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Synthesis And Characterization Of Manganese Dioxide Nanoparticles Prepared By Chemical Preciptation Method

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P A P E R I N F O

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A B S T R A C T

In this research, manganese dioxide nanoparticles (MnO_2 NPs) were prepared using a simple chemical precipitation method, using sodium thiosulfate ($Na_2S_2O_3$) as an effective reducing agent to control the size and shape of the nanoparticle crystals. This method represents a promising approach for preparing nanomaterials due to the possibility of tuning their structural properties under mild conditions, opening the way for innovative medical applications. The prepared particles were characterized using advanced analytical techniques, including Ultraviolet-visible (UV-Vis) spectroscopy to study the optical properties, Fourier-transform infrared (FTIR) spectroscopy to determine the chemical bonds, X-ray diffraction (XRD) to analyze the crystal phase and calculate the crystal size. The results showed that the particles belong to the α-MnO_2 phase and crystallized in the tetragonal crystal system, Transmission electron microscopy (TEM) and scanning electron microscopy (FESEM) to study the shape and size, with images showing that the particles exhibit a somewhat spherical structure, with slightly heterogeneous proportions, Energy-dispersive X-ray (EDX) analysis to determine the elemental composition.The results showed that the prepared particles are characterized by their small size, relatively regular shape, and large surface area, characteristics that contribute to improving their effectiveness in biological applications. Manganese dioxide nanoparticles show promising potential in the medical field, especially in nanomaterial-based therapeutic and diagnostic applications.

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NOMENCLATURE			
MnO2	Mnganese dioxide	FTIR	Fourier Transform Infrared spectroscopy
NPs	Nanoparticles	XRD	X-ray Diffraction
$N_2S_2O_3$	Sodium thiosulfate	TEM	Transmission Electron Microscopy
UV-Vis	Ultraviolet-Visible spectroscopy	FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray spectroscopy	GSH	Glutathione
eV	Electron Volt	%	Percent transmittance
cm^{-1}	Wavenumber	Sol-gel	Solution-Gelation

1. INTRODUCTION

Nanomaterials science has witnessed many developments in recent years due to their unique properties that cannot be found in their bulk counterparts. Small particle size within the scale (1-100 nanometers) leads to changes in physical and chemical properties such as high surface area, optical properties, catalytic efficiency, and electrical conductivity make the resulting nanomaterials unique and ideal for numerous applications, such as energy, the environment,

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electronics, and medicine (Hajinasiri & Esmaeili, 2022). Among these materials, manganese dioxide (MnO_2) is a substance with multiple crystalline forms, and it also has the ability to react strongly as an oxidizing agent, It also has the advantage of chemical stability and wide applications. The properties of its molecules are significantly improved when prepared using nano-methods(Hoseinpour et al., 2018). These properties include high catalytic properties(Sobańska et al., 2021), ease of surface modification(Shi et al., 2018), antibacterial properties(Ikram et al., 2024) , high surface area(Wang et al., 2017), high environmental compatibility, and other properties, which makes it more effective in practical applications such as medicine(Yang et al., 2017). MnO_2 NPs are an ideal candidate in medical applications that combine diagnosis and therapy (neurotherapeutics) (Sisakhtnezhad et al., 2023). MnO_2 exploits the properties of the tumor environment, such as high glutathione (GSH) (Yang et al., 2021), and low oxygen levels (hypoxia), to perform several functions, such as generating oxygen within the tumor(Chen et al., 2023) , and improving the effectiveness of chemotherapy and radiotherapy (Song et al., 2016) It also activates the response to treatment by stimulating oxidative stress within cancer cells(Yang et al., 2021) (Bonet-Aleta et al., 2022). MnO_2 also has the ability to modify its surface to work on the synthesis of anti-cancer drugs(Liu et al., 2024) , or to coat it with molecules directed towards specific receptors present on the surface of cancer cells, which makes it a directed and effective drug carrier (Xie et al., 2024). Several methods have been developed to prepare MnO_2 NPs, including chemical methods such as co-precipitation (Kapil et al., 2024) (Kahattha & Santhaveesuk, 2019) , sol-gel (Tang et al., 2014) , hydrothermal synthesis (Islam et al., 2024), and chemical reduction (Yadav et al., 2023). Physical methods include, laser evaporation(Corrales et al., 2022) , plasma irradiation(Kim et al., 2016) , mechanical grinding(Ochirkhuyag et al., 2020) and high-energy ball milling(Song et al., 2024). In addition to the previous methods, environmentally friendly methods have been developed that use plant extracts as reducing agents (Ghorbani et al., 2023) or use microorganisms to stimulate sedimentation (Ogunyemi et al., 2020) . In this research, we adopted the chemical precipitation method using sodium thiosulfate ($Na_2S_2O_3$) as a reducing agent. It is a method characterized by its simplicity, low cost, and ease of control over reaction conditions. In addition to the fact that the reducing agent ($Na_2S_2O_3$) is a relatively non-toxic compound (Schulz et al., 2010) (Neuwelt et al., 2006) its use allows the formation of nanoparticles with good dispersion without the need for harsh conditions of temperature or pressure .

MATERIALS AND METHOD

1.Materials

Sodium thiosulfate were brought from Alpha Chemika. Manganese sulphate ($MnSO_4.H_2O$, 99%) and sodium hydroxide were purchased from QualiKems Fine Chem and CDH, respectively. Deionized water was utilized as reactor medium.

1.1 .preparation of the reducing Agent:

Weigh 0.09 g from Sodium thiosulfate ($Na_2S_2O_3.5H_2O$) and add 25 mL of deionized water in a 50 ml beaker Stir the solution at room temperature with a magnetic stirrer for 5-10 minutes look at Figure1.

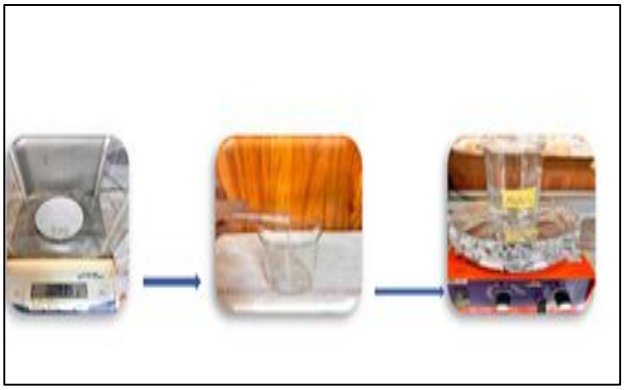

Figure 1: shows the preparation of sodium thiosulfate solution.

1.2. preparation MnO_2 NPs

Weigh 0.1267 g of manganese sulfate ($MnSO_4.H_2O$) and add 75 ml of deionized water in a 200 ml beaker Stir the solution at room temperature with a magnetic stirrer for 10-15 minutes.Gradually add drops of 0.1 M sodium hydroxide solution until the pH becomes 11 (checked with litmus paper), We notice that the solution turns light brown. Then, sodium thiosulfate solution is added as a reducing agent gradually and the temperature is raised to 70°C with magnetic stirring for two hours, the color of the solution gradually changes from light brown to dark brown, indicating the formation of nano manganese dioxide particles. Then we separate the precipitate from the solution using centrifugation (2500 rpm) and then wash the precipitate several times to get rid of impurities. The precipitate is dried at 100°C for 30-60 minutes in a selector drying oven then grind well. , the dried sample is then burned in a laboratory furnace (MAFEL) for 2-3 hours at 400°C to remove organic matter. A very dark brown precipitate is then obtained, which represents nanoscale manganese dioxide particles (MnO_2 NPs). the formation steps of MnO_2 nanoparticles was summariz in Figure 2.

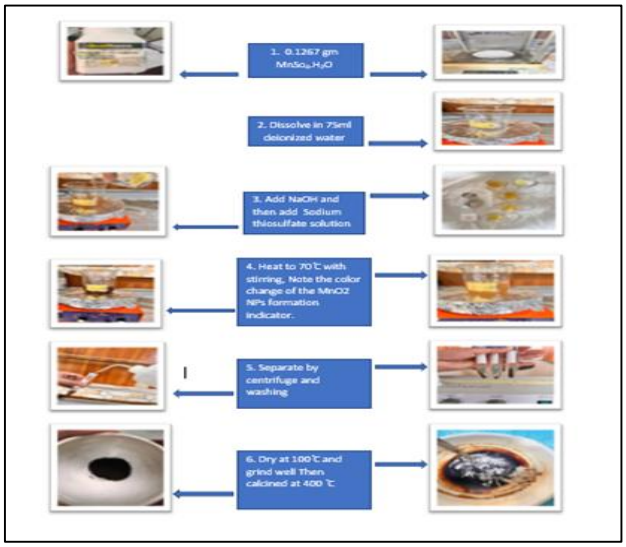

Figure.2 schematic showing the formation process of MnO2 nanoparticle using sodium thiosulfate solution .

CHARACTERIZATION

We studied the optical properties of the prepared manganese dioxide (MnO2) nanoparticles, using UV-visible spectroscopy, The morphology and particle size of the prepared particles were examined using field emission scanning electron microscope (FESEM) and transmission electron microscope (TEM), Elemental analysis was performed using energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD) was used to determine the crystal structure of the prepared MnO2 particles, Fourier transform infrared spectroscopy (FTIR) was used to identify the functional groups on the surfaces of the nanoparticles.

RESULTS AND DISCUSSION

The color change from light brown to dark brown after heating for (1-2) hours is an indicator and evidence of the formation of chemically manufactured MnO2 particles look (Figure 3) In addition to providing further evidence confirming the formation of MnO2 nanoparticles by studying the optical properties of the particles.

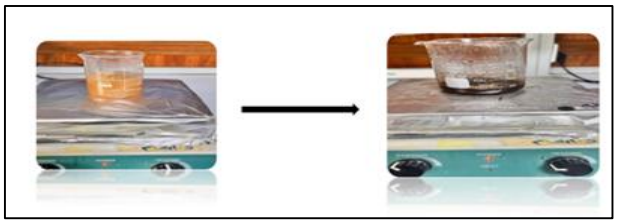

Figure 3. Images shows the color change to brown indicating the formation of MnO2 nanoparticles.

Optical Properties

We studied the optical properties of manganese dioxide nanoparticles formed by reacting manganese sulfate solution MnSO4.H2O with basic medium (NaOH 0.1 M) and adding sodium thiosulfate solution as a chemical reducing agent and ideal reaction conditions of 70°C, pH 11 and reaction time of 2 hours. To know the optical properties of the prepared MnO2 sample, we used the UV-Vis spectroscopy technique. Figure 4(a) shows the spectra of manganese oxide nanoparticles, where an absorption peak appeared at a wavelength of , which is smaller than the spectrum of bulk manganese dioxide, which usually appears at a wavelength of (380 nm). This shift towards lower wavelengths is due to the phenomenon of quantum confinement, where the smaller the size of the produced material and the greater its surface area, the shorter wavelengths appear(Anguraj et al., 2023). The energy band gap of manganese oxide nanoparticles can be estimated from the absorption spectrum using the Tauc plot, according to the following equation:

$$(\alpha h\nu)^n = C(h\nu - E_{(g)})^n$$

Where:

n	0.5 (for direct electron transition allowed)
h	is Planck's constant (6.626×10^{-34} J⁴ s or 4.135×10^{-15} eV⁴)
ν	is the photon frequency,
C	is a constant that depends on the material properties
α	is the absorption coefficient (cm^{-1}),
$E_{(g)}$	is the energy gap.

By plotting the relationship between $(\alpha h\nu)^2$ and photon energy (hν), the energy gap value can be estimated from the intersection of the linear portion of the curve with the horizontal axis. The Tauck curve (Figure 4(b)) showed that the energy gap of the prepared nanoparticles is approximately 4.4 eV, which is higher than the energy gap of the bulk material (MnO_2), which is estimated at approximately 1.6 eV. This difference is due to quantum effects resulting from the small nanoscale size. Reducing the particle dimensions leads to an increase in the energy gap (quantum confinement effect) and a shift in optical absorption toward shorter wavelengths (blue shift). These results support that the prepared particles fall within the true nanoscale and possess distinct electronic and optical properties that differ from their bulk counterparts(Anguraj et al., 2023).

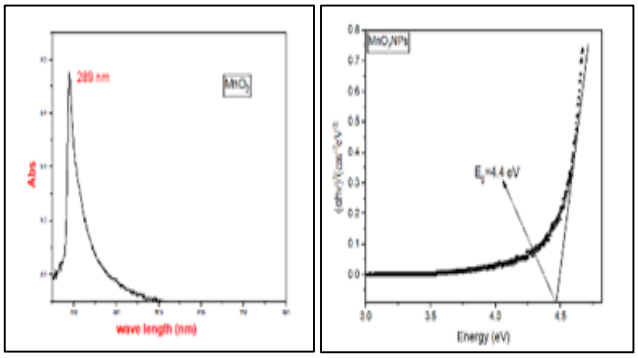

Figure 4 .(a). UV-Vis spectrum of MnO2 nanoparticle, (b) Energy band gap of MnO2 nanoparticleSynthesized by chemical method.

FTIR spectroscopy is a technique used to identify and determine the type of organic and inorganic compounds by knowing the functional groups present in the

$$n\lambda = 2d \sin \Theta \qquad 2$$

n rank of deviation
ʎ which is equal to 0.154 nm(the wavelength of X-ray (Cu Kα))
Θ is angle of diffraction
d layer thickness

where it is found to be equal 2.48 nm.
Where the results of the Scherrer equation:

$$D = K\lambda / \beta \cos \Theta \qquad 3$$

D crystallite size (in radians)
β is the complete width at half maximum
K Constant scherrer = 0.9
λ which is equal to 0.154 nm(the wavelength of X-ray (Cu Kα))
θ is angle of diffraction (in radians)

compound to be analyzed. It is also used to detect the purity of samples, as any impurities present in the sample are identified, which gives it great importance in the quality of scientific research.In addition to its speed and the lack of need for complex sample preparation, it is also non-destructive to the compound after analysis.Figure 5 shows the FTIR spectrum of the prepared sample (MnO2NPs) showing the appearance of two medium intensity peaks at (615 cm-1) and (489 cm-1) due to O-Mn-O stretching vibrations, which clearly indicates the formation of MnO2 nanoparticles. The broad low peak that appeared at (3415 cm-1) is due to the -OH stretching vibrations and H-O-H bending of water molecules, most likely due to the sample being exposed to moisture during transport for analysis, The simple peak that appeared at (1634cm-1) reflects the bending vibrations of water molecules, while the peak at(1118cm-1) indicates the extensions of the S-O or S=O bond resulting from Na2S2O3 residues on the surface of the nanocomposite as a result of its use as a reducing agent (Jaganyi et al., 2013) (Sivakumar & Prabu, 2021).

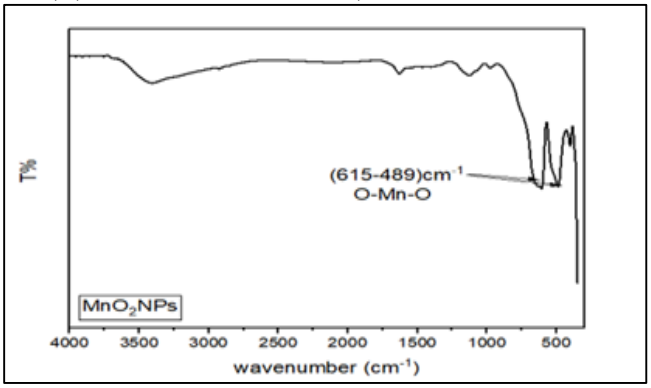

Figure 5. FTIR spectrum plot between wavenumber (cm-1) and transmittance (%) of MnO2 nanoparticle.

Structural Properties

XRD it is a technique that uses X-ray diffraction to study the crystalline structure of solid materials and to determine the size of nanomaterials. The interpreted results confirmed that the prepared material is α MnO2 and has a tetrahedral crystal structure. According to the apparent peaks (18.25) (29.01) (32.68) (36.23) (60.11) , the diffraction angles are consistent , With crystal faces (101) (310) (102) (110) (521) respectively look at Figure (6), according to the card of MnO2(JCPDS card No. 44-0141), These results are very similar to previous results of (Abdullah et al., 2021), The crystal structure was confirmed by the Bragg equation :

$$(\alpha h\nu)^n = C(h\nu - E_{(g)})^n \qquad 1$$

Where:
The crystallite size was found about 9.118 nm.
Field Emission Scanning Electron Microscopy (FESEM) analysis: This technique is used to provide high-resolution images of the surfaces of nanomaterials

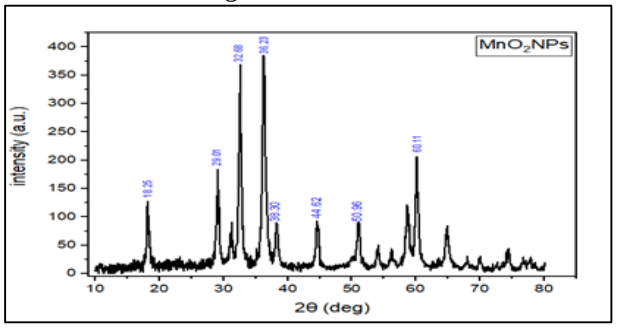

Figure 6: XRD pattern of α-MnO2 nanoparticles

for study and determination of surface composition with high accuracy. The morphology of the MnO2 NPs sample shows that the particles are irregularly spherical in shape due to their small external dimensions and high surface area, with a diameter of approximately 40 nm look to Figure 7(a), The particles are irregularly distributed with clear clumps of dark and light colours as a result of exposure to moisture during storage(Vetrikarasan et al., 2023) (Sivakumar & Prabu, 2021).Elemental analysis confirms that the (EDX) of the sample is composed of 71% manganese and 29% oxygen note Figure 7(b). The presence of gold in the analysis result is due to the sample being coated with gold to increase conductivity, as the sample is a semiconducto(Ghandali et al., 2024).

Transmission Electron Microscopy (TEM) This technique is one of the most accurate techniques, as it uses a beam of electrons that passes through the sample and is not limited to just its surface. This technique is used to study the crystal structure accurately, image the particles from the inside, and determine the shape and size of the particles of the sample, The results, as shown in Figure 7(C), showed that the particles had a somewhat spherical shape with simple clusters. Using the Image J analysis program, it appeared that the nano-size was approximately (Ghorbani et al., 2023).

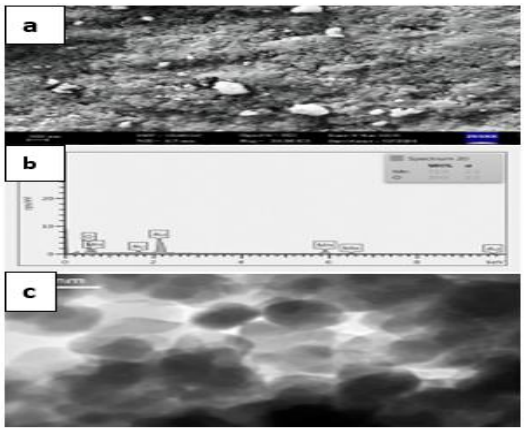

Figure 7 : FESEM aimages of MnO2 nanoparticle synthesized in the presence of Na2S2O3 (a) , EDX chart for MnO2 (b) ,and TEM image (c) .

CONCLUSION

In this study, nanosized manganese dioxide (MnO_2) was prepared by a simple chemical method using sodium thiosulfate ($Na_2S_2O_3$) as a reducing agent. This agent proved effective in controlling the size and structure of the crystalline particles. X-ray diffraction (XRD) analysis results showed that the prepared material crystallizes in the well-known α-MnO_2 crystal phase and belongs to the tetragonal crystal system, indicating the regularity of the material's structural conformation. The size of the nanocrystals was calculated using the Scherrer equation, with an average size of approximately 9.118 nm, confirming the nanoscale nature of the produced particles.Various characterization techniques (UV-Vis, FTIR, XRD, SEM, TEM, and EDX) also revealed that the prepared particles exhibit small sizes and favorable surface structures. Transmission electron microscope (TEM) and scanning electron microscope (FESEM) images revealed that the particles were relatively spherical in shape, with some slight variation in the degree of homogeneity, possibly due to slight differences in crystal growth rates during the reaction. This spherical formation contributes to an increased relative surface area, an important factor in biological applications.Based on the acquired structural and morphological properties, including a stable crystalline phase, regular nanoscale size, and favorable surface area, the prepared nano-MnO_2 exhibits promising potential for multiple future medical applications, especially in areas requiring high reactivity and improved surface properties.

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