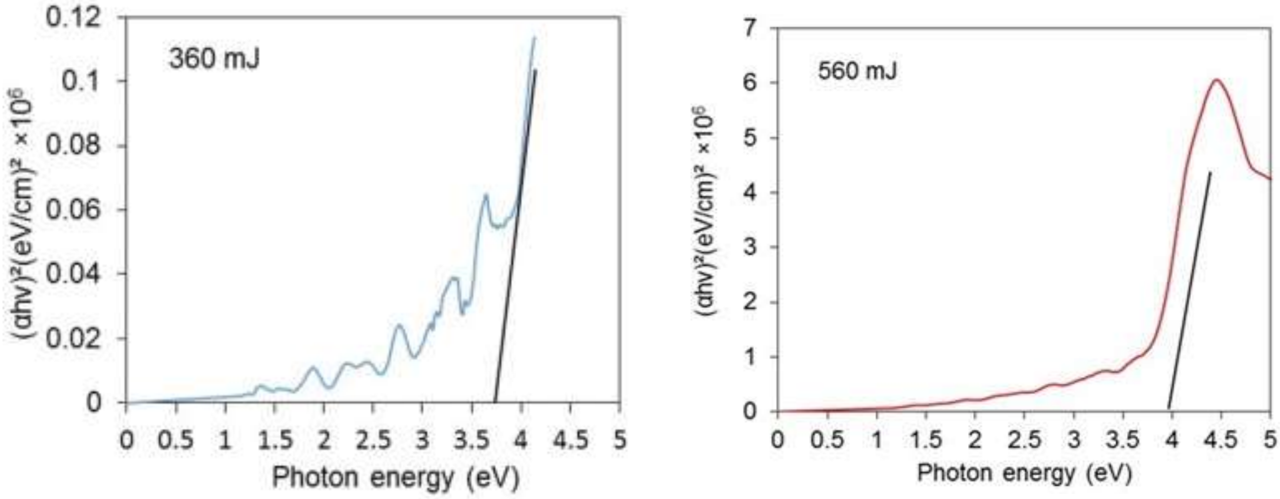

Figure 7: Tauc relation band to determined energy gap of CdO films prepared at different laser energies

(FTIR) is a good way to find out what functional groups are in the samples being studied. In the 400–4000 cm^{-1} range, the functional groups were found. In Figure (8), you can see the FTIR spectrum of the CdO at different laser energies (360 mJ and 560mJ). The signals at 3444, 3448 cm^{-1} show that the O-H group is present in the water molecule that has been bound [19]. The C-H asymmetric stretched band can be seen at 2928 and 2925 cm^{-1}. The synthesized materials have several hydroxyl groups attached to their surfaces that make them more photo catalytic. This can be seen in the H_2O bending mode that corresponds to the band at around 1638 cm^{-1}. The main peaks of absorption at 1650 cm^{-1} show how the carboxyl acetate group (C=O) is stretching unevenly. Molecular CO_2 and water in the air during the sample production process may have made this bond. Between 1421 and 1469 cm^{-1} is where the peak for the C-C stretching vibration is found. The strong peak in the 1386 cm^{-1} range is caused by the C–H stretching vibrational mode. In the FTIR spectrum, the unique absorption at 1168, 1151, and 1290 cm^{-1} is due to C–O. The normal frequency range for the CdO mode is 400–1000 cm^{-1}, which is where most metal complexes are likely to be [20-22].

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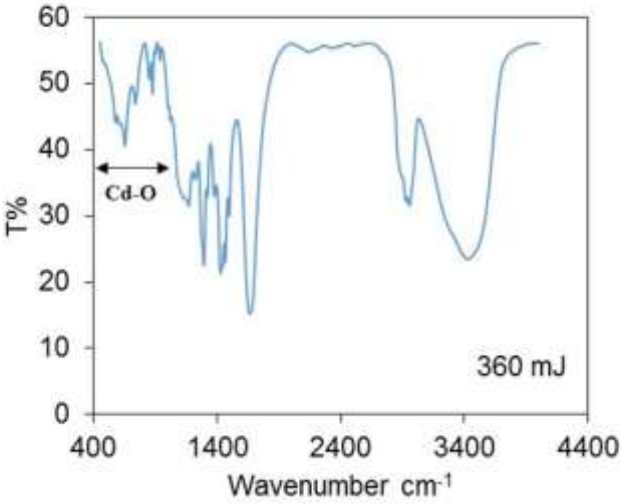

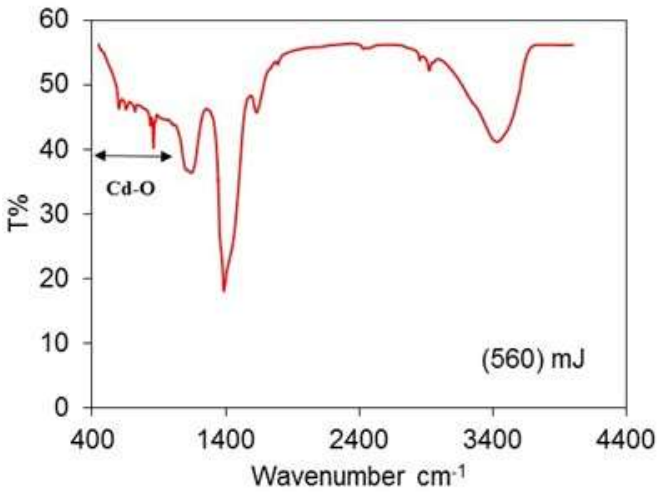

Figure 8: FT-IR spectra of the prepared CdO films at different energies 360mJ and 560mJ.

The Anti-bacterial properties of nano-particles have received a lot of interest lately, particularly when it comes to microorganism resistance [23, 24]. Because they have more surface atoms, smaller NPs can come into touch with bacteria on a greater surface area. Larger NPs are therefore less active than Small NPs. Microorganism susceptibility may vary depending on the type of structure of the outer wall surrounding the cell that controls the permeability. Lipopolysaccharides found in Gram-negative bacteria's outer membrane block Entry of relatively large molecules and some hydrophilic substances. Gram-positive bacteria, on the other hand, either produce very little or no lipopolysaccharides. These bacteria's cell walls additionally include glycerin chains which has a negative charge (teichoic acid) and several peptidoglycan layers. Cadmium may cause cell wall rupture when it interacts with the cell wall's negative charge. Furthermore, NPs might affect the bacterial growth-signaling system, which would reduce cell viability [25]. CdO NPs inhibit bacterial cells because they make substantial interaction with the thiol groups existing in the critical bacterial enzymes. As CdO NPs move across the membrane, an interaction occurs between its positive electrical charge and the negative proteins charged existing on the external surface of the bacterial cell, leading to a major change in the cell internal structure [26]. ROS are produced when the NPs liberated ions interact with the thiol groups in proteins, disrupting cell structure and, in turn, cell function. Cell lysis results from the reduction of dynamic transport, dehydrogenase, and enzymatic inactivity in the periplasm zone caused by CdO NPs binding the protein layer. This inhibits the fabrication of proteins, RNA, and DNA. CdO NPs were created by Shivashankarappa et al. using Escherichia coli. When tested against a number of foodborne pathogens, such as Pseudomonas aeruginosa, Bacillus licheniformis, Escherichia coli, and Aspergillus flavus, the produced CdO NPs had stronger Anti-bacterial and antifungal activity than traditional treatments [23]. The anti-microbial activity of CdO nanoparticles that prepared at different energies 360mJ and 560mJ were carried out by agar well diffusion method against Staphylococcus aureus and candida albicans. The anti-microbial ZOI plate photos of CdO nanoparticles are shown in Figure (9). CdO NPs demonstrated anti-bacterial activity against Candida albicans and Staphylococcus aureus. As laser energies increased from 360 to 560 m J, so did the anti-bacterial property and the diameter of the bacterial inhibition zone. Due to an increase in CdO concentration at high laser energies, it was found that the inhibition zones of Staphylococcus aureus and Candida albicans were 12 mm and 19 mm at 360 mJ, respectively, while it recorded 14 mm and 20 mm at 560 mJ. The ability of CdO nanoparticles to readily impregnate into bacterial cell walls accounts for their anti-microbial activity. The negative charge of the proteins on the bacterial surface interacts electrostatically with the positive charge of the CdO nanoparticles. Reactive oxygen species are created when the ions released by the CdO nanoparticles interact with the thiol groups of the proteins in the cell wall, killing the cells [27].

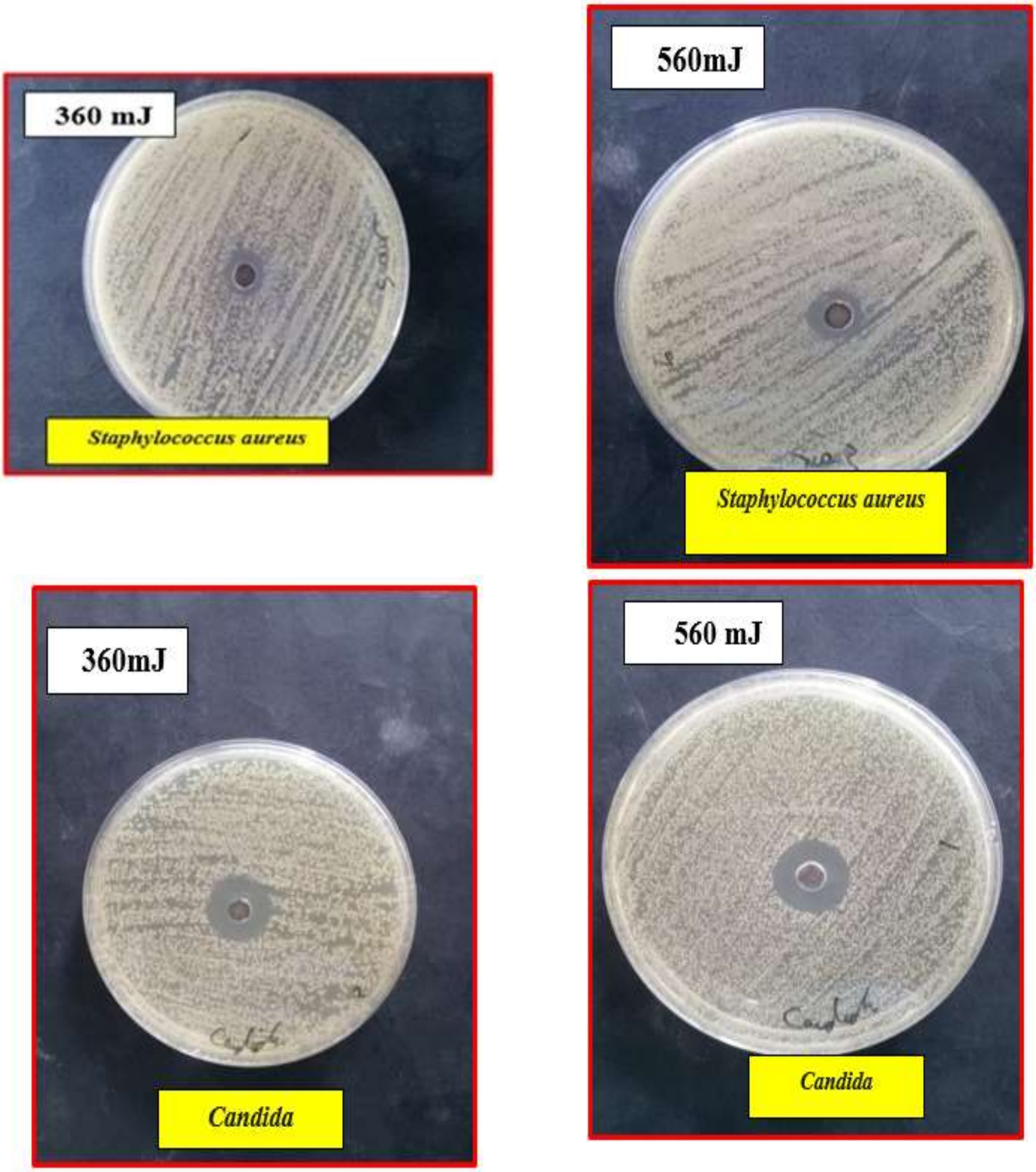

Figure 9: Anti-microbial activity of CdO nanoparticles against Staphylococcus aureus and candida albicans

5. Conclusion

Laser ablation is a secure and effective method for producing stable and highly pure CdO Nano colloids in distilled water. The study has successfully achieved its objectives in producing CdO thin films using the PLAL process (at different energies) and investigating their potential applications in the field of biomedicine. XRD pattern indicates that the intensity of the diffraction peaks of CdO escalates with higher laser energy, where the crystalline size increase. The data of SEM and AFM illustrate the alterations in surface shape and optical properties with differing energy levels. In biomedical applications, increasing particles size and aggregation affect the effectiveness of prepared material in inhibiting microbial growth. Cadmium oxide (CdO) nanoparticles exhibit remarkable anti-microbial activity against bacteria and fungi, and this activity was successfully demonstrated using the well diffusion method. The highest inhibitory activity was observed against both bacteria and fungi when prepared using high energy due to increase the concentration of CdO. our results agree with the previous studies mentioned in this paper, where increases the energy, led to more ablation in the material which increases the concentration, but that affects the particles size and may be cause a shift in the absorption edge towards long wavelength.

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Conflicts of Interest: The authors declare no conflict of interest.

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