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Investigations on tri manganese tetra oxide nano particles prepared by thermal decomposition

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Oxides of manganese have large number of applications in the field of sensors, piezoelectric crystals etc. In the present work, Mn_3O_4 nano materials were synthesized by using manganese acetate, adopting the method of thermal decomposition. The Nano materials thus prepared were characterized by employing various techniques like PXRD, FTIR, UV and Thermal analyses. The average particle size, calculated using Debye-Scherrer formula, was found to be in the range of 51 – 62 nm. The presence of Mn_3O_4 is also confirmed from FTIR. Thermal studies were also carried out. The optical band gap for the prepared nano materials was obtained from the UV-spectroscopic studies.

Keywords: Nano particles, thermal decomposition, PXRD, FTIR, UV, thermal studies.

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1. Introduction

Nanotechnology provides the ability to create new materials or devices with new functions and properties. The current age is characterized by increased technological advances and rapid nanotechnology development [1]. Technologies with such a system of materials having at least one of its dimensions within 100 nm are referred to as nanoscience. The fundamental, physical, chemical and natural properties of materials are considerably altered as the size of their consistent grains is decreased to the nanometer scale. Most of the nanostructured materials have properties significantly different from those of the bulk materials due to the factors such as large fraction of surface atoms, high surface energy and reduced imperfections [2, 3].

It is evident that nanostructured materials are expected to have improved optical properties compared with bulk materials, as these properties depend on their size, shape and local dielectric environment [4]. In recent years, the synthesis of semiconductor materials with specific size and morphology has attracted a lot of interest due to their significant mechanical, electrical, optical and magnetic properties for potential applications in various fields [5]. Nanoscience and nanotechnology find applications in almost every branch of science and technology, electronics, astrophysics and in medical science. Recent progress in the preparation and characterization of materials on nanometer scale has introduced a new point of view for scientists in reduced dimensions. This will change the crystalline and electronic structure considerably [2].

Transition metal oxide nanoparticles are very attractive due to their variety of applications [6]. Mn_3O_4 is an important transition metal oxide due to its extensive applications in magnetic, electrochemical, lithium ion batteries, catalytic applications, super capacitors and dilute magnetic semiconductors etc. [7]. It is one of the most stable oxides of manganese [6]. Due to its nanometer size and large surface area, different morphologies are expected to display better performance in all the above mentioned applications [6]. The optical properties are the most fascinating and very useful properties of nano metal oxides. Oxides of manganese have optical properties which are associated with the intrinsic and extrinsic effect. The optical transition between the electrons in the conduction band and holes in the valence band causes the intrinsic effect. Mn_3O_4 is a direct band gap semiconductor and a transparent conducting material. By using a large number of techniques such as optical absorption, photo-reflection, photoluminescence etc. the optical transitions can be widely used in variety of applications.

In the present study, Mn_3O_4 nanoparticles were prepared using the thermal decomposition technique. Manganese oxide nanoparticles are water insoluble, brownish black powder and have no odor. Mn_3O_4 has the spinel structure [3], where the oxide ions are cubically closed packed and the Mn^{II} ions occupy tetrahedral sites whereas the Mn^{III} occupy the octahedral sites [3, 7].

2. Experimental procedure

Tri manganese tetra oxide nanoparticles were prepared by adopting the method of thermal decomposition. The required quantity of manganese acetate tetra hydrate [$(CH_3COO)_2Mn{\cdot}4H_2O$] in its dry form is taken in a ceramic container and is heated in a muffle furnace for a period of 24 hrs, maintained at a temperature of 950 °C. As a result, a brownish black powder of Mn_3O_4 particles was obtained and collected for further characterization.

3. Result and discussion

3.1. XRD characterization

The prepared Mn_3O_4 nanomaterials were characterized by powder X-ray diffraction. X-ray powder diffraction measurements were performed using the X-ray diffractometer operating with Cu-Kα radiation ($\lambda = 0.15406$ nm). Fig. 1 shows the X-ray diffraction spectra for the prepared Mn_3O_4 nanoparticles.

Figure 1 shows the crystalline structure with several significant peaks that can be readily indexed as (112), (200), (103), (211), (004), (220), (105), (224), (312) and (400) crystal planes respectively, which is in good agreement with the standard value, JCPDS No. 024-0734. All the peaks in the pattern can be indexed to a tetragonal phase with lattice constants $a = b = 5.7621$ Å, $c = 9.4696$ Å.

The particle size is calculated according to Debye-Scherrer formula:

$$2d = \frac{0.9\lambda}{\beta cos\theta},$$

where λ is the wave length of Cu-Kα radiation, β is the full width at half maximum, corresponding to the diffraction angle 2θ. The crystal size for the prepared Mn_3O_4 nano particle was found to be 51 – 62 nm.

3.2. FTIR characterization

Figure 2 shows the FTIR spectra of the prepared Mn_3O_4 nano particles which show several significant absorption peaks that help to understand various chemical bonds present in it.

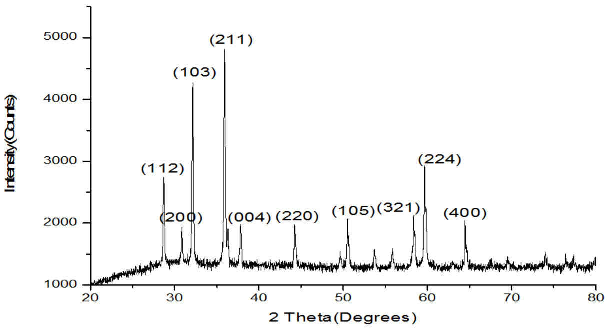

FIG. 1. XRD Spectra for the prepared Mn_3O_4 nano particles

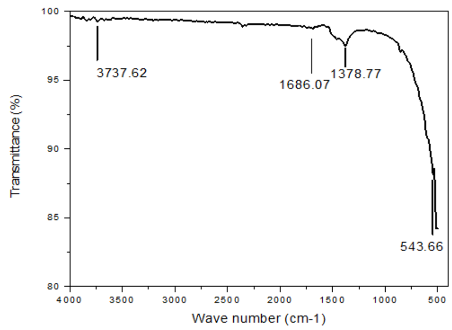

FIG. 2. FTIR Spectra for the prepared Mn_3O_4 nano particle

The vibration frequency located at 543 cm^{-1} corresponds to the vibration of Mn–O stretching modes. Moreover, the broad band at 3738 cm^{-1} and the narrow one at 1666 cm^{-1} correspond to O–H vibrating mode of the adsorbed water. Thus, FTIR provides concrete evidence for the presence of manganese oxide.

3.3. Thermal analysis (TGA/DTA)

The thermal stability of the prepared Mn_3O_4 nano particles was carried out between 250 – 1050 °C.

Figure 3 represent the TGA/DTA spectra for the prepared Mn_3O_4 nano particles. In the temperature range up to 300 °C a weight loss of 2.85 % can be related to the release of weakly adsorbed water molecules. It is found that the sample was completely decomposed within the given range.

3.4. UV characterization

The UV absorption spectra for the prepared Mn_3O_4 nano particle is shown in Fig. 4. The optical absorption peak intensity was found to be 316.507 nm. The optical band gap of the sample is calculated using the formula, $E_g = hc/\lambda$, where E_g – energy gap, c – velocity of light and λ – wave length. The optical band gap for the prepared Mn_3O_4 nano particle was found to be on the order of 3.824 eV.

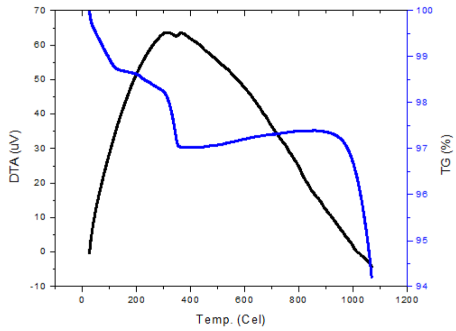

FIG. 3. TGA/DTA Spectra for the prepared Mn_3O_4 nano particle

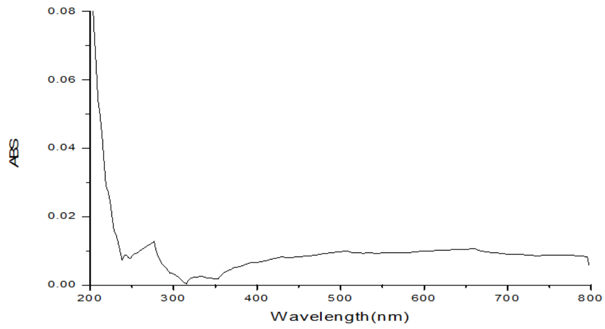

FIG. 4. UV Spectra for the prepared Mn_3O_4 nano particles

4. Conclusions

Mn_3O_4 nano particles were successfully synthesized by using manganese acetate by adopting the method of thermal decomposition. The nano materials thus prepared were characterized by adopting various techniques like XRD, FTIR, UV and thermal analysis. The average particle size is calculated using Debye-Scherrer formula as 56 nm. Presence of Mn_3O_4 is also confirmed from FTIR. Thermal studies were also carried out. The optical band gap for the prepared nanomaterials was found to be 3.824 eV.

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