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Synthesis, Structural and Morphological Characterization of CTAB- Mn_3O_4 by CO Precipitation Method

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Abstract: We report on the synthesis of CTAB-Mn_3O_4 nanoparticles with the spinel structure. These oxide nanoparticles is obtained from the decomposition of metal acetate precursors synthesized by co-precipitation method .the final product were thoroughly characterized with X-ray powder diffraction (XRD), scanning electron microscopy(SEM) and Fourier transform infrared spectroscopy(FT-IR). Our studies reveal that the shape, size and morphology of precursors and oxides vary significantly with the method of synthesis .XRD and FTIR analyses confirmed the composition and structure of CTAB-Mn_3O_4
Keywords: Spinel; CTAB-Mn_3O_4; co-precipitation method.

Introduction and Experimental:

Tri manganese tetra oxides (Mn_3O_4) as a magnetic transition metal oxides has gained significant attention due to its uses in a wide range of important applications such as molecular adsorption, catalysis, ion exchangers ,high –density magnetic storage media, sensor, electrochemical materials, super capacitors and dilute magnetic semiconductor[1]. In particular, material fabricated on a nanoscale can exhibit better optical, magnetic, thermal and electrical properties than bulk materials. Nano meter-sized manganese oxide (Mn_3O_4), with remarkably, increased surface area and greatly reduced size, are expected to performance better in all the above mentioned application[2].

Mn_3O_4 is a transition metal oxide with the normal spinel structure. Its stable room temperature phase is tetragonal hausmannite ($I4_1/amd$), with Mn^{3+} and Mn^{2+} ions occupying the octahedral and tetrahedral positions of the spinel structure respectively[3]. The oxygen octahedral co-ordination is tetragonally distorted due to the Jahn-Teller effect on the Mn^{3+} ions. Very different routes in the synthesis of Mn_3O_4particles have been reported and a few papers dealing with the subject are cited here in[4]. A good quality phase pure spherical/cubic shape nano powders of $Mn_3O_4$with size ranging from 10-50nm were formed instantly at 50°c by Apte et al. Spherical and rod –like structures with the sizes ranging from 60-100nm was synthesized by Xinli Hao et al .The sample was so sensitive to hydrochloric acid (HCl) resulting in colour change from brown to black. The tetragonal hausmannite Mn_3O_4 films were formed without any pin holes; cracks were synthesized and confirmed by Dubal et al. This indicates the hydrous nature of Mn_3O_4which is responsible for enhanced super capacitors. Mn_3O_4calibers of diameter 50-200nm by calcinations of this PVA/manganese acetate composite fibres were prepared by chang Hu shao et al .This shows calcinations temperature influenced the crystalline phase and morphology of nanofibres .

Spinel oxides of Co and Mn nanocrystals with high uniformity and crystallinity under optimized reaction conditions were synthesized by Azadeh et al. This shows superior catalytic performance when compared to the bulk catalysts. In this work, we report Mn_3O_4 nanoparticles (Nps) have been successfully synthesized by CTAB- Mn_3O_4 using co-precipitation method.

Experimental:

Materials: Manganese acetate ($Mn(CH_3COO)_2.4H_2O$,Cetyltrimethyl ammonium bromide (CTAB), sodium hydroxide(NaOH),acetone, and ethanol were all purchased from Merck and used as received without any further purification .Deionized water was used in all experiments .

Preparation of Mn_3O_4: In a typical procedure, 0.34mol of Mn $(CH_3COO)_2.4H_2O$was added 0.68mol of NaOH aqueous acetone solution (40/60). Then 1.92mmol cetyltrimethyl ammonium bromide (CTAB) was dispersed and homogenized under slowly magnetic stirring, for duration of 10hours. Afterwords the wet precipitate was centrifugally separated and washed by deionized water and ethanol twice, then dried at 70℃ for 18hr under vacuum.

Chacterisation Techniques: X-ray powder diffraction (XRD) analysis was conducted on a rigaku smart Lab diffractometer operated at 40kv and 35ma using cukα radiation (λ=0.15406nm) crystallographic data (lattice parameters, space group) were determined by using the high score software.

Fourier –transform infrared (FTIR) spectra were recorded in the wave number range of 4000-400cm-1 with a Perkin Elmer BXFTIR, spectrometer .The powder samples were ground with KBR and compressed into a pellet to investigate the nature of the chemical bond formed .

The morphology and size of the as-obtained product were investigated using field-emission scanning electron microscopy (FESEM:JEOL-7500,2Kev).

Results and discussion:

XRD Analysis: The XRD pattern of the as-synthesized CTAB -Mn_3O_4 is shown in fig.1. All diffraction peaks were indexed to the tetragonal hausmannite crystal structure model of Mn_3O_4 (which are consistent bulk value-ICDD card number 24-0734) with a lattice constant a=b=5.766Å and c=9.49Å with a small difference in peak intensities. The crystallite size of the as prepared sample was calculated from the major (211) diffraction peak using the Debye Scherrer approximation .The crystallite size was found to be about 60nm.

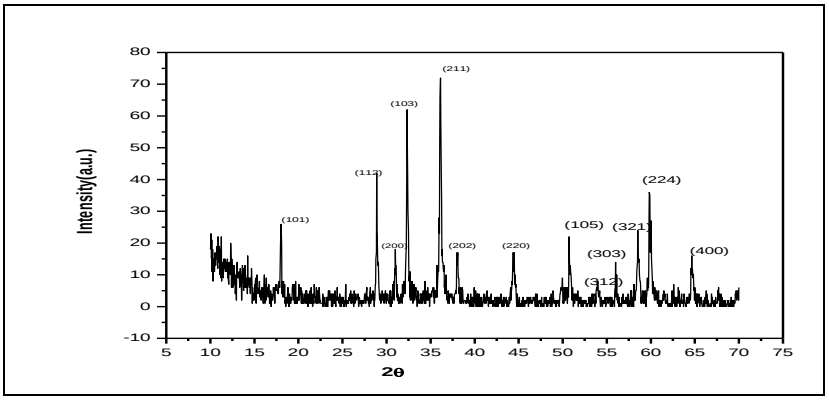

Fig.1.XRD pattern of CTAB-Mn_3O_4nanoparticle

FTIR Analysis: FTIR analysis was performed to investigate the surface characteristics of the prepared samples and the resultant spectra are presented in fig2.In the region from 750 to 500cm^{-1}, two absorption peaks were observed at 747.87 and 521.82cm^{-1}, which may be associated with the coupling of the Mn-O stretching modes of tetrahedral and octahedral sites, as expected from a normal spinel structure[5]. The two intense adsorption peaks located at 2343.07 and 2369.49 cm^{-1} are assigned to the symmetric and asymmetric stretching modes of the $–CH_2$ group of the CTAB surfactant bounded to the surface of Mn_3O_4 respectively. The weak band at 1020.21 cm^{-1}, which can be attributed to Mn-O-H vibration of ethanol molecules on the surface of the hausmanitte (Mn_3O_4) particles. The peak in the range of 3200-3500cm^{-1} is related to the hydroxyl groups on the surface of nanoparticles[6].

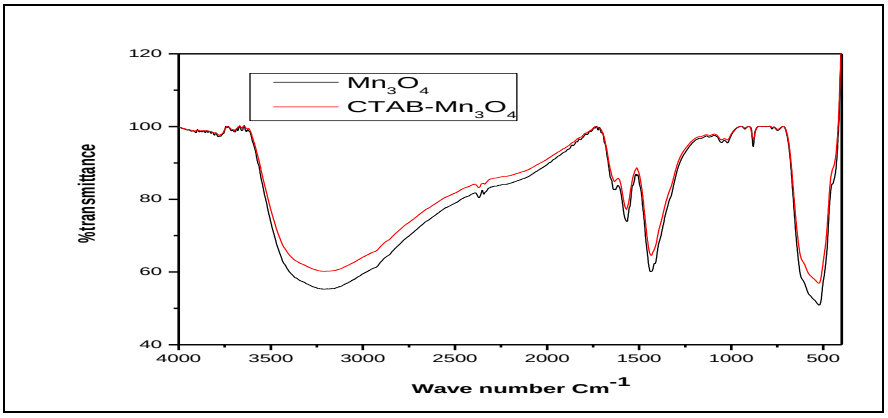

Fig.2. FTIR spectra of CTAB-Mn_3O_4

SEM analysis: The SEM images in fig.3 show the typical morphology of such as-synthesized products prepared at 70°c for 18hr. The scanning electron microscopy corresponding to the oxide is depicted in fig.3. It shows sphere –shaped grain with a narrow particle size distribution around 500 nm and 1µm.

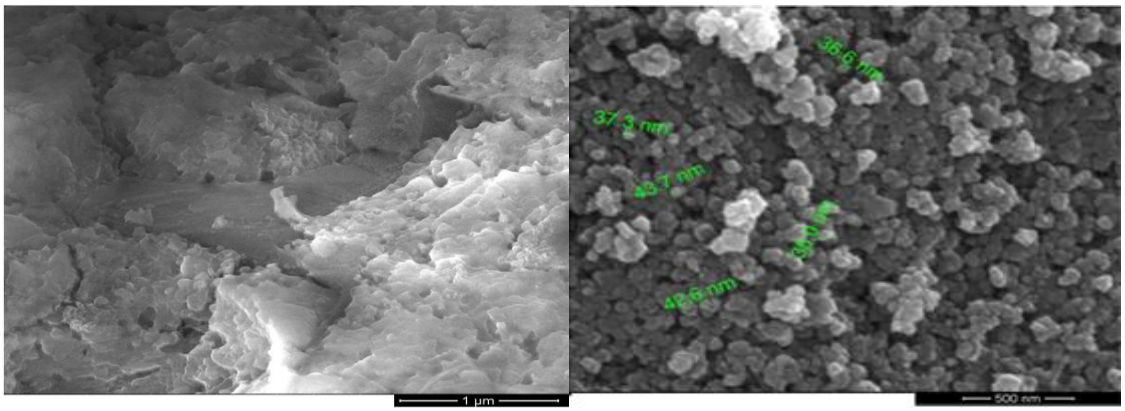

Fig.3.SEM image of CTAB–Mn_3O_4

Conclusion:

A co-precipitation method can be controlled successfully to prepare CTAB-Mn_3O_4 nanoparticles. To the best of our knowledge, this is the report on the synthesis of CTAB-Mn_3O_4nanoparticles by using only Mn $(CH_3COO)_2.4H_2O$ as starting material. XRD and FTIR analysis confirmed the structure of Mn_3O_4(Tetragonal Hausmannite) nanoparticles as spinel oxide.

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