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Examination Of Structral Morphological and Magnetic Behaviour of $Znfe_2o_4$ Nanoparticle Synthesised by Co-Pricipitation Method

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Abstract*— The Zinc Ferrites ($ZnFe_2O_4$) nanoparticles (NPs) successfully synthesized by co-precipitation method. As prepared NPs were annealed withdifferent temperature at 800 and 1000 ℃. The crystalline phase, surface morphology andelemental distribution were confirmed by XRD, SEM and EDS respectively. The prepared $ZnFe_2O_4$XRD pattern confirm the cubic crystalline nature. The surface morphology of the prepared sample was found irregular rock like structure due to ferric metal. FTIR, EDS studies prove charactristics behavioural vibration of Zn-Fe-O elements and purity of the sample correspondingly. Prepared $ZnFe_2O_4$ sample exhibit a super paramagnetic nature with the saturated magnetization of 22.066 emu/g and coercivity of 15 Oe by VSM analysis.

Keywords: Zinc-ferrite NPs, Co-precipitation, High-density magnetic storage media, Super paramagnetic.

I. INTRODUCTION

Nanoparticles intense from current interest in basic scientific studies to miniaturization of devices, as well as verity of applications in energy storage devices [1-2] magnetic storage media [3], medicine [4-6] and bio-sensing and sensing [7-11]. Depending upon the physical properties differ intensely from their bulk complements rising behind their small size. In a particular manner, magnetic nanoparticles (MNPs) from their smaller size, non-toxicity, inject ability, biocompatibility and high leveling in biomedical applications [12, 13]. Transition metal oxides and metal sulfides are extensively studied due to their verity of potential applications [14-17]. Among them, the binary metal oxide shows the peculiar properties due to the combination of two elements and doping effect, by this way the binary metal oxides having applications in multidisciplinary area [18]. Recently, Binary magnetic oxides materials are becoming trendy materials for multifunctional applications in different

Along with various materials, particularly spinel-ferrite structured (XFe_2O_4, X=Zn, Ni, Co, Bi, etc.) materials are admired magnetic materials for different applications due to peculiar properties such as, electric generators, inductor coils, transformer, recorder, nano fluids and bio-medical applications. Moreover, spinel- ferrite structures are more feasible with cost-effectively [18]. Zinc ferrite is an essential material for widely used magnetic materials and these spinel-ferrite structured materials have good photocatalytic properties. In addition, the catalyst can be easily isolate by applying magnetic field for catalyst reusability [19]. Zinc ferrite structured materials suggested an enhanced displaying with attractive photo sensitivity in narrow band width of ~1.9 eV for photocatalytic applications [20].

Here, we employed zinc ferrite ($ZnFe_2O_4$) NPs by co-precipitation method and confirmed with different studies like structural, morphological and magnetic behaviors. The prepared $ZnFe_2O_4$ sample exhibits a super paramagnetic nature.

II. MATERIAL SYNTHESIS

A. Chemicals

All reagents were used with A.R grade was used. Zinc nitrate ($Zn(NO_3)_3•6H_2O$), Iron nitrate ($Fe(NO_3)_3$ and Sodium hydroxide (NaOH) were purchased from sigma Aldrich. The solvents ethanol and De-ionized water were performed for entire reaction.

B. Synthesis method

In this typical synthesis co-precipitation method was adapted for Zinc ferrite NPs synthesis. Equal molar ratio of ($Zn(NO_3)_3•6H_2O$), as precursors and NaOH as alkaline medium. The process in co-precipitation reaction involves three major steps, they are, nucleation, growth, coarsening and agglomeration process. When the precipitation process starts, the nucleation occurs simultaneously. After that they form an aggregation which makes the particles to become quite higher size nanoparticles and thermodynamically stable. This is followed by the coarsening in which formed particles grow further to produce nanoparticles.

The ferrite nanoparticles were formed by co-precipitation and ferritisation processes. The base medium converts the metal salts into hydroxides. On heating the formed hydroxides, the nanoparticles get transformed into ferrite but it takes some time in co-precipitation method. Hence the precipitate was collected and washed extensively with ethanol dried in hot oven for 7 h.

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C. Characterization Technique

The prepared nanoferrite samples were characterized with Powder XRD by Rigaku Miniflex II X-ray Diffractometer. The XRD patterns were recorded with CuKα monochromatic radiation (1.5406 Å). The morphology and elemental analysis were analyzed using the FEI-QUANTA–FEG 250 (SEM) equipped with EDX analyzer. The Shimadzu IR Trace-100 spectrometer was employed to find functional groups presence in zinc ferrite NPs with the help of KBr pellet system. The magnetic nature of prepared $ZnFe_2O_4$ NPs was carried out in a vibrating sample magnetometer (VSM) Lake Shore is having an electromagnet capable of delivering fields up to 1.5 Tesla. The magnetometer is able to detect magnetic moments down to 10^{-6} emu, at room temperature.

III. RESULTS AND DISCUSSION

A. Powder XRD Analysis

The phase composition of phases and crystal structure of samples was analyzed by powder X-ray diffraction. The Fig. 1 shows, as prepared sample exhibited amorphous nature. Further, the sample was calcined with different temperature at 800 ºC and 1000 ºC. After calcination clear broad peaks was appeared and all the diffraction peaks are closely coincides with cubic structured zinc ferrite crystalline structure (JCPDS card No. 74-2397) in this 2θ range 29.9, 35.26, 36.84, 42.8, 53.09, 56.59 and 62.1 with corresponding hkl planes (220), (311), (222), (400), (422), (511) and (440) respectively.

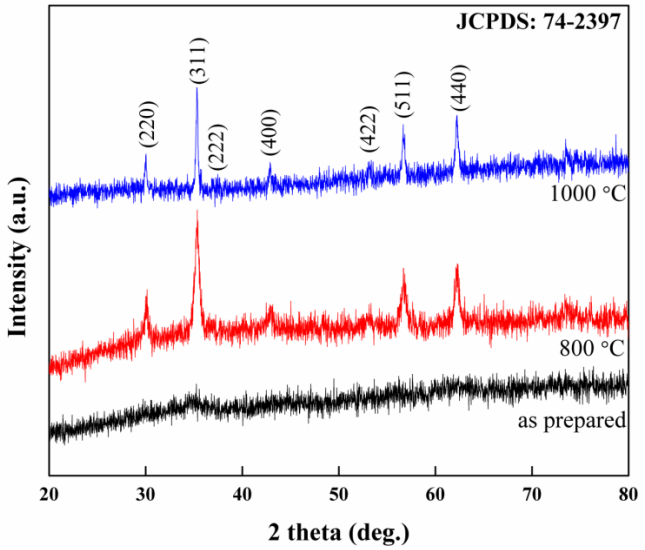

Fig. 1. XRD Pattern Of (a) $ZnFe_2O_4$,calcined $ZnFe_2O_4$ at (b) 800 °C and 1000 °Cnanoparticles.

This all crystalline peaks which were confirmed the formation of zinc ferrite NPs. The XRD pattern of zinc ferrite nanoparticles calcined at 800 ºC shows the emergence of few peaks of zinc ferrite indicating the origin of crystalline phase. This can also be done throughexamining the XRD pattern of the prepred nanopaticles calcined at 1000 ºC. Further increase in calcination temperature to 1000 ºC increased the sharpness of the peaks [15,18,21]. The sharp peaks good indicated the size increament and crystallinity.

B. *FTIR analysis*

The formation of spinel $ZnFe_2O_4$ structure in the pure Fig. 2.(a) and calcined zinc ferrite nanoparticles Fig. 2.(b-c) was further confirmed by FT-IR spectrum. The FTIR spectrum of zinc ferrite samples is shown in Fig. 2.The peaks at 550 cm^{-1} and 680 $cm^{-1}$was due to the stretching vibration of metal-oxygen (Fe-O) bond interaction and it also shows the formation of spinel structure in zinc ferrite compound. The spectra showed a strong band at 433 cm^{-1} and 1082 cm^{-1} which is a characteristic of Zn-O vibrational band. Thestretching vibration between Zn-O-Fe bondswere ascribed in two bands at 1355 cm^{-1} and 860 cm^{-1}The broad absorption at 3400-3600 cm^{-1} corresponds to the O-H stretching vibrations [15, 18].

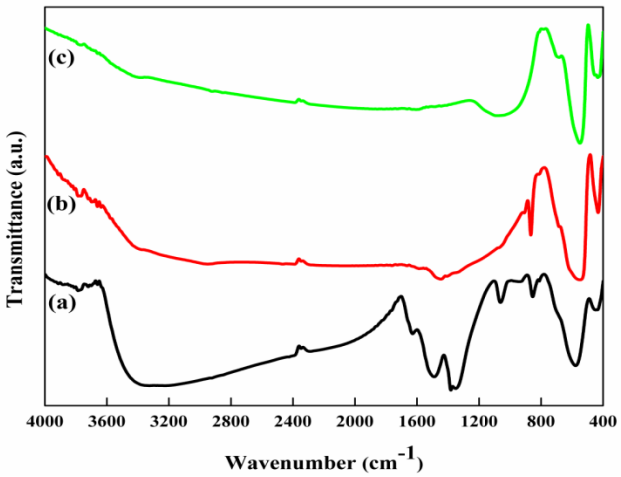

Fig. 2. FTIR spectra of (a) as prepared (b) calcined at 800 °C and (c) calcined at 1000 °C $ZnFe_2O_4$ nanoparticles.

C. Surface and Elemental analysis

Fig. 3. reveals the morphology of the zinc ferrite nanoparticles calcined at different temperature. The SEM images of $ZnFe_2O_4$ sample reveals irregular shape particles with different magnifications all the particles are appeared rock like structures due to Fe ions. No major particle size was obtained in pure and calcined samples. The surface morphology of all the samples are observed from SEM analysis 20 nm to few micro meter ranges due to agglomeration and irregular shape.

An EDX spectrum reviles the purity and elemental composition of the $ZnFe_2O_4$ NPs. Fig 3. (b), (d) and (f) shows, the EDX pattern of synthesized $ZnFe_2O_4$ samples and the heat-treated samples. Elemental concentration obtained all three samples is given in Table. I. EDX confirmed the formation and purity of the presence chemical constituents such as oxygen (O), zinc (Zn), and iron (Fe) elements [18].

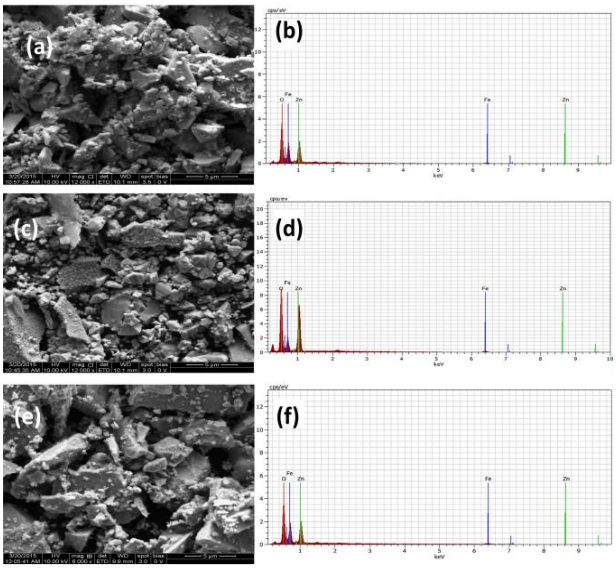

Fig. 3. (a, c, e) SEMimages of as prepared ZnFe2O4 NPs with different calcination temp. 800 and 1000 ºC and (b, d, f) EDX spectra of corresponding samples.

The fig 3(c) shows that the elemental composition of the prepared composite. The carbon contains the peak at the Kα radiation characteristic x-ray emit at the range of 0.277keV and the intensity was high. It shows the moreover the percentage of carbon was very high from the EDAX graph 38.1%. The Oxygen exhibits the range of radiation characteristic x-ray Kα 0.525keV and the oxygen contain 33.5%. Finally, the Ni peak contains in the range of 7.47keV in 28.4%.

TABLE I. EELEMENTAL ANALYSIS OF ZINC FERRITE NANOPARTICLES

Temperature	As prepared		800◦C		1000◦C	
Element	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
O	40.80	69.72	38.43	70.20	42.02	73.21
Zn	30.40	13.61	31.69	14.17	29.45	12.55
Fe	28.80	16.67	29.88	15.64	28.53	14.24
Total	100.0	100.0	100.0	100.0	100.0	100.0

D. VSM Analysis

The magnetic properties of the calcined at 1000ºC $ZnFe_2O_4$ NPs was determined by VSM. Fig. 4 show the magnetic field dependence of magnetization (M-H) of $ZnFe_2O_4$ NPs measured at ambient temperature (300 K) with an expanded scale of the low field in the insets. The saturated magnetization and coercivity of the sample are tabulated in Table. II.From the Hysteresis loop $ZnFe_2O_4$ sample was exhibit a superparamagnetic nature with the saturated magnetization of 22.066 emu/g and coercivity of 15 Oe, respectively [18]. The sample gets magnetized with the applied field and when the field is increased the magnetization also increased and vice–versa. Their coercive fields (Hc) are almost negligible which indicate the sample exhibits the super paramagnetic behavior and it is clearly signifying the insert image. The biocompatible magnetic nanoparticles may play an important role in biomedical applications.

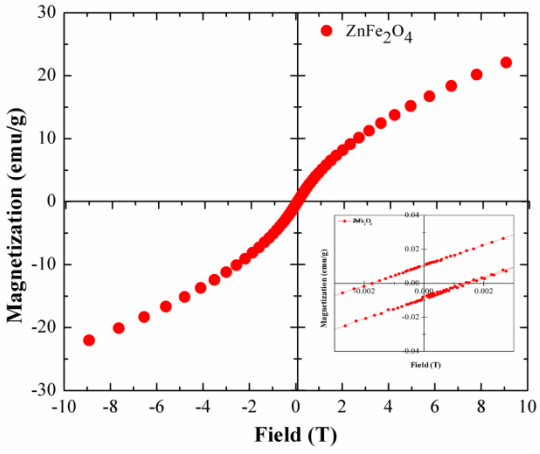

Fig. 4. Magnetic Hysteresis Loops of $ZnFe_2O_4$ nanoparticles calcined at 1000 ºC

TABLE II. MAGNETIC PROPERTIES OF ZINC FERRITE NANOPARTICLES CALCINED AT 1000ºC.

Samples	Coercivity (T)	Saturation magnetization (emu/g)
$ZnFe_2O_4$	0.00150	22.066

IV. CONCLUSION

The Zinc Ferrite NPs have been successfully prepared via co-precipitation technique. The base medium was used for the reaction with the help of aqueous sodium hydroxide. The as prepared $ZnFe_2O_4$ NPs were annealed at two different annealing temperatures 800°C and 1000°C. The crystalline phases, functional groups and surface morphology of the samples were evaluated using XRD, FTIR, SEM and EDX respectively. XRD spectra confirmed the formation of zinc ferrite nanoparticles. On maximizing the annealing temperature, the diffracted peaks became sharper and decreased intensity and peak width. It clearly proves that, increase the crystallinity at high temperature. The FTIR spectra showed a strong band at 433$cm^{-1}$and 1082cm^{-1} which is characteristic of Zn-O vibrational band. From the SEM analysis, the surface morphology consists of irregular shapes and the particles size was found 20 nm to few micrometer range. The existence of elements and their percentage was analyzed by EDX which confirmed the presence of chemical constituents such as oxygen (O), zinc (Zn) and Iron (Fe) elements. VSM analysis of $ZnFe_2O_4$ nanoparticles have confirmed the superparamagnetic nature at room temperature with a saturation magnetization of 22 emu/g respectively with coercive forces 0.0015 T. $ZnFe_2O_4$ nanoparticles can be considered as potential materials for various applications. Thus developed method can be a potential method to obtain biocompatible $ZnFe_2O_4$ nanoparticles for wide spectrum of applications particularly in biomedical field.

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