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Synthesis and Characterization of $NiMn_2O_4$ Nanoparticles via Co-Precipitation by Using CMC Solution as a Capping Agent.

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ABSTRACT:

$NiMn_2O_4$ nanoparticles were successfully synthesized in existence composed of naturally occurring substances such as carboxymethylcellulose (CMC). CMC played significant role in fabrication of $NiMn_2O_4$ nanoparticles as capping agent as well as defending agent in development of nanoparticles. Synthesized $NiMn_2O_4$ nanoparticles were characterized using X-ray diffraction (XRD), Scanning electron microscope (SEM) Energy dispersion spectroscopy (EDS) and UV visible Spectroscopy. XRD results confirmed single-phase cubic structure of present sample and lattice parameter was found to be 8.3131Å. XRD results also show good crystallinity with an average crystallite size was found 27.18nm. Bandgap of product was calculated to be nearly 1.7 eV after optical properties of intended products were examined using UV–vis diffuse reflectance spectroscopy.

1. Introduction:

Particles which have diameters of 1 to 100 nanometers are called nanoparticles. These NPs are little items that exist as full unit while retaining their attributes. Numerous shapes and sizes of different nanomaterials have been found in recent years. Different physical properties of nanoparticles compared to their alike bulk counterparts have resulted in renewed interest in science of nanomaterials. Distribution of particle sizes and morphology for instance show unique and enhanced characteristics at nanoscale that aren't found in larger bulk material particles. Nanoparticles can have variety of structures according to various synthesis procedures. [1] These nanostructures are far smaller than everyday objects defined by Newtonian physics yet they are much larger than atoms or molecules defined by quantum mechanics. Nanoparticles have unbelievable qualities, which is why they are so significant in industries such as food, oil, energy and medicine. It would be difficult to detail all uses separately, thus only handful are covered here. [2]

Development of nanoparticles or particles with size between 1 and 100 nm, using variety of synthesis techniques, particle structure and size change is known as nanotechnology. This technology has been adopted by industries such as Separation by magnets, biological technology, storage devices, targeted medication delivery, textile, coating , sensors and pre-concentration of target analyte. [3]

Metallic nanoparticles or metal nanoparticles are novel referrals that have emerged in field of nanomaterials in recent years. [4] Transition metal Nanoparticles are type of Metallic nanoparticles composed of transition metal atoms in Nano scale range includes elements such as Manganese, nickel, copper and, zinc. These metals have common properties such as high melting and boiling points, good electrical conductivity and ability to form complex ions and compounds. Transition metals have partially filled d orbitals which give them unique electronic and optical properties that can be exploited for various applications, they have large surface area relative to volume ratio and they have wide range of oxidation states which allows for variety of chemical reactions. [5]

Preparation techniques have direct impact on characteristics of nanoparticles. Changes in intrinsic distribution of divalent and trivalent cations between both of these sites, as well as particle size, distribution and shape of crystallites, all affect magnetic characteristics. [6] Nanocrystal line compounds may be made using a variety of physical and chemical techniques. [7] Co-precipitate, hydrothermal reaction, sol–gel, nonchemical breakdown and thermal degradation employing organic precursors are some of often utilized chemical techniques. Lately, simple oxides have been produced chemically by reacting inorganic salts with gelatin for a brief period of time at a low temperature. Organic intermediates approach is straightforward strategy among these that is being employed more and more. [8]

A different synthesis technique is to use natural items like mineral oil, carboxymethylcellulose (CMC), etc. as surfactants to create nanocrystal line inorganic substances. In this work, we report on co-precipitation approach used to synthesis $NiMn_2O_4$ nanoparticles utilizing CMC solution as a surfactant. Fact that CMC is a nutritious substance, nontoxic, and biodegradable is crucial consideration when choosing it. Compounds that were obtained underwent structural and morphological characterization.[9]

2. Materials and methods:

Reagents used are Nickel Chloride hexahydrate ($NiCl_2.6H_2O$), Manganese chloride ($MnCl_2$), sodium hydroxide (NaOH) and carboxymethylcellulose (CMC) 1% solution. $NiMn_2O_4$ nanoparticles have been successfully synthesized by wet chemical method (Co-precipitation method). After mixing and stirring for 30 minutes, water-based solutions of 0.2 M $NiCl_2.6H_2O$ and 0.4 M $MnCl_2$ were combined. After adding 3 M NaOH, CMC solution was added to previously described solution as capping agent. PH of mixture was consistently observed and kept within 11–12 range. For 60 minutes, Solution was heated to 80°C while being continuously agitated. After that, mixture was given time to return to ambient temperature. A rather black precipitate has been separated and repeatedly washed with ethanol and water. Following 12-hour drying period at 110°C, precipitate turned dark. Using temperature of 500°C for six hours, samples were heated in furnace. Finally, we obtained confirmed $NiMn_2O_4$ nanoparticles.

3. RESULT AND DISCUSSIONS:

3.1. XRD Analysis

$NiMn_2O_4$nanoparticles were synthesized, and their structure, crystallinity, and phase purity were verified using patterns generated by X-ray diffraction (XRD). Figure 1 shows XRD pattern of synthesized $NiMn_2O_4$. Primary peaked associated with planes (111), (220), (311), (222), (400), (511) and (440) which are exactly matched with stand $NiMn_2O_4$ with JCPDS card Number 00-001-1110 representing cubic structure with space group F. this XRD pattern shows that there is no primary develop in $NiMn_2O_4$nanoparticles.Lattice parameter derived from XRD peak positions are calculated by following formula.

$$a = \frac{\lambda}{2sin\theta}\sqrt{h^2 + k^2 + l^2}$$

Where $\boldsymbol{\lambda}$ is wavelength of X-ray beam used and $\theta$is Bragg's angle and hkl are miller indices of each plane XRD pattern.

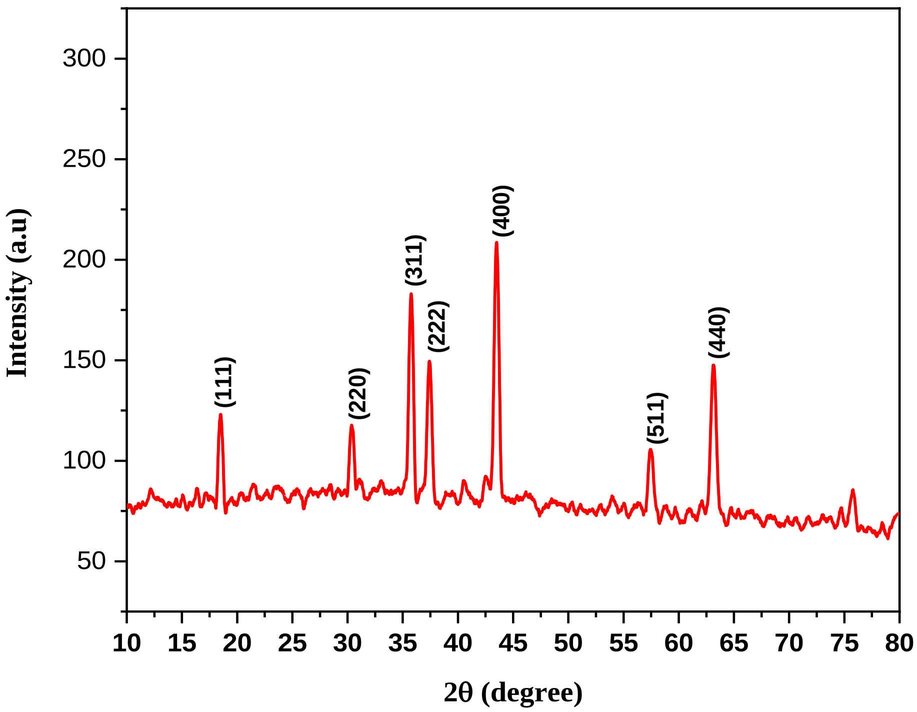

Figure 1: XRD diffraction of $NiMn_2O_4$.

X-Ray density of Synthesized $NiMn_2O_4$ nanoparticles is calculated from following formula.

$$V_{cell} = a^3$$

$$\rho_x = \frac{ZM_w}{V_{cell}\, N_A}$$

Where Z is formula unit present in unit cell, Mw is molecular weight of synthesized nanoparticles and NA is Avogadro's number.

From XRD peaks average crystallite size were calculated by using the Scherrer's formula as follows:

$$D = \frac{k\lambda}{\beta cos\Theta}$$

Where k is Scherer's constant, **λ**is wavelength of X-ray beam and β is full width half maximum of peak and average crystallite size of synthesize nanoparticle are illustrated in Fig 1

3.2. SEM analysis

3.2.1 Scanning Electron Microscope (SEM)

Surface morphology as well as distribution of particle size (using imagej software) of synthesized $NiMn_2O_4$ nanoparticles are given in histogram chart as shown in Figure 3. In present sample chunk, shaped nanoparticles of different diameter were observed. Some of agglomeration also observed in synthesized nanoparticles because of Vander Wall forces and magnetic dipolar couplings between synthesized nanoparticles and total interface energy continues to decreased so no distinguishable morphology appear in Figure 2.

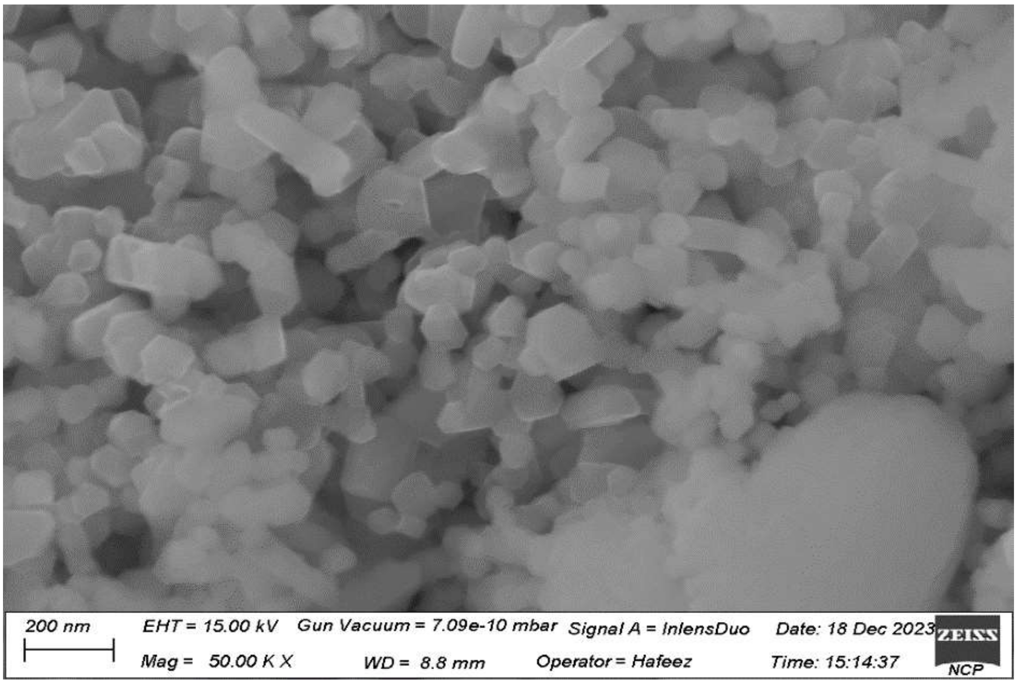

Figure 1: SEM image of $NiMn_2O_4$ nanoparticles

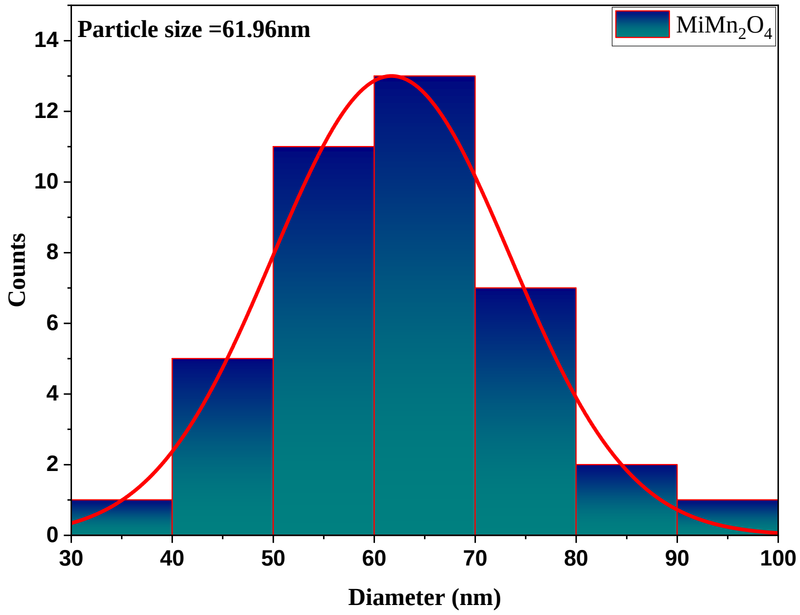

Figure 3: Particle size using imagej software

The average particle size of nanoparticles was created by considering the 40 nanoparticles using the imagej software as shown in the Figure 3. Furthermore, crystallite size calculated from XRD analysis illustrated in the Fig 2 is good agreement with SEM results.

3.3. Energy dispersion Spectrum (EDS):

For elemental composition of synthesized $NiMn_2O_4$ nanoparticles, spectrum of EDS is used. Presence of Ni, Mn, Ca and O were confirmed by using EDS analysis and their percentage as shown in the Figure 4.Presence of $NiMn_2O_4$nanoparticles phase and exact weightage of elements were confirm through EDS analysis. Which means that co-precipitation technique can greatly improve $NiMn_2O_4$ nanoparticles synthesis. Some peaks of Ca were also observed in EDX spectrum because we used carboxymethylcellulose (CMC) during synthesis of samples. To enhance validity of morphology of samples Carbon (C) was spray coated on them before SEM analysis. That is why some minor peaks of C also observed during Energy Dispersion Spectrum (EDS) analysis.

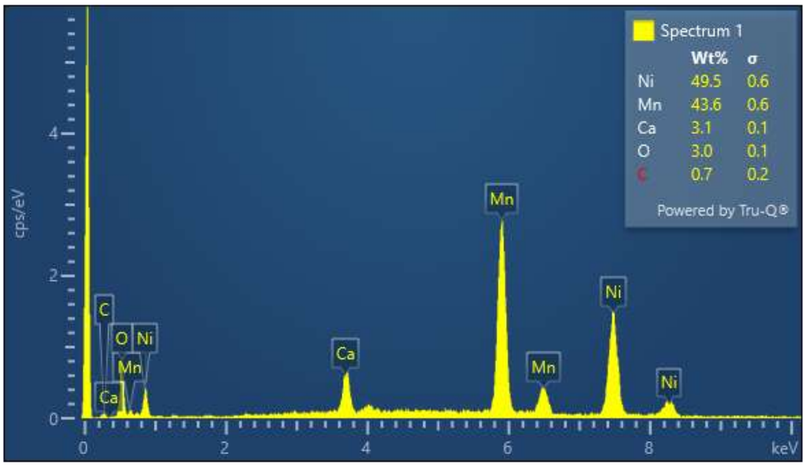

Figure *4*: EDX spectral analysis for elemental distribution of $NiMn_2O_4$ nanoparticles.

4. Conclusion:

A new method of synthesis of nanocrystal line $NiMn_2O_4$ with discrete nanoparticle structures stabilized by carboxymethylcellulose (CMC) capping ligands has been proposed. Irregular geometries of $NiMn_2O_4$ nanoparticles have been synthesized using co-precipitation method a mixture of salts including transition metal chlorides in CMC solutions at 500 °C. XRD results confirmed single-phase cubic structure of present sample and lattice parameter found to be 8.3131Å. XRD results also show good crystallinity with average crystallite size was found 27.18nm.. Morphological analysis were confirmed by SEM and found average particle is 61.96nm and elemental composition were done by EDS analysis.

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