

# FE<sub>2</sub>O<sub>3</sub>-DOPED CDO NANOPARTICLE SYNTHESIS AND CHARACTERIZATION FOR ANTIBACTERIAL ACTIVITY

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Article history:		Abstract:
<b>Received:</b>	20 <sup>th</sup> August 2025	Fe(NO <sub>3</sub> ) <sub>2</sub> and Cd(NO <sub>3</sub> ) <sub>2</sub> were used as precursors in the eco-friendly method to prepare the iron-cadmium oxide (Fe <sub>2</sub> O <sub>3</sub> @ CdO) nanoparticles using apple leaves extract. First, combine the two salts in a weight ratio of 1:1. Next, dissolve 1.4 g of the salt mixture in 100 mL of deionized water. The calcination process for the Fe(OH) <sub>2</sub> -Cd(OH) <sub>2</sub> nanoparticles at 550 °C produced Fe <sub>2</sub> O <sub>3</sub> @ CdO nanoparticles. XRD, FE-SEM, and TEM methods were used to characterize the nanoparticles. Fe <sub>2</sub> O <sub>3</sub> @ CdO nanoparticles with an average size of 41.73 nm are confirmed to have formed by XRD. While TEM produced heterogonous morphologies, FE-SEM demonstrated that the Fe <sub>2</sub> O <sub>3</sub> @ CdO nanoparticles will produce spherical morphology nanoparticles and significant agglomeration. Gram negative (Escherichia coli and Aeromonas hydrophila) and gram positive (Rhodococcus rhodochrous and Staphylococcus aureus) bacterial strains are evaluated against the produced Fe <sub>2</sub> O <sub>3</sub> @ CdO nanoparticles. The antibacterial activity of the α-Fe <sub>2</sub> O <sub>3</sub> @ CdO nanoparticles is seen to be good (zone of inhibition: E. coli-14 mm).
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## INTRODUCTION

A physical or chemical substance that has at least one dimension between 1 and 100 nm is called a nanomaterial. It has drawn a lot of interest because of its special optical, electrical, and mechanical qualities that set it apart from its bulk equivalents [1]. The emergence of microbes that are resistant to several drugs poses a serious threat to global health [2]. As a result of pathogenic bacteria's increasing resistance to conventional antimicrobial medicines, therapies are failing and rates of morbidity and mortality are rising [3]. Novel therapeutic platforms are desperately needed to address this expanding clinical dilemma since the widespread abuse and overuse of antibiotics is a major contributing factor that encourages the selection and dissemination of resistance bacteria [4]. The development of novel materials with antibacterial capabilities has been made possible by nanotechnology in recent decades. Nanoparticles have distinct size-dependent physicochemical features that set them apart from their bulk material counterparts [5]. Due to their advantageous chemical and physical characteristics, effectiveness against resistant strains, and reduced toxicity concerns, metal and metal oxide nanoparticles are attracting interest as possible antibacterial materials [6]. Due to its many applications in biomedicine, sensing, catalysis, and other domains, iron oxide nanoparticles (Fe<sub>2</sub>O<sub>3</sub>-NPs), which are semiconducting MONPs, are attracting a lot of attention [7]. Additionally, the cadmium oxide (CdO-Nps) reduced the amount of medication that was administered and improved the oral bioavailability and solubility of the chemotherapeutic medicines [8]. Physical, chemical, and environmentally friendly methods can be used to synthesis Fe<sub>2</sub>O<sub>3</sub> - NPs, which exhibit excellent chemical and thermal stability, exceptional catalytic activity, and nanoscale biocompatibility [9]. CdO-NPs high surface area to volume ratio makes it possible for them to effectively touch bacteria and fungus, preventing their development and survival [10]. With a wealth of information on their biological in vitro and in vivo activity available in recent literature, CdO-Nps is one of the most intriguing alternatives for biomedicine [11]. Different plants have been used to create CdO NPs, which has an impact on the final nanomaterials' shape [12]. Because of their innate broad-spectrum antibacterial properties, CdO-NPs can be used in medical settings [13]. However, in drug-resistant strains and biofilms, CdO-NPs' antibacterial activity might not be enough on their own. Moreover, the large quantities needed to achieve adequate biocidal effects raise questions about the cytotoxicity of mammalian cells. Therefore, regulating NPs' functional efficiency continues to be a significant research problem [14]. Doping with secondary metal ions, which advantageously alter structural, optical, electrical, and antibacterial properties, is one efficient tactic. Improved optical, catalytic, and antibacterial properties have been demonstrated by the addition of metals such as manganese, nickel, cobalt, lanthanum, and zinc [15]. When CdO-NPs are integrated with silver, a number of studies have demonstrated a synergistic effect on their biological activity [16]. Both the intracellular generation of ROS and the direct bactericidal actions of Fe<sup>+2</sup> ions are responsible for the iron oxide nanoparticles' (Fe<sub>2</sub>O<sub>3</sub>-NPs) well-established broad-spectrum antibacterial activity [17]. In this study, iron oxide and cadmium oxide nanoparticles were combined to enhance their catalytic and antibacterial properties.

**Experimental part****Materials**

Chemicals of analytical purity (CDH) Iron (II) nitrate  $\text{Fe}(\text{NO}_3)_2$  (98%), (Alpha Chemika) cadmium nitrate  $\text{Cd}(\text{NO}_3)_2$  (99%), deionized water and apple leaves were used to synthesize the hematite  $\alpha\text{-Fe}_2\text{O}_3$  and CdO nanoparticles.

**Instruments and Apparatus**

Every sample analysis was carried out at the University of Tehran, Iran's College of Science. The Siemens model D500 is utilized to irradiate the samples in order to obtain the XRD pattern using the XPERT-Pro diffractometer, which operates at 30 mA and 40 kV for scanning angles ranging from 20 to 80°. The transmission electron microscope (TEM) type Philips model: CM120 and the field emission scanning electron microscope (FE-SEM) ZEISS model: Sigma VP are utilized for the surface investigations.

**Synthesis of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles using apple leaves extract**

Apple leaves from trees in the Diyala governorate were used to make the plant extract. They were thoroughly cleaned with ordinary water and then with distilled water. After two days of shade drying, the leaves were chopped and thoroughly ground, and 10 g were added to 200 mL of distilled water while being stirred. To remove any leftover live elements, the extract is filtered, placed in test tubes, and centrifuged for 10 minutes at 2000 rpm. It is then collected in a vial and stored at 4°C. A magnetic stirrer was used to thoroughly mix 50 milliliters of deionized water with 0.5 grams of mix salts ( $\text{Fe}(\text{NO}_3)_2$  and  $\text{Cd}(\text{NO}_3)_2$ ). In a burette, we take 10 milliliters of the apple leaf extracts and add them drop by drop to the mixture at 25 degrees Celsius. We also add sodium hydroxide solution (0.1M) gradually until the solution turns basic and a precipitate forms. Pollutants and silt are washed away using ethanol and water. After a 24-hour drying process, a mixture of nano iron and cadmium hydroxide powder is created. The resulting powder is then burned for five hours at 550 °C to produce  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles.

**Result and dissection****XRD analysis**

The XRD patterns of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles (calcination at 550°C) are shown in Figure 1. The existence of sharp and distinct peaks corresponding to the (012), (104), (110), (113), (024), and (116) corresponding to  $\text{Fe}_2\text{O}_3$  according to the [18] makes it evident from the examined data that  $\text{Fe}_2\text{O}_3$  nanoparticles (black star) and CdO nanoparticles (red circle) formed as a nanoparticles with average diameter 43.67 nm. The significant intensity of the peak corresponding to (110) indicates that the growth along the (110) plane is dominating. The formation of the  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles is further confirmed by the XRD results of the CdO NPs (red circle), which show a number of additional peaks to hematite that are indexed to CdO [18]. These peaks correspond to the (111), (200), (220), and (311) planes. Furthermore, no other peaks were seen, which amply demonstrates that there are no additional potential impurity phases present.

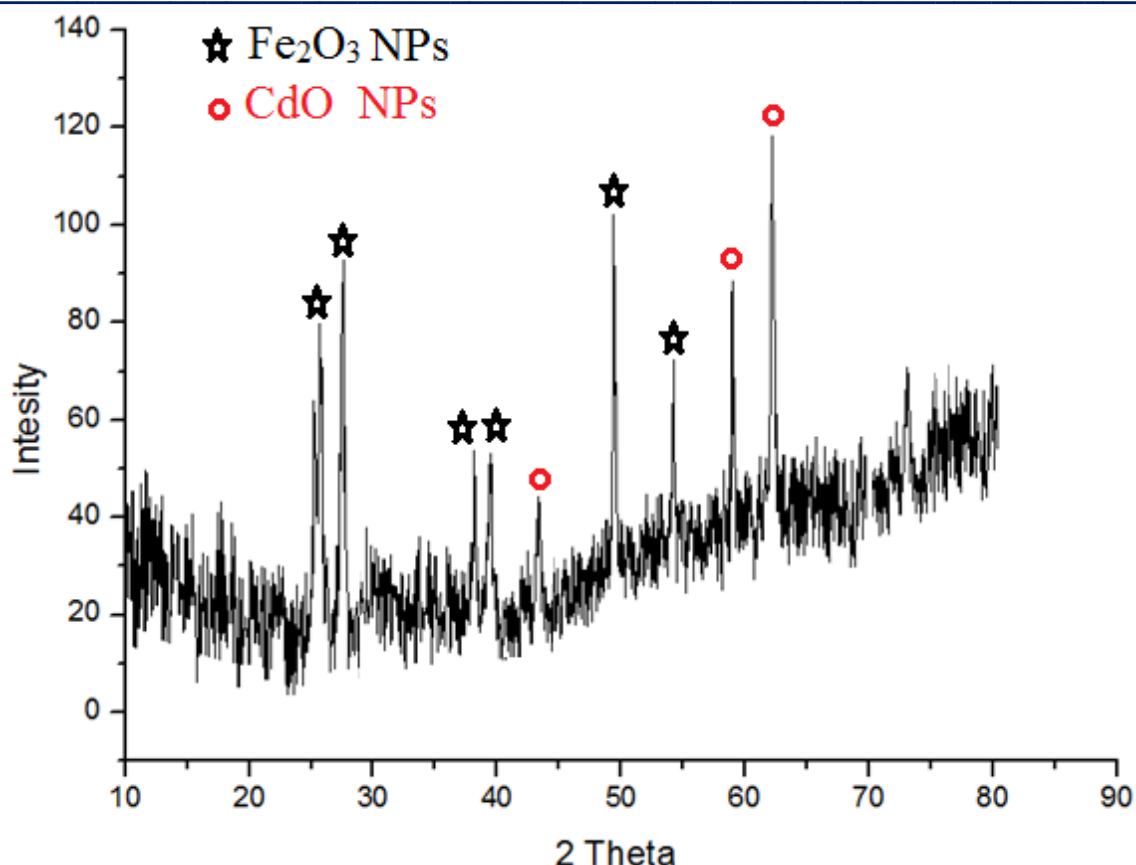


Fig 1. XRD pattern of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles

### FE-SEM analysis

A basic analytical method that is frequently used in many scientific fields, especially materials science research, is FE-SEM. FE-SEM is a high-resolution imaging technique that creates detailed images that show the topography of a subject by scanning its surface with an electron beam. This method makes it possible to examine a material's surface at high magnification levels, providing important information on its composition, shape, and structure. The presence of spherical, cubic and oval nanoparticles with smooth surfaces was shown by the FE-SEM images at 100 kx and 200 kx (Fig. 3 A and B). The average size of the produced nanoparticles was 46.43 nm, according to the consistent with XRD results.

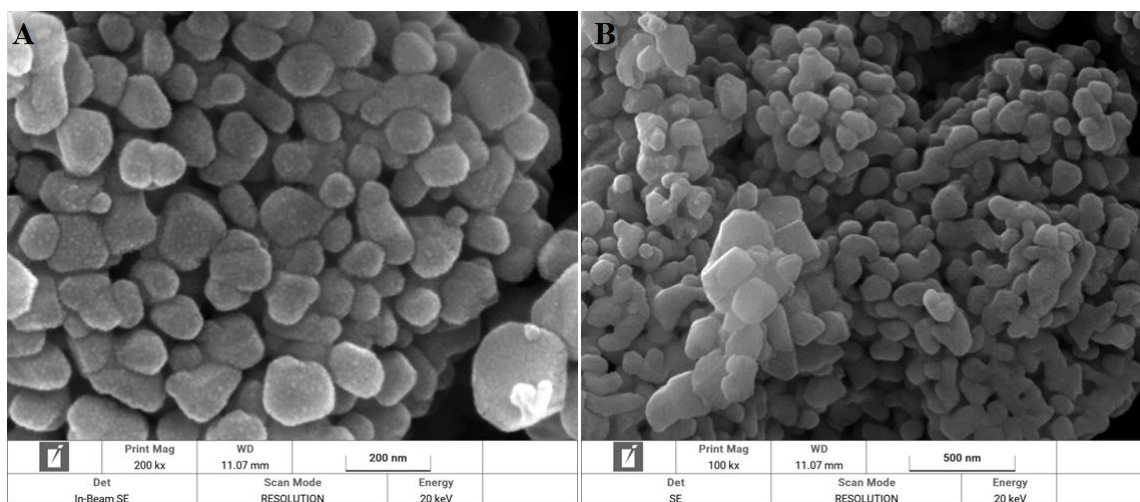


Fig 2. FE-SEM images of (A)  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles at 200 kx (B)  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles at 100 kx.

### TEM analysis

For assessing nanoparticles at the nanoscale, TEM is an effective method. By passing an electron beam through a tiny specimen, this approach allows researchers to acquire high-resolution photographs of the internal structure and shape of nanoparticles. At resolutions of a few angstroms, TEM enables precise, in-depth examination of particle size, shape, and distribution. These in-depth analyses offer important insights into the physical properties of nanoparticles, supporting their thorough characterisation and expanding our understanding of their characteristics for use in materials science and nanotechnology. The produced Fe<sub>2</sub>O<sub>3</sub> @ CdO NPs are shown in the TEM picture in Figure 3 and are distinguished by their spherical shape and uniform size distribution. After image calibration, the pixel measurements from the TEM images were converted into nanometers to investigate the sizes of the nanoparticles in detail. The thorough examination unequivocally confirmed that the produced Fe<sub>2</sub>O<sub>3</sub> @ CdO NPs were nanoscale, with average diameters of 31.42 nm and sizes ranging from 18 to 48 nm.

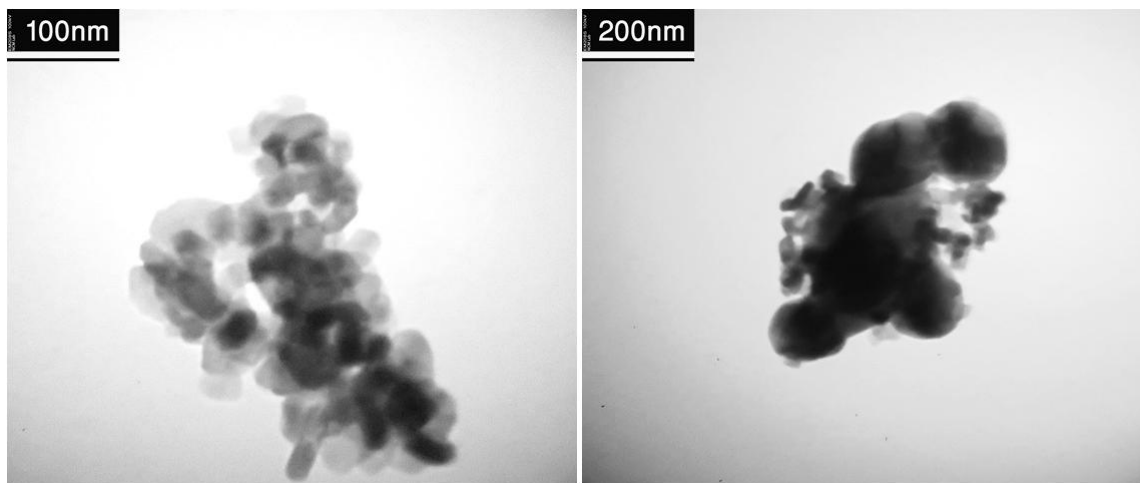


Fig 3. TEM images of Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles at 100 and 200 nm

### Antibacterial activity of Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles

Bacterial resistance to the external environment is largely attributed to their cell walls and membranes. The bacterial cell wall in particular is crucial for preserving the bacterium's organic shape. Gram-positive and Gram-negative bacteria as well as NPs have different adsorption paths thanks to the components of the cell membrane. Furthermore, LPS is a unique component of Gram-negative bacteria's cell walls that provides a negatively charged region that draws NPs [19]. Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles' antibacterial qualities were examined against both Gram-negative (*Escherichia coli* and *Aeromonas hydrophila*) and Gram-positive (*Rhodococcus rhodochrous* and *Staphylococcus aureus*) bacteria (see Figs. 4, 5). The concentration of Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles is 100 µg/mL. Figures 4 and 5 depict the zone of inhibition (zone), which shows how the Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles impact bacterial growth. At this point, the significant effects were evident. Table 1 displays the Fe<sub>2</sub>O<sub>3</sub>@CdO nanoparticles' zone of action against the bacterial strains *Aeromonas hydrophila*, *Escherichia coli*, and *Rhodococcus rhodochrous* and *Staphylococcus aureus*. In actuality, the metal nanoparticles interact with essential elements like the phosphorous (P) and sulfur (S) groups of bacterial DNA to bind to the proteins and DNA of the pathogens. This leads to the destruction of bacterial DNA replication<sup>64</sup>. The generation of free radicals is one potential mechanism for the antibacterial effect. The mix nanoparticles Cd<sup>2+</sup> and Fe<sup>2+</sup> ions pierce the pathogens cell walls through the damaged surface. When ions are liberated from the nanoparticles, reactive oxygen species (ROS) are created. Among the ROS components with significant bactericidal action are hydrogen peroxide, superoxide radicals, hydroxyl radicals, and singlet oxygen [20].

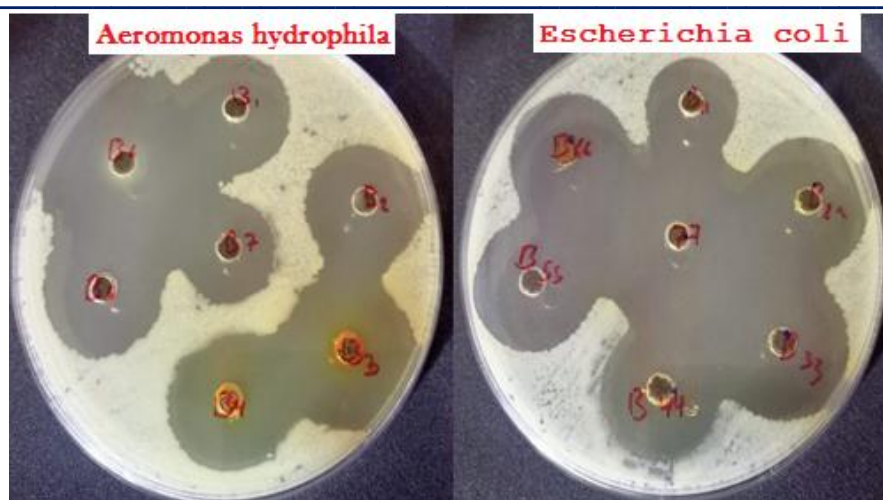


Figure 5. Antibacterial activity of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles against Gram-positive bacteria (*Rhodococcus rhodochrous* and *Staphylococcus aureus*).

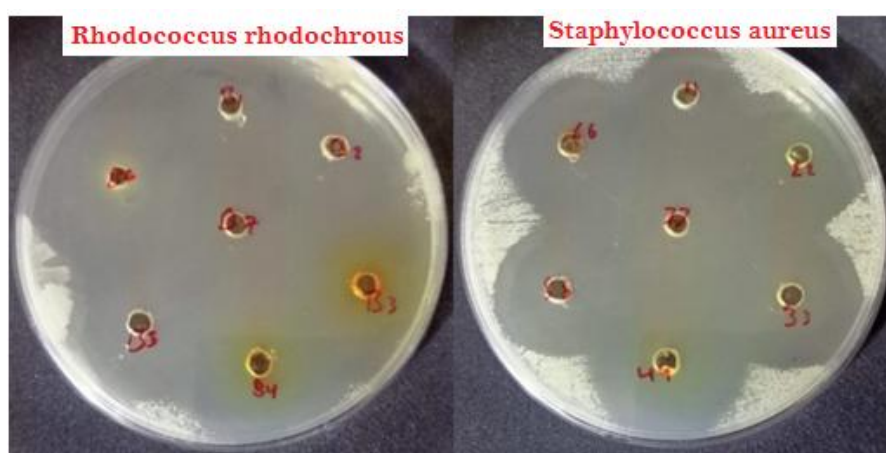


Figure 6. Antibacterial activity of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles against Gram-negative bacteria (*Escherichia coli* and *Aeromonas hydrophila*).

Table 1. The types of bacteria used in the study at 100  $\mu\text{g}/\text{mL}$  concentrations.

Sample	Concentration ( $\mu\text{g} / \text{mL}$ )	Zone of inhibition (in mm)			
		Bacteria			
$\text{Fe}_2\text{O}_3@\text{CdO}$ NPs	100	E. coli	A. hydrophila	R. rhodochrous	S. aureus
		29	39	> 50	34

## CONCLUSION

In summary,  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles were successfully prepared, and their physical characteristics were examined using XRD, FE-SEM, and TEM. Their antibacterial qualities were also investigated. The presence of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles with an average diameter of 43.67 nm is confirmed by XRD studies. While TEM revealed a spherical shape and consistent size distribution, FE-SEM revealed spherical, cubic, and oval morphologies of  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles with an average diameter of 46.43 nm. Based on the findings, the  $\text{Fe}_2\text{O}_3@\text{CdO}$  nanoparticles shown excellent antibacterial activity against a range of Gram-positive and Gram-negative bacteria, making them a promising option for bacterial disinfection.

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