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Research Paper

SYNTHESIS AND CHARACTERIZATION STUDY OF MICRO STRUCTURAL AND MAGNETIC PROPERTIES - USING CHEMICAL PRECIPITATIVE METHOD

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Abstract

At presently, manganese oxide nanoparticles (MnO_2 NPs) have intrigued material science researches XRD widely owing to its extensive variety of applications. In view of the study, the manganeseoxide nanomaterials were prepared via using manganese chloride Tetra hydrates salts. This samples were considered to novelty the structural, functional andmagnetic properties by XRD, VSM, HR-SEM and EDAXcorrespondingly. An attempt is finishedtowardreview thecomplete field dependent magnetization study of the synthesized material is presented. Structural studies by XRDindicate that the synthesized material as tetragonal crystal structure. The Antiferro magnetic behavior was detected at room temperature with no saturation magnetization and hysteresis in the region of measured field strength. These dimensions as a role of temperature and field strength showed a reduction in Anferromagnetic temperature.The HR SEM images revealed typical two-dimensional layer sheet-like morphologies with multiple folds for non-intercalated MnO2 whileinterpolated MnO2 presentedfurther flattered and planar shaped. The EDAX spectrum confirmed the presence of manganese oxide.

Keywords: *Nanoparticles; Manganese oxide; Metal oxide; Transition metal oxide,HR-SEM,,VSM*

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Introduction

Materials at the nanoscale have recently taken up a lot of attention among researchers due to their sole physical and chemical appearancesassociatedtoward the bulk counterparts.. A nanomaterial is defined as any item with at least one dimension on the nanoscale scale.Nanomaterials are categorized according to their dimensions..Oxides can also be utilised to create microelectronic circuits, sensors, piezoelectric devices, fuel cells, corrosion-resistant surface coatings, and other devices. Metal oxides have piqued material scientists' interest owing to an optical, electrical, thermal, magnetic, mechanical, and catalytic properties, which type them scientifically and technically important. Metal oxide nanostructures have recently received a lot of interest because of their potential use as functional components for nanoelectronics, optoelectronics, and sensing devices with an electronic structure. They may be metallic, semiconducting, or insulating in nature. It is essential to understand the link among the properties and structure (both physical and chemical) of oxide materials and catalysis [**Vijayamari .,2016**][Foster, 2006] and [Hasan Bagheri, 2020],.Manganeseoxidesis one of the most common minerals on the world and they may be found in a wide range of natural sediments, soils, and ores. They're also in desert rock varnish and marine manganese oxides [Achurra2009],Among them, Manganese Oxide (MnO) has a wide range of potential uses in both industrial and commercial settings. Manganese (Mn) oxide is the most powerful natural oxidant agent and may be found in a variety of natural habitats and oxidation states such as Mn(II), Mn(III), and Mn(IV) and is connected to their oxidation state, crystalline phase, and surface area. [2019] Vicentede Oliveira Sousa Neto. Because of its excellent corrosion resistance, MnO2 has uses in catalysis, hazardous waste cleanup, as additives in refractory, paint, and superconductor goods, and in steel manufacture [Kumari et al., 2009]. Manganese-oxide nanoparticles have piqued the interest of researchers due to their potential uses in electrical, optical, and mechanical devices based on changing oxidation states. Metal oxide nanopowder is synthesised using several techniques, although published procedures have little control over particle functioning [Lind., 1988].As a result, it is critical to change the cost-effective method in a chemical and environmentally friendly way.TheseMnO nanoparticles are significant due to their distinct characteristics, which include a high surface-to-volume ratio, high crystallinity, chemical purity, and phase selectivity when compared to their bulk equivalent. It has been observed that the physical and chemical characteristics of a substance are related to its stoichiometry, particle size, and shape. MnO2, Mn3O4 nanoparticles have

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the potential to be utilised in a variety of applications, including electrodes [Lind1988], catalysis [Xi, 2004], sensors [Li., 1997], and optoelectronics [Shchukin., 2007].

Experimental

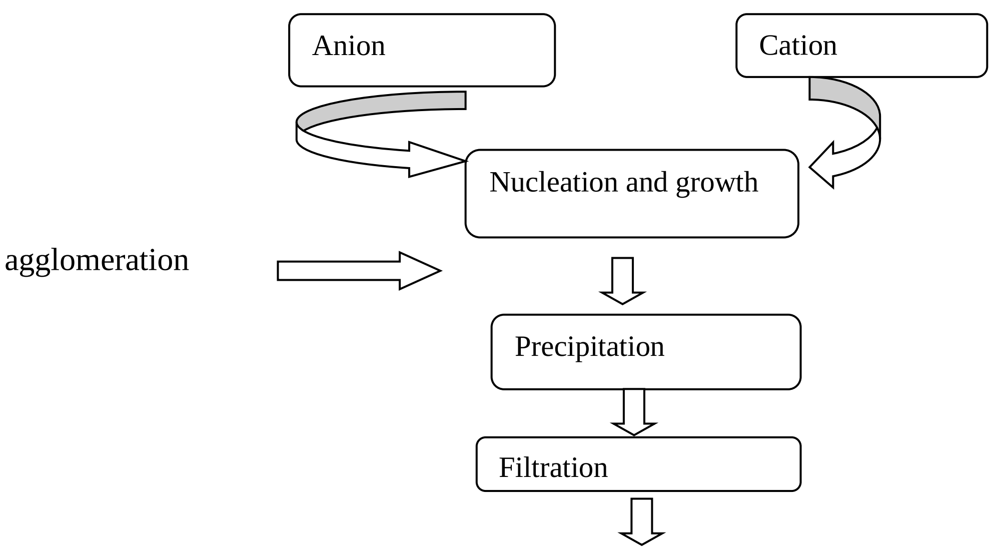
Materials and Methods

chemicals

Manganese Chloride [$Mn(Cl_2)_2.6H_2O$], AR/LR grade, which is commercially accessible. 4H2O (98%), sodium hydroxide NaOH (99%), and Trisodium citrate (98%) were produced and utilised without additional purificationASimple chemical route was used to synthesize manganese oxide nanoparticles. X-ray powder diffraction method FTIR and VSM were used to characterize structural and vibrational sample magnetometer studies respectively.

At room temperature, a manganese oxide nanoparticle was created using a simple chemical precipitation technique. The synthesis process for 1M MnCl2 was thoroughly documented. In 100 ml of distilled water containing 0.66 g of NaOH aqueous solution, 4H2O was dissolved. With continual stirring, 25 mg of trisodiumcitrate was added as a surfactant, which can prevent nanoparticle reunion. After 24 hours of stirring, a brown colour precipitate was produced, indicating that the reaction had completed. The precipitate produced was filtered, washed several times with Deionised water, followed by ethanol, and dried overnight in a hot air oven at about 80 °C. The surfactant Trisodium citrate was removed by calcining the as-prepared sample at 500 °C for 4 hours under air at a ramping rate of 5 °C min-1.

Co-precipitate method



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Calcinations

Fig 3.1 photo image of typical Mn-O2 preparation process

RESULTS AND DISCUSSIONS

X-RD pattern of manganese oxide nanopowder

As shown in Fig.3.1, the XRD pattern is utilised to identify the phase and purity of the produced MnO2 nanoparticles. The diffraction peak locations are acquired using an XRD diffract metre at the values of 18.1°, 29°, 32.4°, 36.2°, and 60°, which correspond to the crystal planes (101), (112), (103), (211), and (224), respectively. All of the distinctive diffraction peaks are properly indexed to and agree with the previously reported tetragonal structure of MnO2 single phase (JCPDS card no. 24-0734). (Vazques-olmos 2005, America.)

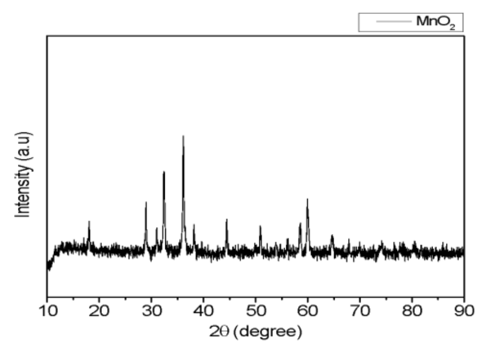

Fig.3.2 shows the XRD pattern of the annealedmanganese dioxide nonamaterial

There is no proof that the diffraction peaks for impurities are present, indicating that the particles are of high purity. Furthermore, tiny peaks with high intensities indicate the particles' excellent crystallinity. The diffraction peaks are extremely broad hump and have a very low intensity, implying that the size of the produced Mn3O4 nanoparticles is quite tiny [Y.Li, H.Tan 2011].

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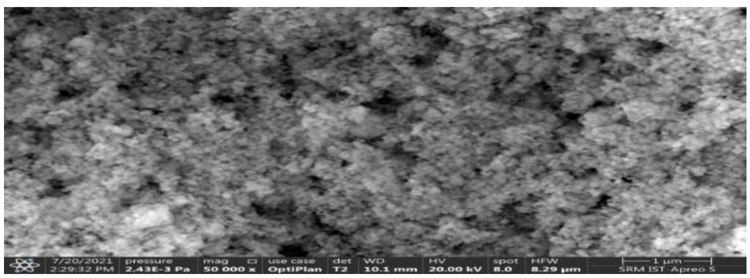
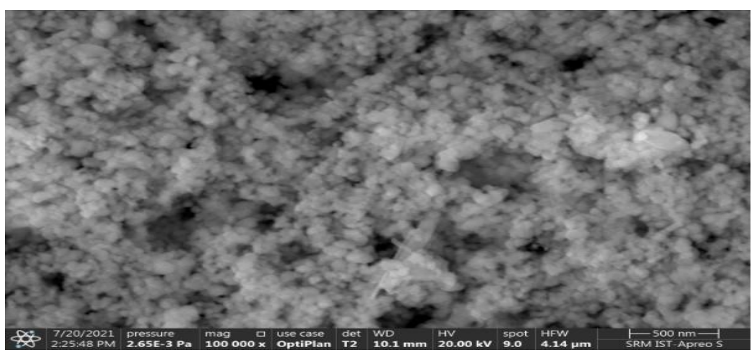
Morphological observations:

Using a High-Resolution Scanning Electron Microscope (HRSEM), the morphology, particle size, and crystallinity of MnO2 nanoparticles were studied (Fig. 3.4). MnO2 has agglomerated well-defined spherical particles as well as a nanosheet-like shape, as seen in Fig.3.3. The HR SEM images revealed typical two-dimensional layer sheet-like morphologies with multiple folds for non-intercalated MnO2. Interestingly, many small particles of MnO2 are also seen in the micrographs which are lying over the nanosheet.



MnO_2

(a)



MnO_2

(b)

Fig .4.HR-SEM images of MnO2

Magnetic behavior of MnO_2

Figure 1 depicts the M-H curve of MnO2 nanoparticles at room temperature. The measured M-H curve of MnO2 indicates that magnetization in the low field area tends to saturate, but magnetization in the high field zone clearly displays unsaturated open linearity. The saturating component is connected to the particle core's uncompensated moment, whereas the non-saturating

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part is related to the spins of the disordered surface. This finding shows that the ferromagnetic component follows the Langevin pattern, but the antiferromagnetic component has a linear dependency. The values of remanent magnetization and coercivity of $MnO_2$are listed in Table 3.2.

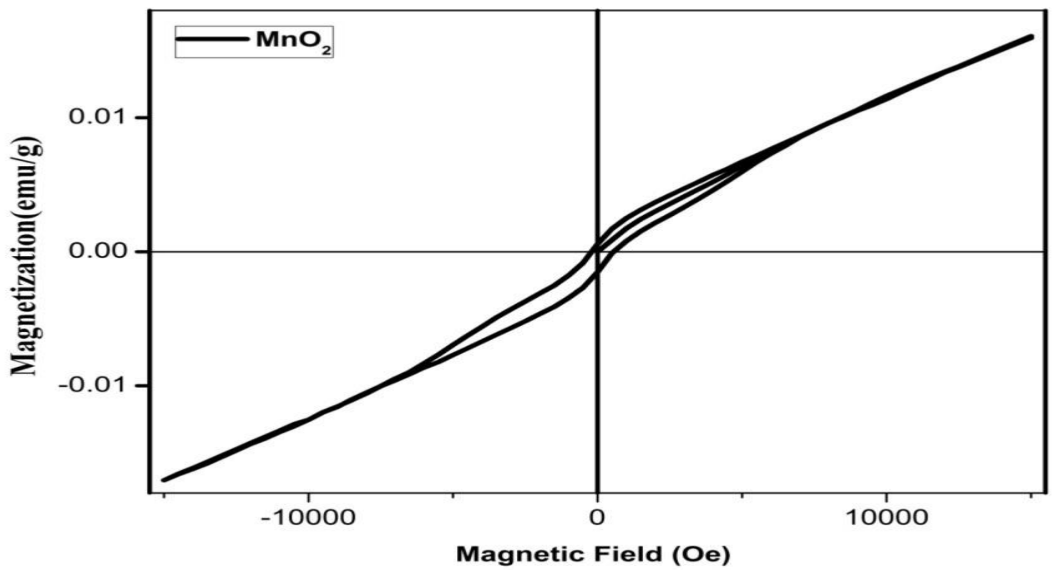

Table:1 Magnetization values of MnO_2

S.No	Remanent Magnetization (M_r x 10^{-3}) inemu/g	Coercivity (H_{ci}) Oe
MnO_2	1.047	380

CONCLUSION

The present study demonstrates the effect of the structural,magnetic properties, microstructural and elemental analysis of Mn_2O nanostructures prepared through a simple chemical precipitation method. Additionally, the products annealed at 500°C were analyzed for their structural, functional and magnetic properties analyzed. The XRD patterns revealed that the particles exhibit a pure cubic structure. Simple chemical method has been used to successfully prepare MnO2nanoparticles, which

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are indexed as tetragonal Pyrolusite.The nanoparticles were estimated to be between 20 and 30 nm in size. The Antiferro magnetic natures` wasobserved through the room temperature with there is no saturation magnetization as followed by hysteresis in the section of measured field strength. In these measurements as a purpose of temperature and field strength displayed a reduction trendyAnferromagnetic temperature..HR-SEM study reveals spherical shape morphology with diameters ranging from 20 to 30 nm. Photocatalysis (e.g., CO2 reduction, CH3 CHO degradation, dye degradation, etc.) is one of the potential uses of the produced materials, as are dye sensitised solar cells, lithium batteries, supercapacitor nanofluids with high thermal conductivity, heavy metal ion removal, and MRI .This chemical way can be measured as an cheap and facile style to make MnO2 nanoparticle.

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