



Original Research Article

Biosynthesis, Characterization, and Applications of Bismuth Oxide Nanoparticles Using Aqueous Extract of Beta Vulgaris

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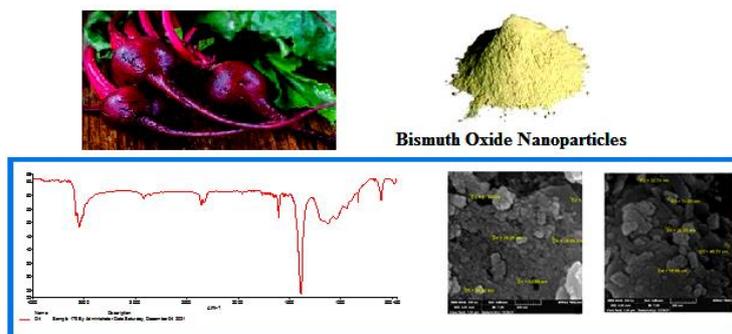
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ABSTRACT

Bismuth oxide nanoparticle Bi₂O₃NPs has a wide range of applications and less adverse effects than conventional radio sensitizers. In this work, Bi₂O₃NPs (D₁, D₂) were successfully synthesized by using the biosynthesis method with varying bismuth salts, bismuth sulfate Bi₂(SO₄)₃ (D₁) or bismuth nitrate. Penta hydrate Bi(NO₃)₃.5H₂O (D₂) with NaOH with beta-vulgaris extract. The Bi₂O₃NPs properties were characterized by different spectroscopic methods to determine Bi₂O₃NPs structure, nature of bonds, size of nanoparticle, element phase, presence, crystallinity and morphology. The existence of the Bi₂O₃ band was verified by the FT-IR. The Bi₂O₃ NPs revealed an absorption peak in the UV-visible spectrum, with energy gap E_g = 3.80eV. The X-ray pattern (D₁) matching that of card of COD File-No-96-152-6459 indicating the presence of homogeneous Bi₂O₃NPs, Scanning Electron Microscopy (SEM) displayed shaped monoclinic phase with average diameter 30.28 nm. The size, structure and composition of synthetic Bi₂O₃ Nps were determined using the (EDX) pattern. The XRD pattern (D₂) corresponds to JCPDS File No. 27-50. The SEM of D₂ showed crystalline rhombohedral phase with average diameter 34.89 nm. The EDX for both (D₁, D₂) samples reveals an aggregation of thin sheet cluster. The antibacterial activity of Bi₂O₃NPs from (D₁, D₂) was tested against (G⁻) *Escherichia coli* and (G⁺) *staphylococcus aureus*. All of these clinical pathogens were examined for antifungal activity against *Candida albicans fungus*, and the results were compared with the standard medication. The adsorption experiment was successfully conducted on the following metal ions (M⁺² = Co, Ni and Cu), where the results proved removal simultaneously from water using Bi₂O₃NPs (D₁, D₂) based on the affinity of three metal ions and Bi₂O₃ NPs surface shape. The removal efficiencies of mixed (M⁺² = Co, Ni and Cu) ions for D₁ were 89.68%, 85.56% and 94.5%. The removal efficiencies for D₂ were 93.3%, 87.7% and 88.54%, respectively.

GRAPHICAL ABSTRACT



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Introduction

Metal oxide NPs (MO – NPs) have proved to be particularly effective in inhibiting bacterial growth [1-3] and antibacterial resistance, treatment to remove multiple heavy metal ions from waste water by using Fe_3O_4 NPs, gas sensor, and catalyst [4-6]. Manufacture oxide nanoparticles with specified sizes and shapes, green synthesis is a challenge [7, 8]. Because of their ease of use, abundant biodiversity and eco-friendly processes, biosynthesis technologies offer a distinct advantage over other traditional synthesis approaches. [9, 10]. *Mentha Pulegium* (pennyroyal) leaf extract was used to make silver nanoparticles for antibacterial use [11, 12]. Bi_2O_3 NPs was synthesized via green synthesis using *Mentha Pulegium* aqueous extract and it showed antibacterial activities [7]. Researchers have synthesized Bi_2O_3 NPs by using *Jotopha multifidi* leaf extract. The Bi_2O_3 NPs that were generated contain a monoclinic structure, a 3.34 eV optical band gap, agglomeration morphology and a particle size of 17.26 nm [13]. At room temperature, the researchers biosynthesized Bi_2O_3 NPs in a size range of 5-8 nm by fungus – *fusarium oxysporum* with $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ [14]. In the present study, we have developed a facile green synthesis method for preparation of Bi_2O_3 NPs using *Beta vulgaris* extract. The objective of this research was to achieve the goals of green synthesis for potential application and antibacterial activity used as adsorbent to metal ions.

Materials and Methods

Sample collection

Beta vulgaris was taken from a nearby source and marked. Bismuth nitrate. penta hydrate $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and Bismuth sulfate, $\text{Bi}_2(\text{SO}_4)_3$ were purchased from Germany/ Merck, NaOH form Alpha India/ Alpha Chemica, Ethanol from Spain/ RBL, also Cobalt sulfate CoSO_4 , Copper sulfate. Penta hydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and Nickel sulfate. Hepta hydrate $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$. All chemicals employed without additional purification. Various spectroscopic and microscopic approaches were used to synthesize and identify

Bi_2O_3 NPs as follows: A Sensitive Electronic Balance type RADWAG, model / As 220 /C / 1, Magnetic stirrer, Centrifuge type PLC, (4000 – 4500 rpm) [(66–90 w) power: 110 L 60 Hz, 230 L 50 Hz], Electric oven type (FAITHFUL) model – WHL. 25 AB, Shaking water bath type (SCL FINETEDI), Tape measure of PH, (UV-vis) kind, (160/ Uv) Shimadzu spectroscopy, FT-IR (8500S) type Shimadzu at university of Baghdad / Collage of science and (XRD) X-ray diffraction type (Phillips/ Holland) were examined in the laboratories of the (center of examinations) at Baghdad. SEM with Field Emission (FE) type (Hv 300/ Ziess sigma -Germany) and X-ray energy dispersion device (EDX) were applied. Transmission electron microscope (TEM) (kind/ EM10C-100Kv Germany) was put to test at Kashan University in Iran. The antimicrobial activity of the synthetic Bi_2O_3 NPs was tested against two reference bacterial strains, (G^+) *Staphylococcus aureus*, and (G^-), *Escherichia coli* and, as well as *Candida albicans fungal*, using the disc diffusion method in a nutrient medium (jellose medium) type Muller Hinton agar, and the same method was used for antifungal activity using the nutrient medium (agar) potato dextrose PDA.

Bi₂O₃ NPs preparation by (Beta vulgaris extract) with Bi₂(SO₄)₃ and Bi(NO₃)₃ · 5H₂O (D₁ and D₂)

Fresh *Beta vulgaris* must be first prepared. *Beta vulgaris* was rinsed with tap water to eliminate any contaminants and dried at 37 °C for one day. About 20 g of *Beta vulgaris* was poured in a 500 mL beaker with 200 mL deionized water, mixed thoroughly and heated at 90 °C for 30 minutes. The extracted solution's hue changed from maroon to red-purple when it was allowed to cool to ambient temperature. Whatman filter paper No. 1 was used to filter the mixture. The resultant solution was centrifuged for 15 minutes at 4000 rpm in a 1.5 mL tube. *Beta vulgaris* extract stock solution was obtained. (7.06 g) of $\text{Bi}_2(\text{SO}_4)_3$ or $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ was dissolved in 30 mL deionized water, then 100 mL *Beta-vulgaris* extract was added as dispersant, with stirring and heating at a temperature 90 Celsius, then a solution of (1 M) NaOH was gradually added to it drop by drop by dissolving 2 g of NaOH in 500 mL of deionized

distilled water until the pH value turned 12. The mixture was left for 48 hours in order to precipitate, forming a pale green precipitate. Next, separating was done by using a centrifuge, washing the precipitate with hot distilled water and hot EtOH, and getting it dried at a temperature of 300 Celsius by using an electric oven for 10 hours, leaving a pale brown powder of D₁ and yellow powder of D₂.

Results and Discussion

The two samples (D₁ and D₂) were analyzed using FT-IR spectrum. Figure 1 shows absorption bands

at 600 cm⁻¹, both of which were due to the stretching mode of Bi-O while the peak of stretching vibration of O-H appeared at 3429 cm⁻¹. Peaks at 2900 cm⁻¹, 2800 cm⁻¹ and 1113 cm⁻¹, 1046 cm⁻¹ are attributed to vibrations of CH₂ aliphatic and C-O bond respectively of beta vulgaris precursor [7, 15].

Spectra for both D₁, D₂ confirmed the presence of Bi₂O₃NPs. As shown in Figure 2 at 326 nm, an absorption peak was seen, attributed to Bi₂O₃NPs. The energy gap was calculated by using equation $E_g = 1239.83 / \lambda$. E_g is the bulk band expressed in eV. λ = lambda is peak absorption, $E_g = 1239.83 / 326 = 3.80$ eV [7].

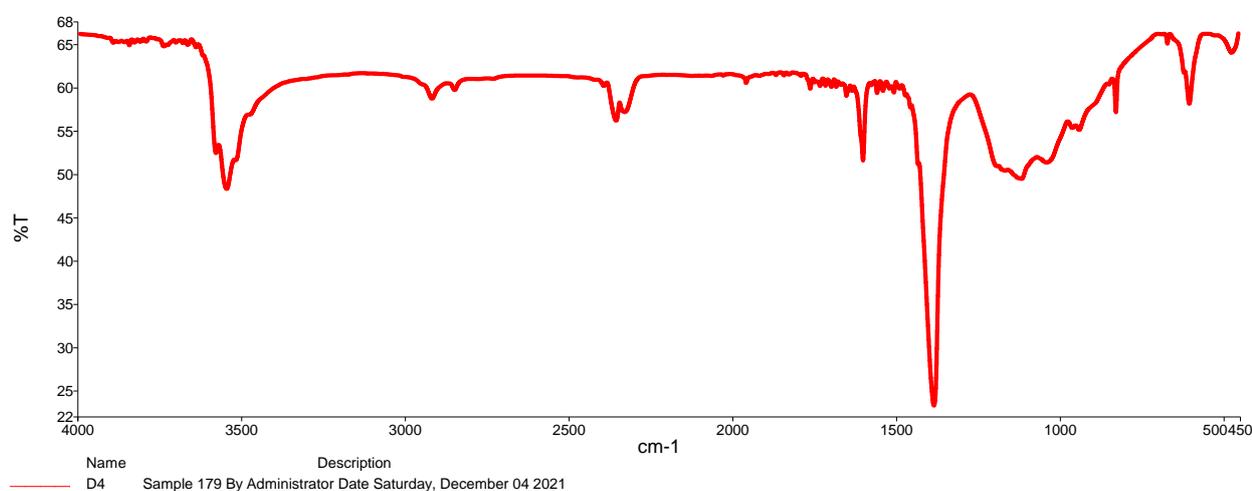


Figure 1: FT – IR spectrum of Bi₂O₃NP for (D₁, D₂ samples)

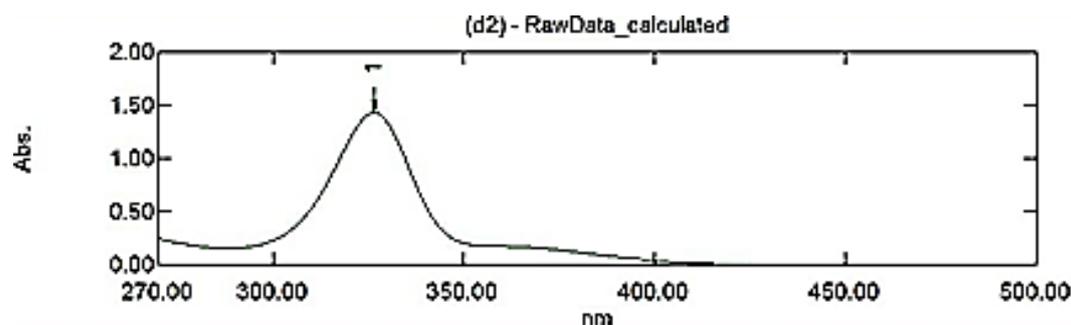


Figure 2: UV-Vis spectrum of Bi₂O₃ Nps

Pattern of Bi₂O₃ Np was synthesized from Bi₂(SO₄)₃ (D₁) of beta vulgaris. Figure 3 gives peaks indicating that the Bi₂O₃ Np are amorphous. The peaks are associated with the monoclinic

crystal phase of Bi₂O₃ (COD: No-96-152-6459) at the 2θ value of 28.56, 34.35, 42.52, 46.50 and 58.58 [13].

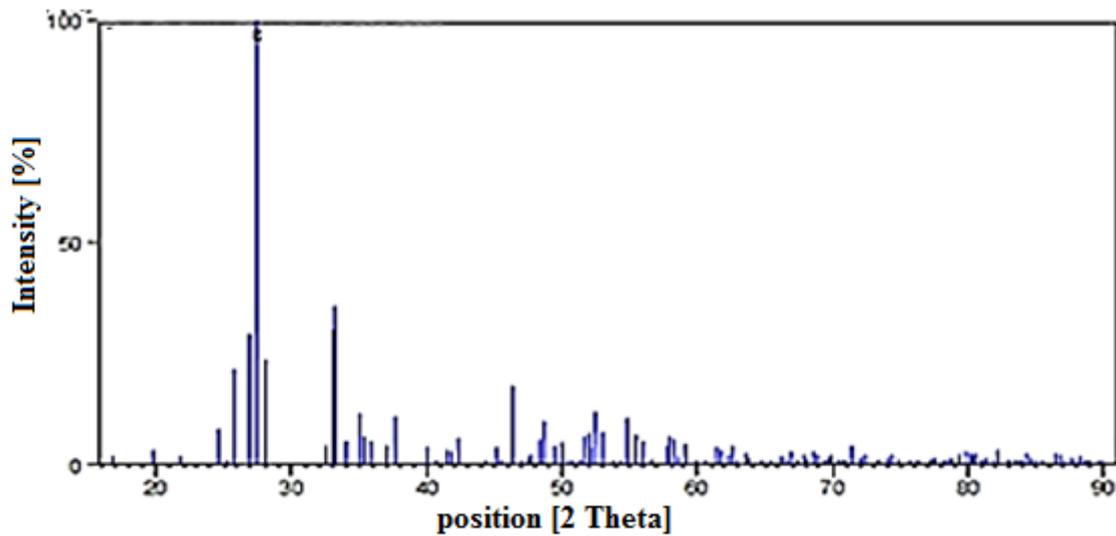


Figure 3: XRD of Bi₂O₃ Nps for (D₁ sample)

The Bi₂O₃ NPs was synthesized from Bi(NO₃)₃.5H₂O (D₂) of beta vulgaris. Figure 4 showed rhombohedral phase of Bi₂O₃ suitable with JCPDS file - 27- 50 (Bi₂O₃) at the 2θ value of 27.66, 31.39, 32.501, 45.89, 55.34, and 74.45. The Debye Scherres equation was used to compute the average crystallite size of both samples, and the average crystal size of D₁ and D₂ was found to be 30.284 nm and 34.896 nm, respectively [16]. The morphology studies of both Bi₂O₃ samples (D₁ and D₂) are shown in Figure 5. The image shows

that spherical particles tend to aggregate with average size of the particles by 30.28 nm and 34.89 nm [17].

The elemental and compositional properties of both sample of Bi₂O₃NPs were studied by EDX. The EDX spectra of Bi₂O₃NPs were synthesized by beta vulgaris extracts. Figure 6 depicts the discovery of strong bismuth (Bi) peaks at 2.5 and a medium peak of oxygen (O) at 0.5. [13].

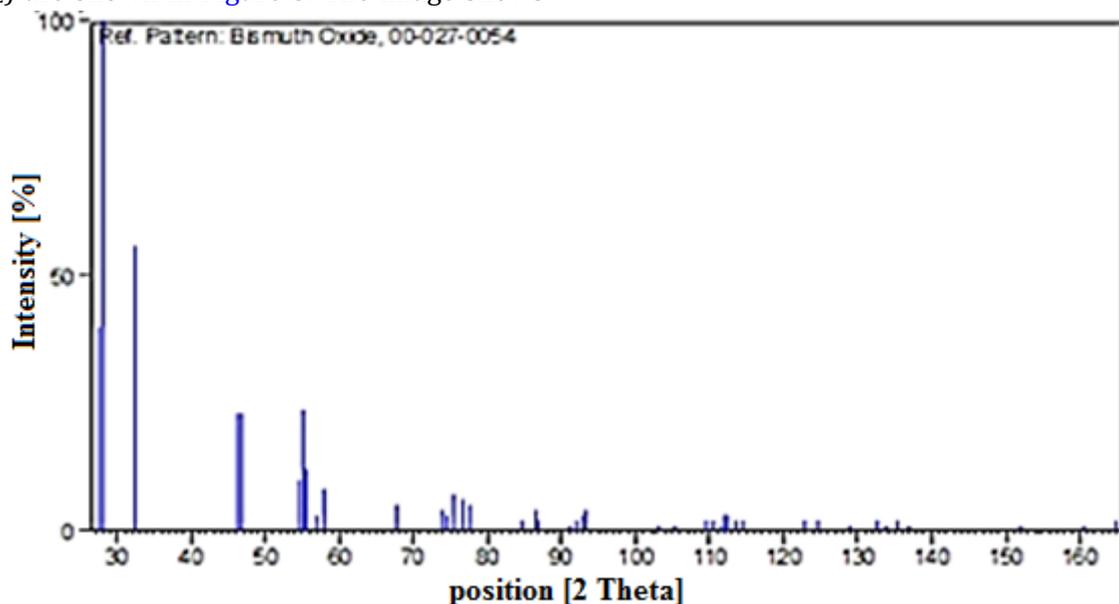


Figure 4: XRD of Bi₂O₃ Nps for (D₂ sample)

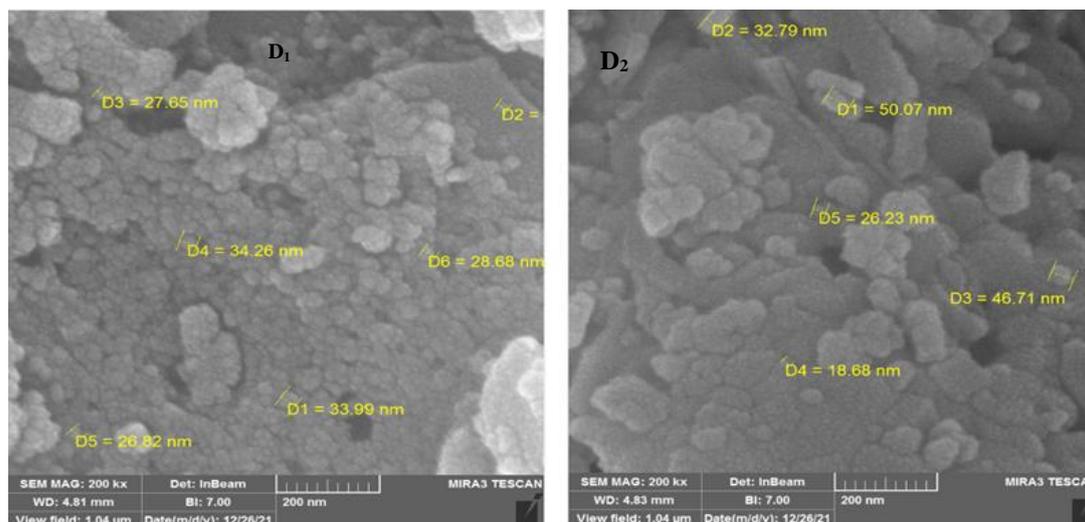


Figure 5: Image of SEM Characterized for D₁ and D₂

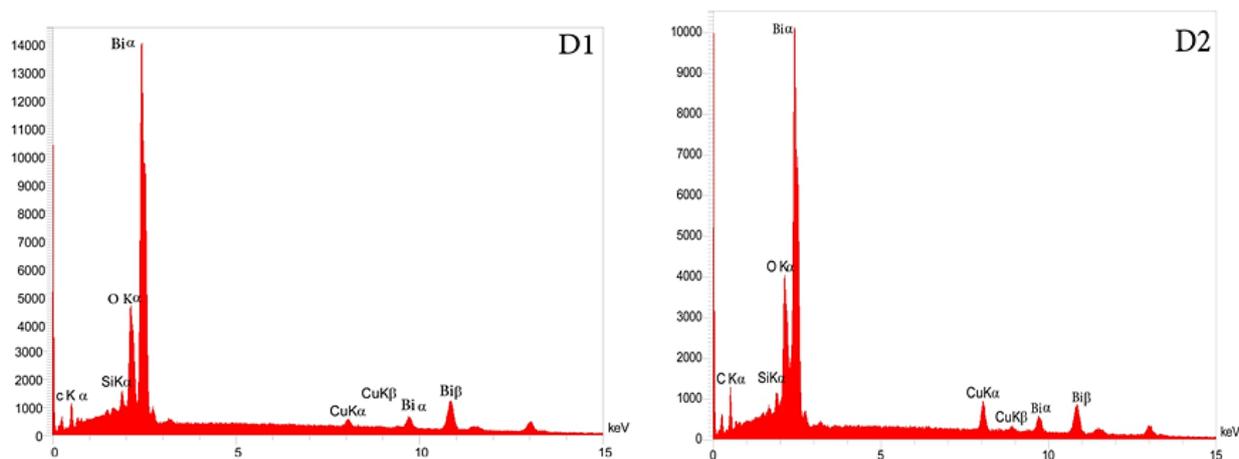


Figure 6: EDX of Bi₂O₃ Nps for (D₁, D₂)

The morphology of Bi₂O₃Nps was studied using a transmission electron microscope (Figure 7). TEM image reveals an aggregation of thin sheet clusters [18].

Adsorption activity of Bi₂O₃NPs

The use of nanomaterials to remove various metal ions present in water simultaneously opens up a new path that is free of secondary contamination and is very affordable. Bi₂O₃ NPs has a large specific surface area, surface area per unit mass, porous material and the structure and surface shape of Bi₂O₃ NPs, as seen in Figures 6 and 7 (SEM TEM), making it a strong adsorption candidate. At room temperature and pH = 6, three metal ions, namely Co²⁺, Ni²⁺, and Cu²⁺, were effectively removed from water concurrently using Bi₂O₃NPs.

The removal of these metal ions from water is shown in this research to have a quick adsorption of Co²⁺, Ni²⁺ and Cu²⁺, with more than 90% adsorption in less than a minute for Ni²⁺. As seen in Figure 8, the rate of metal ions uptake steadily declined and did not appear to grow any more throughout this time. These findings may be explained by the fact that there were more active sites available on the Bi₂O₃ NPs, adsorbent, at the start of the adsorption process, and as time passed, these sites gradually became saturated [19, 20]. For the D₁ sample, the removal efficiencies of Co(II), Ni(II) and Cu(II) ions were 89.68%, 85.56%, and 94.56%, respectively and for sample D₂, the removal efficiencies of Co(II), Ni(II) and Cu(II) ions were 93.3%, 87.7 % and 88.54 %, respectively.

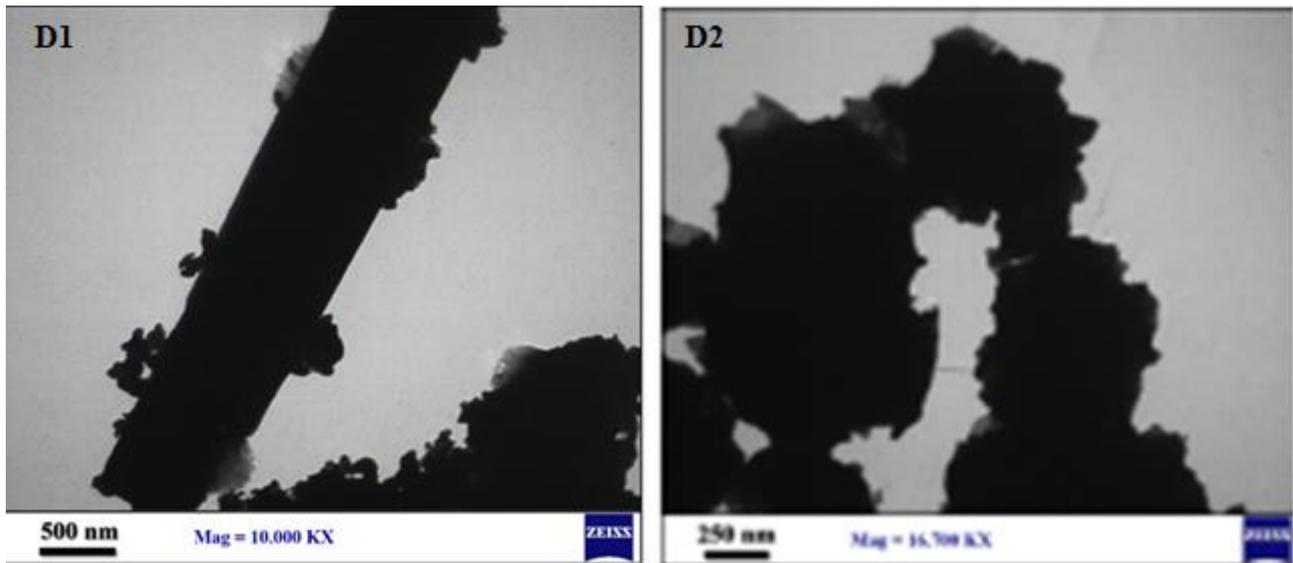


Figure 7: Image of TEM characterized for D₁ and D₂

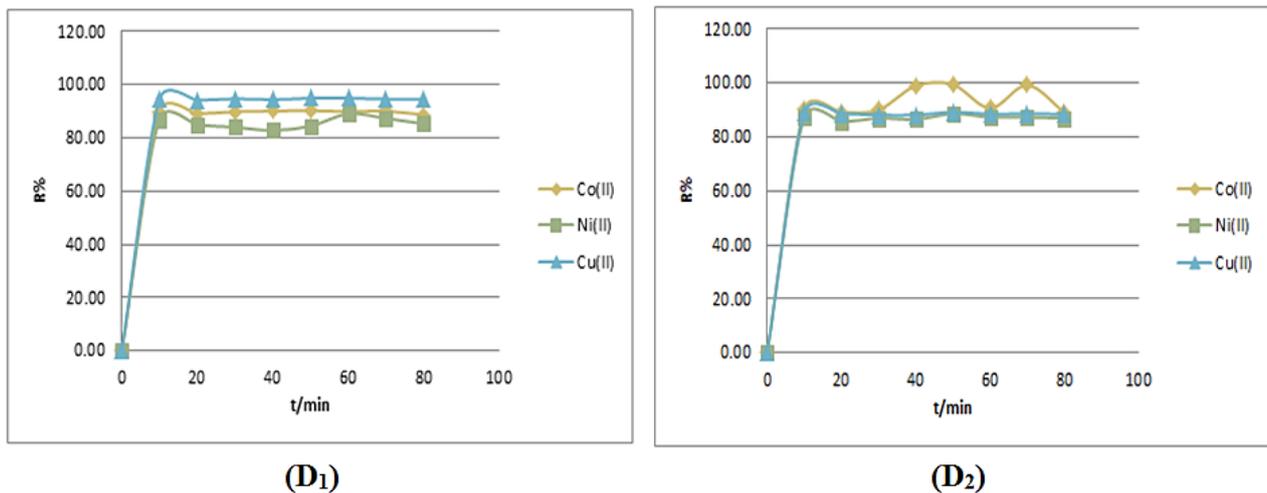


Figure 8: The removal efficiencies of Co(II), Ni(II), and Cu(II) ions for D₁ and D₂ sample

Antibacterial activity of Bi₂O₃ NPs

Bismuth is found as [Bi₂O₃, Bi₂(CO₃)₃, Bi₂S₃] in medicine. Bismuth has been employed as an antidiarrheal to treat vomiting, stomach, and pain nausea. Bi₂O₃NPs porous Nano spheres demonstrated inactivation of (G⁻) and (G⁺) bacteria [21, 22]. To perform a qualitative antimicrobial screening, agar well diffusion and minimum inhibitory concentration experiments are carried out. The zones of inhibition at diluted

concentration of HCl (0.02g / mL) for D₁ are obtained as *E. Coli* and *S. aureus* is (31 and 29 mm) for D₁ is (30 and 30 mm), *candida albicans* (43 and 45 mm) of D₁ and D₂ for Bi₂O₃NPs preparation. After this comprehensive analysis, we concluded that both *Staphylococcus aureus* and *E. Coli* have the highest inhibitory values with sample D₁ and D₂ and also a very high inhibition with *Candida albicans fungal* for the same samples as shown in Figure 9 [23, 24].

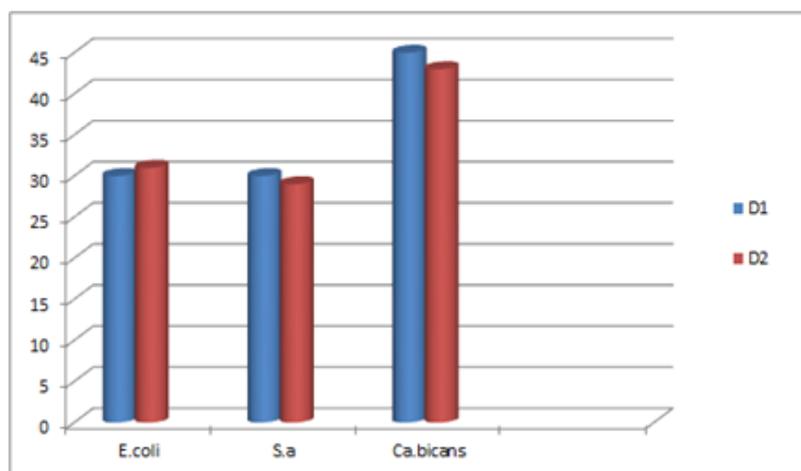


Figure 9: Types of bacteria and fungi tested and inhibition diameters in units (mm) of prepared Bi₂O₃NPs for D₁ and D₂ samples

Conclusion

Bi₂O₃NPs were successfully synthesized by a simple cost-effective biosynthesis method of Bi₂O₃ monoclinic phase with diameter of 30.284 nm produced by using Bi₂(SO₄)₃. Bi₂O₃ rhombohedral phase with diameter (34.896) nm was obtained by using Bi(NO₃)₃.5H₂O. The morphology of Bi₂O₃NPs was as aggregation of thin sheet cluster. They have antimicrobial activity inhibiting the growth of *S. aureus*, *E. Coli* and a very high inhibition with *Candida albicans* fungal. Bi₂O₃NPs were successfully removed simultaneously with three metal ions (M⁺² = Co, Ni and Cu) from water contaminated with them.

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Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose.

References

- [1]. Kadium D., Mahmoud Z., Mousa F., Green Synthesis of Iron Oxide Nanoparticles and testing their Inhibitory against some pathogens, *Asian Journal of Water, Environment and Pollution*, 2021, **18**:119 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [2]. Mohammed M.J., Mahdi W.K., Musa F.H., Green Synthesis, Characterization, and Biological Activity of Zinc Oxide Nanoparticles using Aqueous Extract of Beta Vulgaris and the Seed of Abrus precatorius, *International journal of Drug Delivery Technology*, 2021, **11**:1 [[Google Scholar](#)], [[Publisher](#)]
- [3]. Mahiuddin M., Ochiai B., Green synthesis of crystalline bismuth nanoparticles using lemon juice, *Royal Society of Chemistry Advance*, 2021, **11**:26683 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [4]. Fato F.P., Li D.W., Zhao L.J., Qiu K., Long Y.T., Simultaneous removal of multiple heavy metal ions from river water using ultrafine mesoporous magnetite nanoparticles, *ACS omega*, 2019, **4**:7543 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [5]. Fu F., Wang Q., Removal of heavy metal ions from wastewaters: A review, *Journal of Environmental Management*, 2011, **92**:407 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [6]. Dave P.N., Chopda L.V., Review Article Application of Iron Oxide Nanomaterials for the Removal of Heavy Metals, *Journal of Nanotechnology*, 2014, **2014**:398569 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [7]. Motakef-Kazemi N., Yaqoubi M., Green Synthesis and Characterization of Bismuth Oxide Nanoparticle Using Mentha Pulegium Extract, *Iranian Journal of Pharmaceutical Research*, 2020, **19**:70 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]

- [8]. Song J.Y., Kim B.S., Biological synthesis of bimetallic Au/Ag nanoparticles using Persimmon (Diopyros kaki) leaf extract, *Korean Journal of Chemical Engineering*, 2008, **25**:808 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [9]. Mondal S., Roy N., Laskar R.A., Sk I., Basu S., Mandal D., Begum N.A., Biogenic synthesis of Ag, Au and bimetallic Au/Ag alloy nanoparticles using aqueous extract of mahogany (Swietenia mahogani JACQ.) leaves, *Colloids and surfaces B: biointerfaces*, 2011, **82**:497 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [10]. Dobrucka R., Synthesis of titanium dioxide nanoparticles using Echinacea purpurea herba, *Iranian journal of pharmaceutical research: IJPR*, 2017, **16**:753 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [11]. Abd Kelkawi A.H., Kajani A.A., Bordbar A.K., Green synthesis of silver nanoparticles using Mentha pulegium and investigation of their antibacterial, antifungal and anticancer activity, *IET Nanobiotechnology*, 2017, **11**:370 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [12]. Jafari A., Pourakbar L., Farhadi K., Biological synthesis of silver nanoparticles and evaluation of antibacterial and antifungal properties of silver and copper nanoparticles, *Turkish Journal of Biology*, 2015, **39**:556 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [13]. Nurmallasari N., Yulizar Y., Apriandanu D.O.B., Bi₂O₃ nanoparticles: synthesis, characterizations, and photocatalytic activity, *Materials Science and Engineering*, 2020, **763**:012036 . [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [14]. Uddin I., Adyanthaya S., Syed A., Selvaraj K., Ahmad A., Poddar P., Structure and Microbial Synthesis of Sub-10 nm Bi₂O₃ Nanocrystals ., *Journal of Nanoscience and Nanotechnology*, **8**:3909 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [15]. Mehring M., From molecules to bismuth oxide-based materials: Potential homo- and heterometallic precursors and model compounds, *Coordination Chemistry Reviews*, 2007, **251**:974 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [16]. Eberl J., Kisch H., Mineralization of Phenol and 4-Chlorophenol Induced by Visible Light and Assisted by Semiconducting β-Bi₂O₃, *Zeitschrift für Naturforschung B*, 2010, **65b**:399 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [17]. Li H., Zhang Y., Liu H., Wang J., Large ZnO Mesocrystals of Hexagonal Columnar Morphology Derived from Liquid Crystal Templates, *Journal of the American Ceramic Society*, 2011, **94**:3267 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [18]. Reddy B.C., Manjunatha H.C., Vidya Y.S., Sridhar K.N., Pasha U.M., Seenappa L., Sadashivamurthy B., Dhananjaya N., Sathish K.V., Gupta P.D., X-ray / gamma ray radiation shielding properties of α - Bi₂O₃ synthesized by low temperature solution combustion method, *Nuclear Engineering and Technology*, 2021, **54**:1062 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [19]. Fonseca M.B., An L., Cox B.T., Sulfate as chromophores for multiwavelength photoacoustic imaging phantoms , *Journal of Biomedical optics*, 2017, **22**:125007 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [20]. Prochaska C., Gallios G., Nano-Adsorbents for Cobalt Removal from Wastewater: A Bibliometric Analysis of Research Articles Indexed in the Scopus Database, *Processes*, 2021, **9**:1177 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [21]. Ding L.L., Zhao Q., Zhu J.L., Fan Z.J., Liu B., The preparation and property Research of Bismuth Oxide Nanospheres, *International Conference on Materials Chemistry and Environmental protection*, 2016, 17 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [22]. Iftikhar S., Iqtedar M., Saeed H., Aftab M., Abdullah R., Kaleem A., & Aslam F., Comparative and combinatorial study of biogenic bismuth nanoparticles with silver nanoparticles and doxycycline against multidrug resistant Staphylococcus aureus BTCB02 and Salmonella typhi BTCB06 ., *Revista Mexicana de Ingeniería Química*, 2021, **20**:271 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [23]. Hernandez-Delgadillo R., Velasco-Arias D., Martinez-Sanmiguel J.J., Diaz D., Zumeta-Dube I., Arevalo-Niño K., Cabral-Romero C., Bismuth oxide aqueous colloidal nanoparticles inhibit Candida albicans growth and biofilm formation, *International Journal of Nanomedicine*, 2013, **8**:1645 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [24]. Prince E.D ., Amin F.M., Abu-Yousef I.A., Srinivasan N ., Paliro P., Use of A Hydroalcoholic

Extract of Moringa oleifera Leaves for the Green Antioxidant Activities, *Materials*, 2020, **13**:876
Synthesis of Bismuth Nanoparticles and [Crossref], [Google Scholar], [Publisher]
Evaluation of Their Anti-Microbial and

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