

PREPARATION AND CHARACTERIZATION OF NANO FERRITES

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ABSTRACT:

Nickel ferrite (NiFe₂O₄) and manganese ferrite (MnFe₂O₄) have been prepared by a soft mechano chemical route from mixture of (1) Ni(OH)₂ and α-Fe₂O₃ and (2) Mn(OH)₂ and α-Fe₂O₃ powders in a planetary ball mill. The mixture was activated for varying duration. Soft mechanochemical reaction leading to formation of the NiFe₂O₄ and MnFe₂O₄ spinel phases were followed by X-ray diraction, Raman and infrared spectroscopy, scanning and transmission microscopy. The spinel phase formation was rst observed after 4 h of milling (case 1) and after 3 h (case 2) and its formation was completed after 25 h in both cases. The synthesized NiFe₂O₄ and MnFe₂O₄ ferrites have a nanocrystalline structure with a crystallite size of about 30 and 40 nm for cases (1) and (2), respectively. There are ve Raman and four IR active modes. In order to understand better the whole process of phase formation, the Mössbauer measurements were done.

Keywords: *HPLC, PDA, stability indication method, drug.*

1. INTRODUCTION:

Magnetic nanocrystals and nanosized ferrites have been attracting extensive attention due to their wide applications, such as magnetic memory [1], MRI contrast agents [2], e-cient hyperthermia for cancer therapy [3, 4] and catalysts [5], etc. Among these magnetic materials, spinel-type ferrite nanoparticles, MFe₂O₄ (M = Mn, Co, Ni, Zn, Mg, Fe, etc.), have gained great interest in the past few years because of their good electromagnetic performance and their applications in information storage, medical diagnosis technology, sensor technology, and magnetic warming and cooling technology [6]. The structural formula of spinel-type ferrites can be written as (M₂+ 1-λFe₃+ λ) [M₂+ λ Fe₃+ 2-λ] O₄, where parentheses and square brackets denote cation sites of tetrahedral (A) and octahedral

(B) coordination, respectively. λ, which is determined by the preparation process, represents the so- -called degree of inversion dened as the fraction of the (A) sites occupied by Fe³⁺ cations [7]. NiFe₂O₄ and MnFe₂O₄ ferrites have been synthesized by various methods such as solid-state reaction, co-precipitation, high-temperature self-propagating, microemulsion, solvothermal, mechanochemical synthesis, hydrothermal, solgel and combustion techniques [815]. In this paper, we present the formation of spinel ferrite phase through soft mechanochemical treatment [16], starting from dierent mixtures of powders. The cases were: (1) milling of powders mixture of Ni(OH)₂ + α-Fe₂O₃ during 425 h and (2) milling of mixture of Mn(OH)₂ + α-Fe₂O₃ during 325 h. The Raman and IR spectroscopy in addition to the X-ray diraction, scanning electron microscopy, transmission electron

microscopy and Mössbauer spectroscopy measurements were used to study the samples mechanochemically treated for different milling times.

2. RELATED STUDY:

Nowadays, the material called as Nanosized ferrite has gained more interest in the recent decades. It displays an abnormal chemical as well as physical properties termed to be dissimilar to those of the aggregate materials due to their acutely small measurement and ample specific apparent surface area. Normally, the features of ferrites are said to be non-conductive ferri magnetic bowl compounds acquired from the oxides of iron like hematite (Fe_2O_3) or magnetite (Fe_3O_4) and also the added metal oxides, and are like the best added ceramics, tough and fragile [8]. In conformity with the alluring properties, the diverse ferrites are generally classified as either "elastic" or "rigid", which refers to their low or aerial alluring coercivity. The inorganic compound Zinc oxide is denoted by its formula ZnO . This is impenetrable in water and generally has the appearance of white powder. The powder is broadly acclimated as an accretion into the numerous products and materials involving rubber, glass, ceramic materials and lubricants. Zinc oxide is a vital and imperative material because of its cost effectiveness and ample band gap (3.37 eV), luminescent features and ample excitation binding activity (60 MeV). It is broadly employed in abounding applications like gas sensor, catalyst, filtering properties for UV light, and additionally as retanning and antimicrobial abettor. The black material

cupric oxide (CuO) has the melting point above 1200°C , which includes oxygen loss and is utilized as a colorant in adobe glazes. Numerous colors such as blue, red, and green can be acquired from it. As a mineral, it is accepted as tenorite which has the features of organic solvents, water dissolvent and has the color of red. Copper (II) oxide has appliance as a p-type semiconductor [5]. It is an adapted aggravate to optical equipment brightness for generating dry battery cells as cathode. Cupric oxide (CuO) is an imperative metal oxide transition along with the narrow band gap, and forms the base of several absorbing aerial temperature superconductors and huge magneto resistant materials. Either Iron (III) oxide or Ferric oxide is an inorganic admixture with the chemical formula Fe_2O_3 . It is one of the three imperative iron oxides and the rest is two iron (II) oxide (FeO), which is exceptional and iron (II, III) oxide (Fe_3O_4), which additionally occurs as the magnetite mineral. The mineral accepted as hematite, Fe_2O_3 , is the center antecedent of iron for the purpose of steel industry. The formula Fe_2O_3 has dark reappearance and is voluntarily assailed by acids [2]. The term rust is generally iron (III) oxide, and to some measurement this characterization is functional, because rust shares several luminescent properties with the related composition. The rust is treated as the hydrated ferric oxide and an ill-defined material by chemists. The utilization of the high purity raw materials helps in the preparation of the ferrite sample, because the existence of impurities present in the raw materials may have a great influence on the

prepared material properties. Asuitable percentage of the weight of the oxide is required for mixing the diverse composition. The nano powders of ZnO, CuO and Fe₂O₃ are taken and they are mixed in an equal proportion to attain the uniformity among the components [1]. This nano powder preparation can be executed by the sol-gel method, which is explained in the next section.

3. PROPOSED METHODOLOGY:

The solution was constantly stimulated by utilizing the magnetic agitator by keeping it at the 50 °C in this technique. This combined solution was transferred to a vessel and it was allowed to be heated to 100 °C slowly and stimulated constantly till it remained glutinous, and the color change happened when the solution became a green absorbent dry gel. This type of dry gel was allowed to burn to become a loose powder [7]. A similar technique is involved in preparing copper zinc ferrite by accumulating the hexa hydrated zinc nitrate. This type of powder preparation can be calcined and maintained at various temperatures i.e. 800 °C and 600 °C for about 5 hrs. The samples can be prepared, characterized and synthesized by various techniques like EDAX, XRD, FTIR and SEM.

The same microscope was used to collect the backscattered radiation. The scattering light dispersed was detected by a charge-coupled device (CCD) detection system. Room temperature Raman spectra are in spectral range from 100 to 800 cm⁻¹. The average

power density on the sample was 20 mW mm⁻². • The infrared (IR) measurements were carried out with a BOMEM DA-8 FIR spectrometer. A DTGS pyroelectric detector was used to cover the wave number range from 50 to 700 cm⁻¹. • The morphology of powders and the size of soft mechano-synthesized ferrite crystallites were examined by scanning electron microscopy (SEM, Model TESCAN Vega TS130MM) and transmission electron microscopy (200 kV TEM, Model JEM-2100 UHR, Jeol Inc., Tokyo, Japan). • The Mössbauer spectra of powder samples were measured at room temperature (RT) using a source of ⁵⁷Co in Rh (1.85 GBq). The experiments were performed in standard transmission geometry with constant acceleration calibrated by the laser spectrum. The data were analyzed by The WinNormos Site program [17]. Sample thickness correction was carried out by transmission integral.

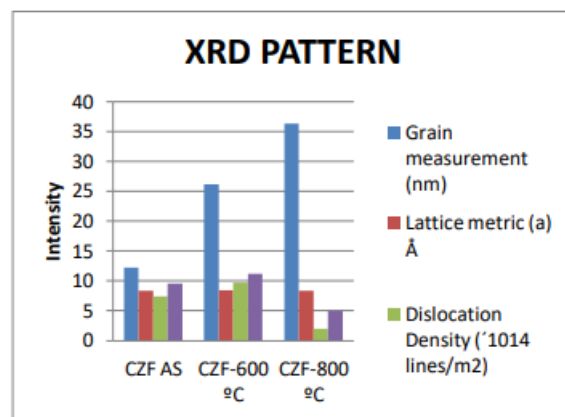


Fig.3.1. Graphical representation.

5. CONCLUSION:

In this paper, we show that it is possible to obtain NiFe₂O₄ and MnFe₂O₄ ferrites by

soft mechanochemical synthesis starting from the mixture of (1) Ni(OH)₂ and α-Fe₂O₃ and (2) Mn(OH)₂ and α-Fe₂O₃ powders. It has been shown that mechanochemical treatment of mixtures with starting materials leads to the amorphization of the starting powders, forming the phase of Ni(OH)₂, NiO, NiO(OH), FeO(OH) and Fe₂O₃ after 4 h and 10 h of milling, and only NiFe₂O₄ phase after 25 h, (case 1). On the other hand, observing case (2), Mn(OH)₂ and Fe₂O₃ phases occur after 3 h of milling, Fe₂O₃, MnO and FeO(OH) phases after 12 h and finally after 25 h only MnFe₂O₄ phase. On the basis of the Raman research there are observed first-order Raman active modes. The intensity of the Raman and IR modes in the formation of NiFe₂O₄ and MnFe₂O₄ ferrite phases is rather low, as it is expected for nanocrystalline samples. The surface morphology of the samples consists of the grains with relatively homogeneous distribution as seen from SEM. Crystallite size estimated from Scherrer's formula is consistent with the results of TEM. The Mössbauer spectra show the one doublet and the singlet, originating from octahedral and tetrahedral coordination, respectively. The cation site preference is affected when the size of particles are small, too. We found out that amount of Mn²⁺ at tetrahedral site decreases as size of particles decreased.

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