Synthesis and characterization of tellurium oxide nanoparticles using pulse laser ablation and study their antibacterial activity

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Abstract: In this study, tellurium oxide (TeO2) nanoparticles were synthesized via PLA of tellurium targets with a 1064 nm laser in distilled water. The morphology and the particle size of the nanoparticles were characterized by scanning electron microscope (SEM) and atomic force microscope (AFM) respectively. The optical properties were studied by UV-Visible Spectrophotometer. Tellurium oxide nanoparticles with diameters of 55 nm were formed in a colloid solution. The UV–vis spectrum of the material shows a strong peak of around 200 nm. In addition, the morphology of gram-negative bacteria and gram-positive attachment of TeO2 nanoparticles was studied by using SEM measurement. The activity of TiO2 nanoparticles toward the inhibition and removal of Escherichia coli, Proteus, Pseudomonas bacteria and Staphylococcus aureus was investigated and discussed.

Keywords: Antibacterial, Tellurium oxide, Antimicrobial, TeO2 nanostructure.

1. Introduction

Various nano-sized materials show unique chemical, optical, biological, photoelectrochemical and electronic properties [1,2]. TeO2 belongs the high index of refractive materials which transmit in infrared region; so that it is very interesting for optical applications. The energy gap of TeO2 is about 4.05 eV. It has also been found to exhibit Raman gain up to 30 times that of silicon dioxide; as a result, it is very successful in fiber optic amplification [3, 4]. Because of their beneficial photo-elastic properties TeO2 crystals a good in acousto-optic devices, also due to their low light absorption, high optical homogeneity and high optical damage resistance [3, 5]. TeO2 is an interesting material both in its amorphous and crystalline forms, such as α-TeO2 finds to have applications as an optical storage material, x-ray detectors, laser devices, optical storage material, gas sensors [6-10] and propane oxidation catalysts [11, 12]. Various techniques have been used for TeO2 thin films preparation such as reactive dip-coating, sputtering [13-15], and laser ablation [16-18]. This work represents the activity of TeO2 NPs toward the removal and inactivation of Escherichia coli, Proteus vulgarus, Pseudomonas spp and Staphylococcus aureus was examined. We have successfully produced TeO2 NPs by in distilled water via PLA method. The morphological and optical properties were calculated for TeO2 NPs on quartz substrates.

2. Experimental

2.1 Synthesis method of TeO2 Nanoparticles and characterization
Tellurium oxide nanoparticles, which is a combination of focused pulsed laser (type HUAFEI) of 1064 nm at energy (600) mJ per pulse of a piece of tellurium metal plates (purity: 99.999%) placed in the quartz vessel containing 2 ml of DW [19-21]. The number of laser pulses applied to the Te metal about 50 pulses. Philips PW 1050 X-ray diffract meter of 1.54 Å from Cu-ka used for X-ray diffraction measurements. The examination of the morphological characteristics of TeO₂ nanoparticles using atomic force microscope (AFM) type (Digital Instrument microscopy imaging with an AFM tapping mode) to observe microstructure. UV-Visible Spectrophotometer (Metertech SP 8001), in the range of (190-1100) nm, were used to study optical properties [21].

2.2 Antibacterial activities of TeO₂Np

Antibacterial activity of TeO₂NP was determined by agar- well diffusion method [22, 23]. Test strains, *Escherichia coli* (E.coli), *Proteus vulgarus*, *Pseudomonas* and *Staphylococcus aureus* strains were kindly provided by the lab of biotechnology- department of applied science, University of Technology - Baghdad. The cell culture suspension of each bacterium was adjusted by comparing to McFarland solution as control and that's equal to 10⁷ CFU/ml [24]. Muller – Hinton agar plates were seeded with 0.1 ml of suspensions of each bacterial culture separately, then wells of 5 mm diameter were prepared in each agar plate. The wells were loaded with 100 µl of different concentrations of TeO₂ nanoparticle solution. Plates were incubated at 37 °C for 24 hours, and then the diameters of the inhibition zones were measured in millimeter [22, 24].

3. Results and Discussion

3.1 Morphological and structural analysis of TeO₂ NPs

3.1.1 SEM analysis

The surface morphology of TeO₂ NPs was analyzed in SEM using Inspect S50, FEI Company, Netherland microscope. It is observed that the surface morphology of TeO₂ NPs is quite homogeneous, the shape of the molecules is random and molecule sizes changes between 100 nm to 1 µm (Figure 1).

![SEM images of TeO₂ NPs at large and small magnifications with a scale bar of 10 and 5µm respectively.](image)

3.1.2 AFM analysis
Atomic force microscope (AFM) images of TeO$_2$ nanoparticles film drop caste on a quartz substrate prepared via Nd-YAG laser. Figure 2 presents (2D), (3D) AFM images and the grain size distribution shows in (c). The AFM parameters also calculated such as average grain size, root, mean square (Sq) and average roughness (Sa) are 55 nm, 4.51 nm, and 3.23 nm respectively.

3.1.3 XRD analysis

X-ray Diffraction (XRD) was applied to analyze the TeO$_2$ NPs drop-casting on quartz substrate via Laser ablation technique as presented in Figure 3. TeO$_2$ nanoparticles revealed a strong and broaden peak related to the (100) reflection plane. Another small peak corresponding (101).the whole of the reflections in Figure 3 can be indexed on a tetragonal tellurium oxide with lattice constants of $a = b = 0.480$ nm and $c = 0.761$ nm (JCPDS No. 78-1713). [24]. This result is confirmed by other literature's data [15-27]. The crystallite size (grain size) (D) were calculated using the Scherrer formula from the full-width at half-maximum (FWHM) ($\Delta$) (Red) [27-29] with other parameters are listed in the table (1). Where the wavelength of the X-rays and Bragg angle are $\lambda$ and $\theta$ respectively.

$$D = \frac{0.9 \lambda}{\Delta \cos \theta} \ldots \quad (1)$$

**Table (1)** TeO$_2$NP parameters for preferred orientation (100).
3.2 Optical properties
The band gap (Eg) values of tellurium oxide nanoparticle film are obtained from the following relation [30-32].

\[ a\nu = A(\nu - E_g)^{0.5} \quad \ldots \quad (2) \]

Where: A is a constant, \( \alpha \) is the absorption coefficient, \( \nu \) is the photon frequency and \( h \) is Planck’s constant. The absorption spectrum of tellurium oxide nanoparticles in a colloidal is shown in Figure 4a. To have a strong absorption peak at (200) nm, suggesting the formation of TeO\(_2\) nano colloidal solution. The band gap energies (Eg) of the prepared tellurium oxide nanoparticles found to be about 5 eV as shown in figure 4 (b) which is larger than the value for the bulk TeO\(_2\) about 4.02 eV. This due to the band gap of the semiconductors has been found to be particle size-dependent [33, 34].
Figure 4. The Absorption spectrum of TeO$_2$ NPs and Bandgap.

3.3 Antibacterial activity of TiO$_2$ NPs application

The Antibacterial potential of TeO$_2$ was evaluated according to the presence of a clear inhibition zone and its diameter around the wells in each plate of the bacterial strains. The results revealed that TeO$_2$ is a potent antimicrobial agent against two of the tested microorganisms. The maximum inhibition zone diameter was obtained in E. coli with diameter 48 mm in 2.0 mg/ml concentration. Similarly, the same concentration (2.0 mg/ml) showed maximum inhibition zone with a diameter of 40 mm in S. aureus, while there was no antimicrobial activity against Proteus vulgarus, Pseudomonas as explained in Table 2. Figure 5 shows inhibition zones of a) Escherichia coli, b) Staphylococcus aureus, c) Proteus vulgarus and d) Pseudomonas strains using the good diffusion method.

Table (2) Antimicrobial activities of different concentrations of TeO$_2$ NPs on test bacterial strains.

| Test organism   | Inhibition zone diameter (mm) for concentration (mg/ml) |
|-----------------|----------------------------------------------------------|
|                 | 0.5 | 1.0 | 1.5 | 2.0 |
| E. coli         | 26.5 | 28 | 32 | 48 |
| S. aureus       | 24 | 28 | 32 | 40 |
| Proteus vulgarus|     |    |    |   |
| Pseudomonas     |     |    |    |   |
From the above consequences, it is generally concurred that the TeO$_2$ nanoparticles have an issue as an antibacterial effect. The presumed explanation is that both gram-negative and gram-positive bacteria carry a negative charge on their cell wall, so there will be an affinity to the positive ions most nanoparticles release, and that will increase the uptake of ions into the bacterial cells and that will lead to intracellular damage. Another presumed mechanism of action of these nanoparticles on a bacterial cell is that the nanoparticles inhibit proteins or Muclopeptides synthesis, but the mechanism of genetic materials from this material is not fully understood. There was no antibacterial effect of TeO$_2$ nanoparticles against *Proteus vulgarus*, *Pseudomonas* and that differs from previous studies. Few studies reported that bacterial strains that multidrug and heavy metal resistant bacteria like *Proteus, Pseudomonas* are unaffected by nanoparticles, which affect other bacteria, and that may be due to producing extracellular matrix that agglomerate and deactivate the nanoparticles [35-38]. Figure 6 a, b shows SEM images of the Gram-negative Bacteria (*E. coli*) and gram-positive (*Staphylococcus aureus*) attachment of TeO$_2$ nanoparticles at different magnifications, respectively. It is clear that the TeO$_2$ nanoparticles seen in the SEM image consist of a number of crystallites.
Conclusion
The results confirm the formation of TeO$_2$ NPs with energy band gap of 5.02 eV for the TeO$_2$ nanoparticles synthesized via PLA method. This result, investigates the good performance of TeO$_2$ nanoparticles colloidal toward the removal and inactivation of *Escherichia coli*, and *Staphylococcus aureus* respectively.

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