SYNTHESIS AND CHARACTERIZATION OF MNO_2 VIA CO-PRECIPITATION METHOD

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Abstract:

The plunge to develop eco-friendly procedures for the production of nanoparticles arises from recent nanotechnology research. Manganese Dioxide (MnO_2) nanostructures are promising important applications in batteries, sensors, catalysis and capacitors, etc. MnO_2 nanostructure was synthesized by co-precipitation method. The MnO_2 nanoparticles were characterized by X-Ray Diffraction (XRD), Field-Emission Spectroscopy (FE-SEM) and Elemental X-ray diffraction (EDX). The average particle size of MnO_2 nanoparticles was observed >100 nm.

Keywords: MnO_2 nanoparticles, co-precipitation method, XRD, FE-SEM, EDX.

I. Introduction

Nanomaterials and nanotechnologies attract tremendous attention towards recent researches. New physical properties and new technologies both for sample preparation and device fabrication educe an enormous role in development of nanoscience. There are various methods of preparing nanomaterials including Gas condensation, Vacuum deposition and vaporization, Chemical Vapor Deposition (CVD), Chemical Vapor Condensation (CVC), Sol-Gel and Chemical precipitation (co-precipitation) method. In the present report we are also focusing the Co-precipitation method for the synthesis of MnO_2 nanoparticles. In this method, the required metal cations from a common medium are co-precipitated usually as hydroxides, carbonates, oxalates and citrates.

MnO_2 can exist in different structural forms α, β, γ, δ, ε and λ types and so on. Based on the different (MnO_2) links, formed structure can be divided into three categories: chin like, the sheet or layered and the 3D-structure [1]. MnO_2 is the best catalysts due to its low toxicity and it is the environmental benign. It is the low band gap material (1.33 eV), having high optical constant which also shows good ferroelectric catalytic and properties [2]. The green synthesis of MnO_2 nanoparticles has been reported from *Syzygium aromaticum* i.e. clove extract (CE) as reducing agent as well as stabilizing agent by Vineet *et al*, 2017. Another study, shows production of MnO_2, from *Kalopanax pictus* i.e. castor aralia leaf extract for degradation of dyes by Sun *et al*, 2015. Similarly, Muhamed et al, 2017, reported the biosynthesis of MnO_2 by lemon and turmeric curcumin extract as reducing and capping agent respectively and also investigated their antibacterial and antifungal activities. Wright et al, 2016 reported *Shewanella laihica* as a strongest oxidizer [3-8].

1. Experimental

1.1 Chemicals

In the present work we have been used AR grade chemicals such as, the source of Manganese (Mn) is $MnCl_2$. Liquor ammonia (NH_3), Ethylene glycol ($C_2H_6O_2$) and Double Distilled Water (DDW).

2.2. Methodology

The Co-precipitation method was performed by using manganese salt like Manganese (II) chloride of 0.1M dissolved in100ml double distilled water then 0.67ml ethylene glycol is added with continuous stirring at constant room temperature. While stirring, NH_3 was continuously added till the pH of the solution become 8 to 10. We get the precipitate (ppt). The stirring was continued for 8 hours at a constant room temperature. Brown precipitate (ppt) formed was then washed with double distilled water. Collect the ppt in the form of material residue, then for the centrifugation of residue ppt keep in ultra-sonicator. Run it for 30 min, it repeated again and again 25 times. After that the material was decreases in size by using the sonicator. Annealing it in to furnace at 400^0C temp for 2 hours, we get the dry powder of nanoparticles (Fig1-4).

Figure 1 (a) & (b): Reaction Set-up

Figure 2(a) & (b):

Figure 3(a) & (b): Probe-sonicator and final product of MnO_2 nanoparticles

Reaction mechanism:

$$MnCl_2 + NH_3OH + H_2O + C_2H_6O_2 \Rightarrow [(MnO_2 NH_3 + 2H_2O) + (MnO_2 + C_2H_6Cl_2)]$$

$$MnO_2.NH_3 + C_2H_6Cl_2 + 2H_2O \Rightarrow MnO_2 + NH_3 + H_2O$$

$$MnO_2 + NH_3 + H_2O \text{ (Washing)} \Rightarrow MnO_2$$

2. Characterization

The structural properties of the films were measured by using X-ray diffractometer (XRD) (Bruker D8 Advance) with Cuk$_{\alpha}$ radiations (λ = 1.5406 Å) in the 2θ range from 20 to 80 degree. The surface morphology of the nanostructure thin films was observed by high resolution field emission scanning electron microscopy (FE-SEM, Model: JSM-6701F). The compositional property of thin films was examined using energy-dispersive X-ray spectra (EDX) spectroscopy attached with FE-SEM unit.

3. Results and Discussion

3.1 X-Ray Diffraction (XRD)

The scanning speed of the specimen was maintained 0.5^0/min. fig. 4 shows the XRD pattern of MnO_2 powders. The 2θ peak observed at 23^0, 32.96^0,38.24^0, 45.18^0, 47.30^0, 55.20^0 and 65.80^0 which correspond to the (211), (222), (400), (332), (422), (440) and (622) planes of reflections all the peaks are well matched with standard JCPDS card number **89-4836**. The XRD spectrum reveals that the material is polycrystalline in nature and orthorhombic in structure of surface activated MnO_2. Fig 4 depicts X-ray diffraction pattern of manganese oxide nanoparticles synthesized by Co-precipitation method. The presences of peaks indicate the polycrystalline nature of the MnO_2 nanoparticle.

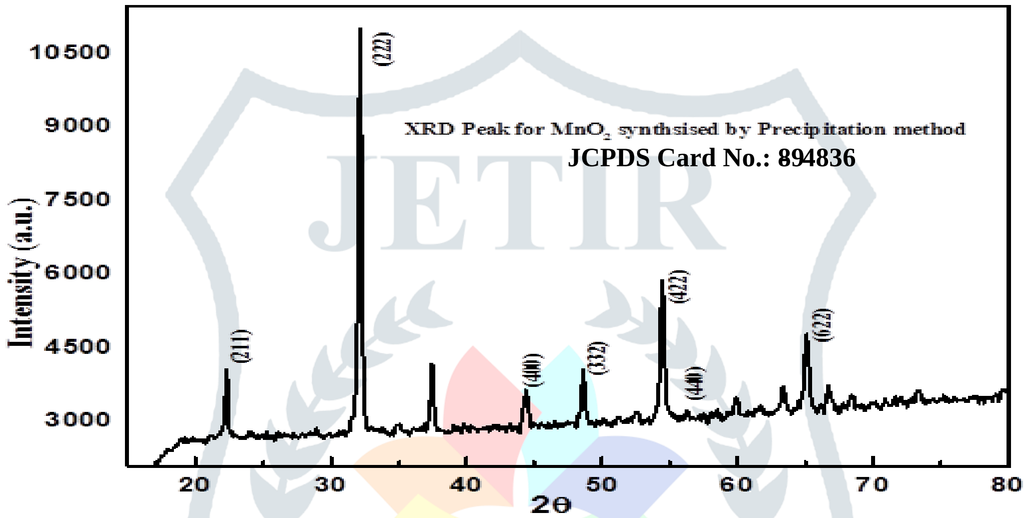

Figure: 4:X-ray diffraction peaks shows the crystalline structure nano-cubes of MnO_2

3.2 Field Emission-Scanning Electron Microscopy (FE-SEM)

Pure MnO_2 material consists of voids and a wide range of randomly distributed grains. The larger grains of MnO_2 occurred due to agglomeration of its smaller grains. Fig. 5 Depicts the nanostructure of MnO_2. The material consists of voids and a wide range of grains with grain sizes ranging from 26 nm to 107 nm distributed non-uniformly. The surface morphological studies of nanoparticles have been performed by Scanning Electron Microscope (SEM). The SEM images of MnO_2 nanoparticles are portrayed in Fig 5. It was well documented that the surface morphology has significant impact on the performance of nanostructure materials. The uniform distributions of grains are observed in the micrographs. The particles are nearly cubic in shape.

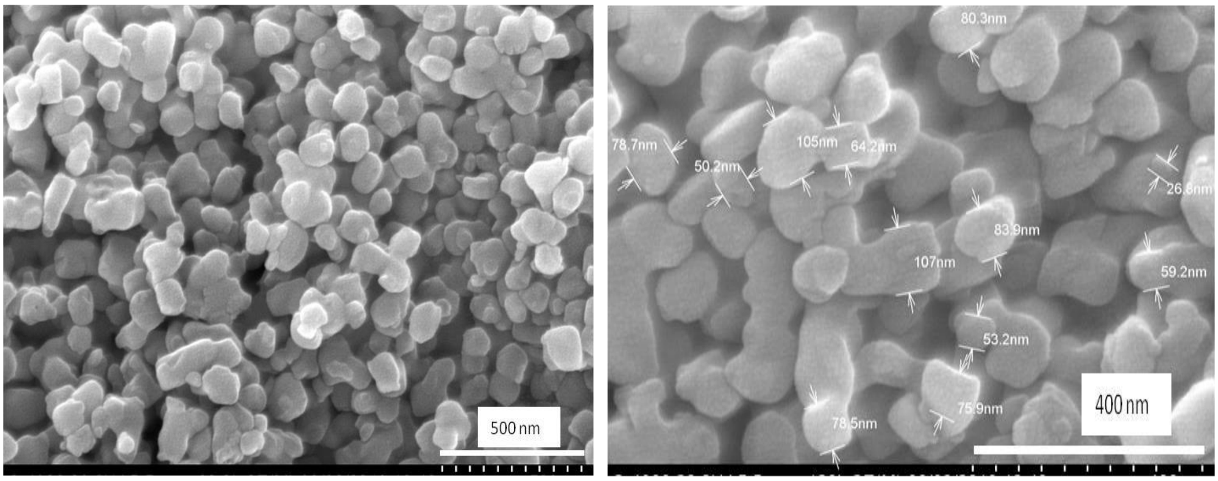

Figure: 5(a) and (b): FE-SEM image shows the nano-cubes of MnO_2

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