Original Research Article

Synthesis of Manganese Oxide Nanoparticles Using Co-Precipitation Method and Its Antimicrobial Activity

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ABSTRACT

Manganese oxide (MnO) nanoparticles are essential materials in various applications such as sensors, catalysis, molecular adsorption, electronics, and exhibited excellent antimicrobial activity. Due to the numerous demerits of current nanoparticle synthetic approaches, there is a surge in the exploration of efficient methods for the synthesis. This study involved the synthesis of MnO nanoparticles through a simple, cost-effective, and time-efficient co-precipitation method by using manganous sulphate (0.03 M) and sodium hydroxide (0.009 M). The UV-Vis absorption showed sharp absorption at 320 nm confirming the presence of MnO nanoparticles. The FTIR spectral analysis depicted absorption bands at 480 cm^{-1} and 515 cm^{-1} correspond to the characteristic peak of the Mn-O bond and XRD spectra predicted the average size of synthesized nanoparticles to be 11 nm. The elemental composition of the nanoparticles was determined by EDX analysis. The synthesized nanoparticles showed significant antimicrobial activity against microorganisms, *Staphylococcus aureus, Klebsiella pnuemoniae,* and *Candida albicans*.

Keywords: nanoparticles synthesis, co-precipitation, antimicrobial activity

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Introduction

Nanoscience and nanotechnology refer to the science and technology of matter, manipulated at the atomic level that deals with the synthesis, characterization, exploration, and exploitation of nanostructured materials for human benefits. The field of nanotechnology is one of the most active areas of research in modern material science [1]. Nanoparticles are particles having one of the dimensions in the nanoscale range, 1-100 nm size, and have received more attention due to their fundamental scientific significance and intriguing electrical, magnetic, and catalytic properties [2]. They possess a high surface-to-volume ratio resulting in both chemical and physical differences in properties [3, 4]. Metal oxide nanomaterials have attracted a large number of scientists over the years and have grown into a distinct stream of research as they are being used in various fields like the fabrication of magnetic data storage devices [5], fuel cells [6], sensors [7], and catalytic reactors [8]. Among the metal oxide nanoparticles, manganese oxide nanoparticles are imperatively utilized in various applications such as catalysis, electrodes, sensors, molecular adsorption, and electronics [9, 10]. Moreover, MnO nanoparticles exhibited excellent antimicrobial activity against various Gram-positive and Gram-negative pathogens like *Staphylococcus aureus,* and *Klebsiella pnuemoniae* including the fungus *Candida albicans* [3]. They are considered one of the best catalysts due to their low cost, low toxicity, and environmental compatibility. These nanoparticles were also used to improve the mechanical and thermal properties of polymers and to enhance the ionic conductivity of polymer electrolytes [11].

Manganese oxide nanoparticles with various shapes and exceptional qualities have been synthesized via several innovative and novel methods, including the hydrothermal method [12], chemical method [13], sol-gel synthesis method [14], ultrasonic bath method [15], thermal decomposition method [2, 16], laser ablation method [17] and green synthesis method [18]. A wet chemical technique, specifically the co-precipitation method is commercially used because of its cost-effective nature. The co-precipitation method offers various advantages in terms of simplicity, rapidness in preparation, convenience to control particle size, and numerous possibilities for modifying the particle surface state and overall homogeneity [19, 20].

This present research study deals with the synthesis of manganese oxide nanoparticles through the co-precipitation method and its characterization along with its antimicrobial activities.

Experimental

Synthesis of manganese oxide nanoparticles

100 mL 0.03 M solution of manganous sulphate ($MnSO_4.H_2O$) [S.D. Fine. CHEM LTD, 98.0 %] was taken in a clean and dry conical flask where 50 mL of 0.009 M sodium hydroxide (NaOH) [HiMedia, 99.99 %] was added drop by drop through burette with constant stirring with the help of magnetic stirrer at a constant temperature of 60 °C. The whole solution mixture was stirred for 2 hours to precipitate the nanoparticles, confirmed by a change of solution color to brown. The solution was centrifuged and the precipitate was collected in the beaker. The precipitate was then dried at 100 °C and kept in a muffle furnace at 500 °C for 2 hours to obtain the desired nanoparticles [3].

Characterization of manganese oxide nanoparticles

The optical properties of the synthesized nanoparticles were analyzed using a double-beam UV-visible spectrophotometer (Model LT- 2802). The Fourier Transform Infrared Spectroscopy (FTIR, Tracer 100) was used to examine the characteristics of functional groups present in the synthesized nanoparticles and the crystal structure of the nanoparticles was determined by Powder X-ray diffraction (XRD) using CuKα (λ= 1.54 Å) radiation and Bragg angle (2θ) in the range of 5° to 90˚. Energy Dispersive X-ray Spectroscopy (EDX-8000) was used to examine the elemental composition and product purity of manganese oxide nanoparticles.

Antimicrobial Assay

The antimicrobial activity of the synthesized nanoparticles was evaluated by the disc diffusion method [21] against one Gram-positive bacteria (*Staphylococcus aureus*), one Gram-negative bacteria (*Klebsiella pnuemoniae*), and one fungus (*Candida albicans*). 150 μL of each fresh liquid microbial seed were spread on the respective Mueller-Hinton Agar (MHA) plates. The wells were made on the surface of the agar and manganese oxide nanoparticles and standard kanamycin 5 mg/mL (10 μL) were loaded. The media plates were incubated for 24 hours at 37 °C and the zone of inhibition was measured.

Results and Discussion

UV-Visible spectroscopic analysis

Optical absorption spectra of synthesized manganese oxide nanoparticles against distilled water as a reference by UV-visible spectrophotometer in the range of 200 nm to 800 nm are shown in Figure 1. An absorption peak at 320 nm indicates the presence of manganese oxide nanoparticles [22, 23].
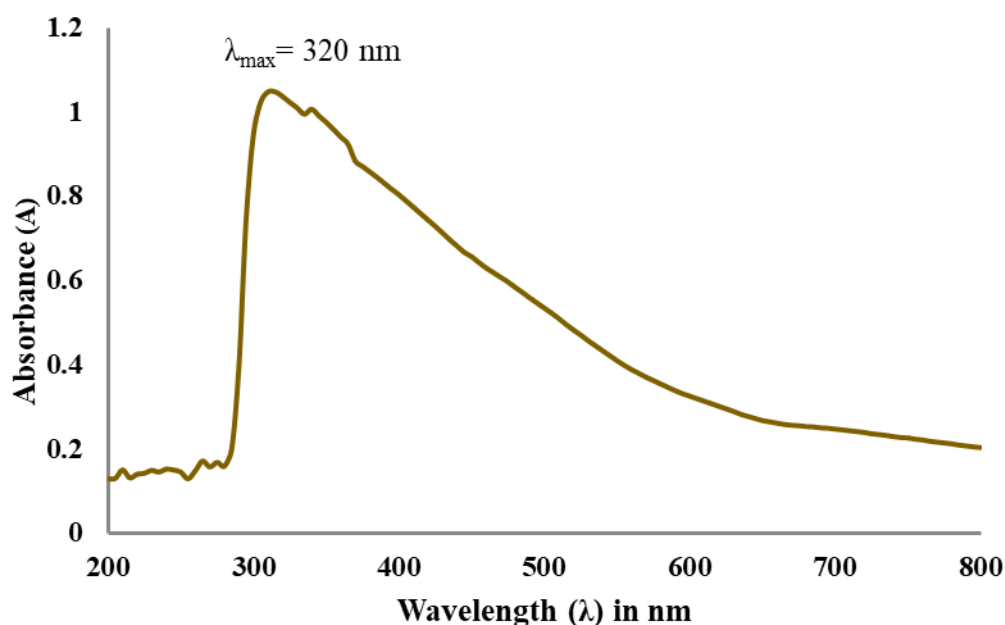

$\lambda_{max} = 320$ nm

Figure 1: UV-VIS spectrum of synthesized manganese oxide nanoparticles

FTIR analysis

FTIR spectroscopy was used to determine the composition and level of purity of manganese oxide nanoparticles synthesized by the co-precipitation method. The FTIR spectra of the functional group present in the synthesized nanoparticles are shown in Figure 2. The distinctive absorption peaks were observed at 3260 cm^{-1}, 2331 cm^{-1}, 2109 cm^{-1}, 1635 cm^{-1}, 515 cm^{-1} and 480 cm^{-1}. The peak at 3260 cm^{-1} may be due to O-H stretching and the peak at 2331 cm^{-1} may be due to O=C=O stretching. The peak at 2109 cm^{-1} is ascribed to the stretching vibration of N=C=S whereas the peak at 1635 cm^{-1} is attributed to the stretching vibration of the C=C bond. The

absorption bands at 480 cm^{-1} and 515 cm^{-1} correspond to the Mn-O bond, which confirms the formation of MnO nanoparticles [20, 24]. This analysis confirms that there are very low or no impurities present in the as-synthesized sample.
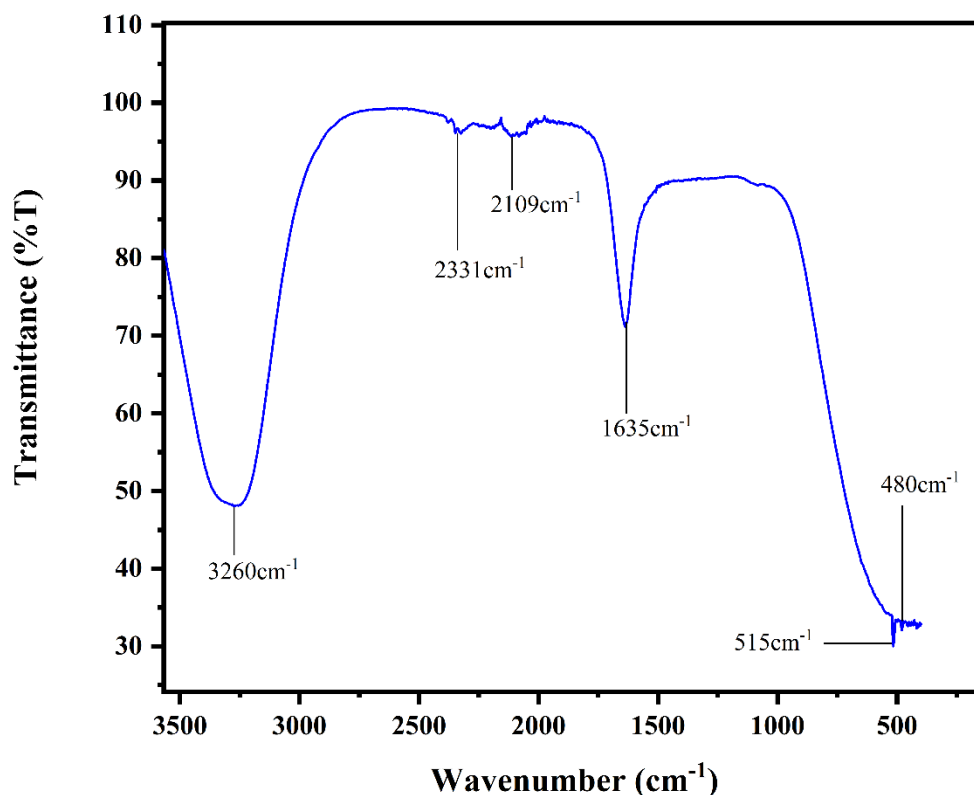

Figure 2: FTIR spectrum of synthesized manganese oxide nanoparticles

XRD analysis

XRD technique was used to study the morphology of as-synthesized manganese oxide nanoparticles. The XRD spectrum of chemically synthesized nanoparticles is shown in Figure 3 along with the standard data for MnO (JCPDS/ICDD No. 01-089-2809). The diffraction peaks corresponding to MnO are consistent with the standard JCPDS data. In the figure, distinctive peaks were observed at 34.78°, 40.28°, and 58.86° which were indexed to (111), (220), and (322) crystal planes respectively; which are comparable with the standard values and suggested by the

literature [25]. The average crystallite size of the synthesized nanoparticles was calculated by using Debye-Scherrer's equation (as given below) and it was found to be 11 nm.

Debye-Scherrer equation is given as,

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where 'λ' is the wavelength of the X-ray, 'β' is full width at half maximum (FWHM), 'θ' is the diffraction angle, and 'D' is the average particle size.
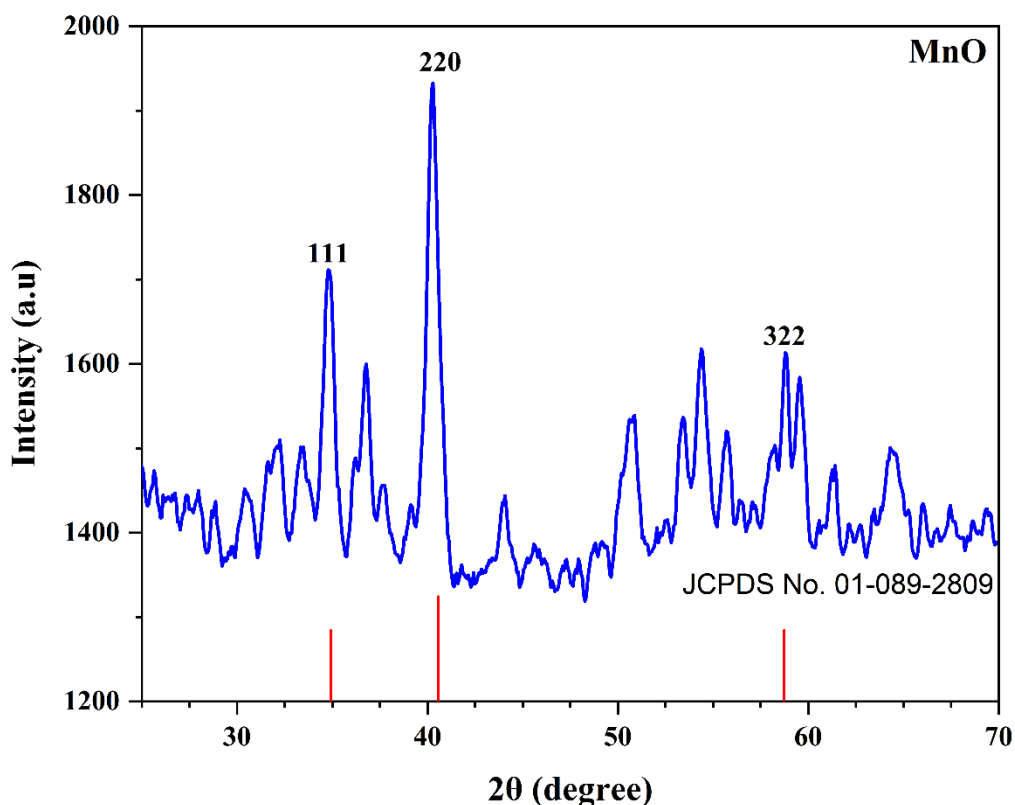

Figure 3: XRD pattern of synthesized manganese oxide nanoparticles

EDX analysis

The energy dispersive x-ray spectroscopic studies were done to know the elemental composition of the synthesized manganese oxide nanoparticles. The EDX spectrum of MnO nanoparticles is shown in Figure 4 and the characteristics peaks confirm the presence of Mn and O in the sample.

The weight (%) of elements present in the synthesized nanoparticles showed that the Mn and O are present in appropriate amounts [26]. The results of the EDX analysis are entirely in agreement with XRD and FTIR analysis results.
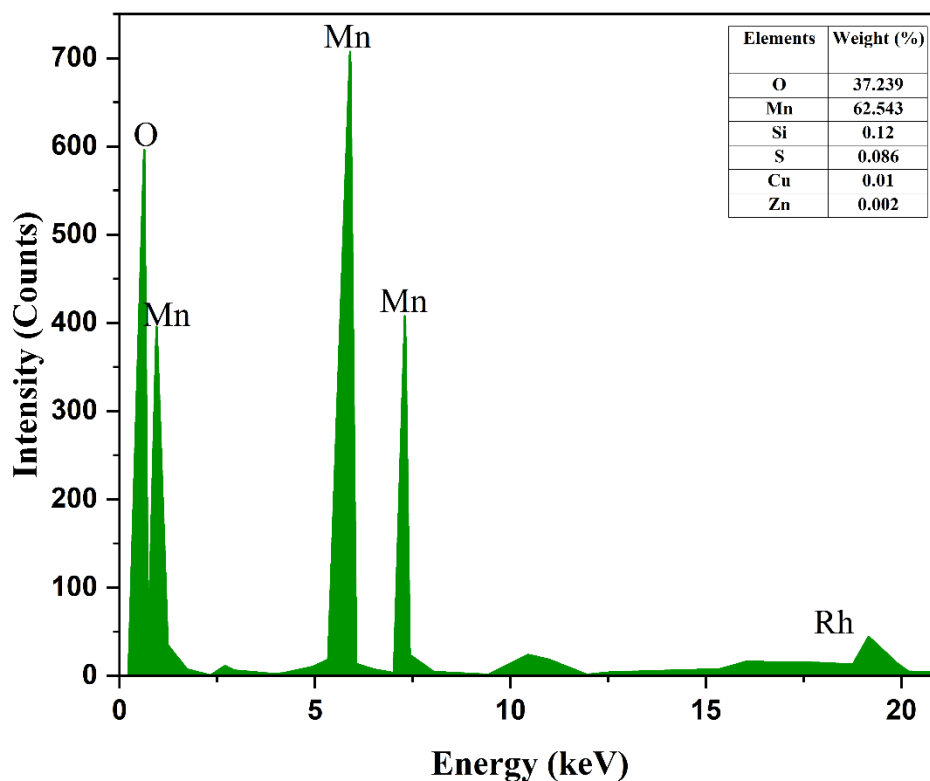

Elements	Weight (%)
O	37.239
Mn	62.543
Si	0.12
S	0.086
Cu	0.01
Zn	0.002

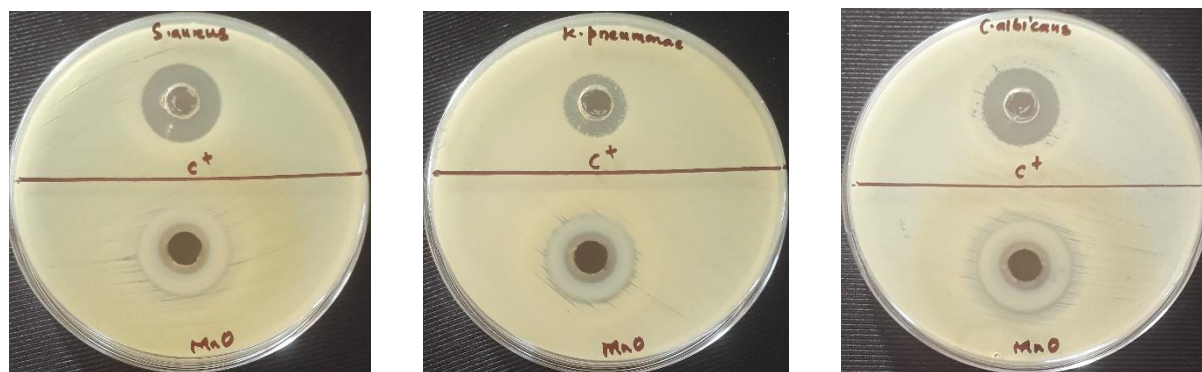**Figure 4: EDX pattern of synthesized manganese oxide nanoparticles**

Antimicrobial activity

The antimicrobial activity of nanoparticles against different microbes was evaluated by measuring the diameter of the inhibition zone in the disc diffusion method. A zone of inhibition was observed against all tested microorganisms, which disclosed manganese oxide nanoparticles as a potential antimicrobial agent. The impact of manganese oxide nanoparticles in the growth of bacterial and fungal strains is shown in Figure 5 and their zone of inhibition value is shown in Table 1. The results show that the Gram-positive bacteria *S. aureus*, Gram-negative bacteria *K. pnuemoniae*, and fungus *C. albicans* were susceptible to MnO nanoparticles with a zone of inhibition of 23 mm.

Table 1: Antibacterial activity of synthesized nanoparticles against pathogenic bacterial and fungus species

Microbes	Reference Culture	Type	Positive Control (c+) mm	Zone of Inhibition (mm)
Staphylococcus aureus	ATCC 6538P	Gram +ve Bacteria	20	23
Klebsiella Pnuemoniae	ATCC 700603	Gram -ve Bacteria	16	23
Candida albicans	ATCC 2091	Fungus	20	23

Positive control = Kanamycin (10 μL of concentration 5 mg/mL)

Figure 5: Antimicrobial activity of synthesized manganese oxide nanoparticles against
(A) *Staphylococcus aureus,* **(B)** *Klebsiella pnuemoniae,* **(C)** *Candida albicans*

Conclusions

Manganese oxide nanoparticles were synthesized by simple co-precipitation method by using manganous sulphate and sodium hydroxide. The synthetic approach was simple, cost-effective, and time-efficient avoiding tedious experimental work and high-energy application. The synthesized nanoparticles were characterized by UV, FTIR, XRD, and EDX, and their antimicrobial property was examined by disc diffusion method. The UV-Vis spectra study confirmed the presence of manganese oxide nanoparticles and FTIR spectral analysis revealed the characteristic peak of the Mn-O bond. The average size of synthesized nanoparticles was predicted by XRD spectra and the elemental composition of the nanoparticles was determined by

EDX analysis. The antimicrobial efficiency of MnO nanoparticles was demonstrated against two bacterial strains and one fungal strain and revealed that it can be used as an antimicrobial agent.

Acknowledgments

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