

Investigation of Structural, Morphological, Magnetic Properties and Biomedical applications of Cu^{2+} Substituted Uncoated Cobalt Ferrite Nanoparticles

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ABSTRACT

In the present work, Cu^{2+} substituted cobalt ferrite ($\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$, $x = 0, 0.3, 0.5, 0.7$ and 1) magnetic nanopowders were synthesized via chemical co-precipitation method. The prepared powders were investigated by various characterization methods such as X-ray diffraction analysis (XRD), scanning electron microscope analysis (SEM), vibrating sample magnetometer analysis (VSM) and fourier transform infrared spectroscopy analysis (FTIR). The XRD analysis reveals that the synthesized nanopowders possess single phase centred cubic spinel structure. The average crystallite size of the particles ranging from 27-49 nm was calculated by using Debye-scherrer formula. Magnetic properties of the synthesized magnetic nanoparticles are studied by using VSM. The VSM results shows the magnetic properties such as coercivity, magnetic retentivity decreases with increase in copper substitution whereas the saturation magnetization shows increment and decrement in accordance with Cu^{2+} substitution in cobalt ferrite nanoparticles. SEM analysis reveals the morphology of synthesized magnetic nanoparticles. FTIR spectra of Cu^{2+} substituted cobalt ferrite magnetic nanoparticles were recorded in the frequency range $4000\text{-}400\text{cm}^{-1}$. The spectrum shows the presence of water adsorption and metal oxygen bonds. The adhesion nature of Cu^{2+} substituted cobalt ferrite magnetic nanoparticles with bacteria in reviewed results indicates that the synthesized nanoparticles could be used in biotechnology and biomedical applications.

Key words: Magnetic nanoparticles, Co-precipitation, Cubic spinel, Morphology, Anti-bacterial activity.

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INTRODUCTION

Great scientific interest has been laid on synthesis of nanoparticles and analyzing its properties for their plenty of applications in past two decades. Specifically it seeks wide attention among scientists because, they act as a bridge between bulk materials and molecules and atomic structures. Among the known nanomaterials, magnetic nanomaterials play a vital role in various fields. Magnetic nanoparticles are present inside us and everywhere around us. It prevails in migratory birds, animals, meteorites and in interstellar space ^[1]. Magnetic nanoparticles has been paid much attention due to their various usages in targeted drug delivery, ferrofluids, gas sensing devices, medical diagnostics, genetic screening, etc., Magnetic nanoparticles are distinct when compared with their bulk counterparts due to their size, shape and surface chemistry. The important parameters which explain the properties of a magnetic material such as coercivity, retentivity and saturation magnetization are functions of the particle size and shape. In the group of magnetic nanoparticles, spinel ferrites have been analyzed widely because of its peculiar magnetic properties. Among them, Fe_3O_4 has cubic inverse spinel structure which possesses various types of magnetic properties ^[2]. In Fe_3O_4 cubic inverse spinel structure, substitution of transition metals such as Co, Ni, Cu, Cr, Mn etc., results in magnetic multilayers. These magnetic multilayer compositions are the interesting topics in material science due to their technological and scientific applications ^[3]. Multilayer films possess fascinating applications in magneto-resistive sensors and biomedical applications ^[4, 5]. Nanosized multilayer films of magnetite possessing transition metals can be synthesized by various methods such as co-precipitation method, hydrothermal method, ball milling, organo-metallic compounds decomposition and microemulsion technique. In multilayer film synthesis, generally magnetite and maghemite metal oxides are used due to their high saturation magnetization and biocompatibility ^[6]. During magnetic nanoparticle preparation processes, the parameters such as particle grain size, chemical ratio of additives, sintering temperature, doping ratio and preparation method play vital role in tuning the properties of spinel ferrites which can be used for different applications. Among transition metal substituted compositions, CoFe_2O_4 is a hard magnetic material which has considerable attention of technical community due to its peculiar enhanced magnetic properties such as high saturation magnetization, high density audio and video recording media, for controlled drug delivery, radio frequency hyperthermia, magnetic resonance imaging and medical diagnostics ^[7, 8]. The transition metal substitutions in CoFe_2O_4 such as Mn, Cu, Zn, Ni, etc., play an important role in enhancing physical properties like magnetic and electrical properties of spinel ferrites ^[9]. In this paper, an effort has been made to reveal the structural, morphological and magnetic properties of Cu^{2+} substituted cobalt ferrite nanopowders which could be used in different fields due to their peculiar magnetic and electrical properties. An experimental work has been carried out in substituting Cu^{2+} in cobalt ferrite nanoparticles. Among the different synthesis process, co-precipitation is the simplest method which provides homogenous nature to the magnetic nanoparticles without addition of any other chemical impurities. Hence the complete work has been carried out in co-precipitation method for analysis and the results are reported. We report the size, structure, morphology and changes in magnetic properties of the synthesized nanopowders. The properties has been analyzed in detail and reported. The possible usages of the prepared nanopowders specifically in biomedical field and also in different fields with respect to their peculiar properties are also discussed in detail.

MATERIALS AND METHODS

Materials

Analytical grade chemicals cobalt chloride (CoCl₂·6H₂O), copper chloride (CuCl₂·4H₂O), ferric chloride (FeCl₃·6H₂O) and sodium hydroxide pellets (NaOH) were purchased from Merck, Co. They were purchased with 99% purity. Hence, there was no need for further purification process.

Magnetic nanoparticle synthesis

Various stoichiometric mixtures of Co_{1-x}Cu_xFe₂O₄ (x = 0, 0.3, 0.5, 0.7 and 1) nanoparticles have been synthesized by chemical co-precipitation method. In magnetic nanoparticles synthesis process, required amounts of CoCl₂·6H₂O, CuCl₂·4H₂O and FeCl₃·6H₂O salts in 1:2 molar ratio were dissolved in 100ml of de-ionised water separately. The prepared aqueous solutions were dissolved in 1000ml of boiling NaOH (0.5M) solution whose temperature was maintained at 70°C. The mixed solution was subjected to constant magnetic stirring for one hour at 100°C. The one hour duration was quiet enough for the conversion of metal hydroxides into spinel ferrites. After one hour consistent magnetic stirring, the solution was left undisturbed for the next four hours to obtain residue. The residual solution was decanted and repeatedly washed with deionised water to obtain pH in between 7 – 8. Finally the residue was washed with acetone to eliminate the impurities and dried at room temperature. Generally in order to obtain magnetic nanopowders with crystalline nature, the dried samples were subjected to sintering process at different temperature with respect to the stoichiometry of chemical composition. In this synthesis process, an effort was made to obtain crystalline structure using hot plate without sintering process. The dried samples were subjected to consistent heating for one hour duration over hot plate and subjected to cooling. The obtained loose powder samples were grinded and subjected to different characterization methods^[10, 11].

Characterization

X-Ray diffraction

X-ray diffraction analyses have been carried out by using XPERT PRO X-ray powder diffractometer Cu K α ($\lambda = 1.54060 \text{ \AA}$) radiation at normal room temperature. The XRD measurement was made with step size (2 θ) 0.05° and scan step time of 10s. The step size (2 θ) 0.05° and scan step time of 10s, was followed for XRD measurement. The average crystallite size of the samples was calculated using Debye- Scherrer formula^[12].

$$D_{\text{xrd}} = \frac{0.89\lambda}{\beta \cos \theta} \quad (1)$$

Here, λ represents the wavelength of X-ray used in \AA , β – is the full width half maximum (FWHM in radians in the 2θ scale), θ represents the Bragg's diffraction angle, D_{XRD} - crystallite size in nm.

Vibrating sample magnetometer

Magnetic studies for the obtained magnetic nanoparticles were carried out by using vibrating sample magnetometer. The magnetic properties measurement parameters such as coercivity (H_c), remanant magnetization (M_r), saturation magnetization (M_s) and hysteresis were obtained using vibrating sample magnetometer (VSM) at room temperature (Model: Lakeshore 7404) with an applied magnetic field of 20000G.

SEM Studies

The observations using scanning electron microscope (SEM) was carried out to visualize the morphology, existence of homogenous nature and nature of chemical composition of the prepared magnetic nanoparticles.

FTIR Analysis

FTIR spectra for the obtained magnetic nanoparticles $\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ (with $x=0, 0.3, 0.5, 0.7, 1$) was obtained using Lambda 35 spectrometer range $4000\text{-}400\text{cm}^{-1}$ with scan speed of $960\text{nm}/\text{min}$ and 1.0nm data interval. The spectra were measured using transmittance method.

RESULTS AND DISCUSSION

X-ray diffraction analysis was used to identify the structure and size of the particles. Figure.1A, 1B and 1C shows the XRD patterns of $\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ ($x=0, 0.3$ and 0.5) magnetic nanoparticles. By considering the nanoparticles of the above samples as spherical in shape, the crystallite size of the particles was determined by using Debye-scherrer's equation ^[12]. The average crystallite size of the prepared particles ranging from 27 nm to 49 nm . The sharp reflection peaks obtained for the planes at (220) (311) (400) (511) (440) indicates that the synthesized particles are with single phase cubic spinel in structure. The corresponding reflection angles for the maximum intensity of the plane (311) were at $35.45^\circ, 35.53^\circ, 35.62^\circ$ ^[13]. The peak shift can be seen from the corresponding reflection angles, it was due to increase in Cu^{2+} substitution in CoFe_2O_4 nanoparticles. The lattice constant (A^0) starts increases with increasing copper substitution ^[7, 14]. The increase in lattice constant with copper substitution was may be due to the occupying nature of copper ions in positions of Fe^{3+} ions. The copper ions ionic radii are about 0.70A^0 which is greater than the ionic radii of Fe^{3+} ions which is only about 0.67A^0 ^[7, 15]. The above mentioned occupying phenomena of Cu^{2+} results in peak shift.

Vibrating sample magnetometer (VSM) was used to determine the magnetic properties coercivity (H_c), retentivity (M_r), saturation magnetization (M_s) etc., of the obtained $\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ magnetic nanoparticles. The magnetization of the synthesized magnetic nanoparticles with respect to the applied magnetic field was obtained by using VSM studies and it was shown in Figure 2. The variation in magnetic properties was shown in Table 1.

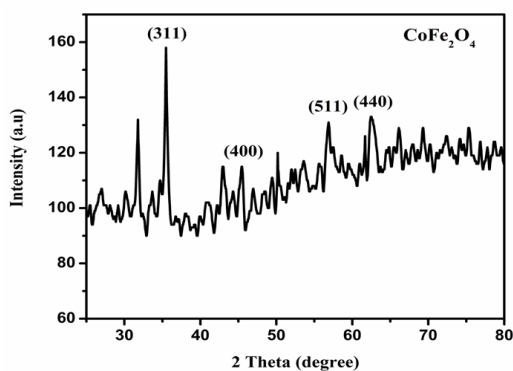
Cu²⁺ Substituted Cobalt Ferrite Nanoparticles

Fig. 1 A

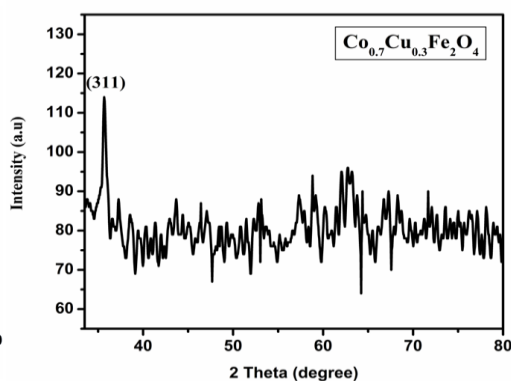


Fig. 1 B

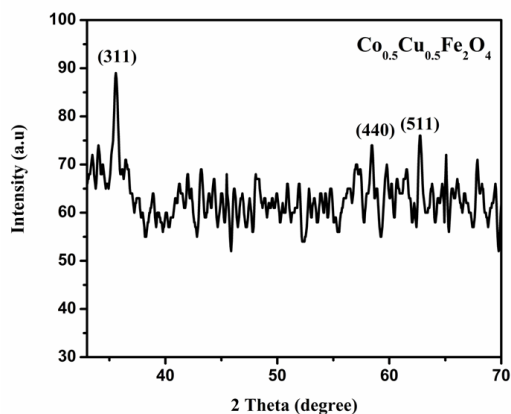


Fig. 1 C

Figure 1A, 1B and 1C. X-Ray diffraction patterns of Cu²⁺ substituted CoFe₂O₄ Magnetic nanoparticles (Co_{1-x}Cu_xFe₂O₄, With x=0.0, 0.3 and 0.5)

Table 1. Magnetic Properties of Cu²⁺ substituted CoFe₂O₄ (Co_{1-x}Cu_xFe₂O₄ With x varying from 0.0, 0.3, 0.5, 0.7 & 1.0) nanoparticles

Samples	Magnetic parameters		
	H _c (G)	Mr (emu)	Ms (emu)
CoFe ₂ O ₄	787.29	0.697	1.6248
Co _{0.7} Cu _{0.3} Fe ₂ O ₄	477.22	0.23044	1.0150
Co _{0.5} Cu _{0.5} Fe ₂ O ₄	391.63	0.19438	0.6326
CuFe ₂ O ₄	34.679	0.0424	0.0578

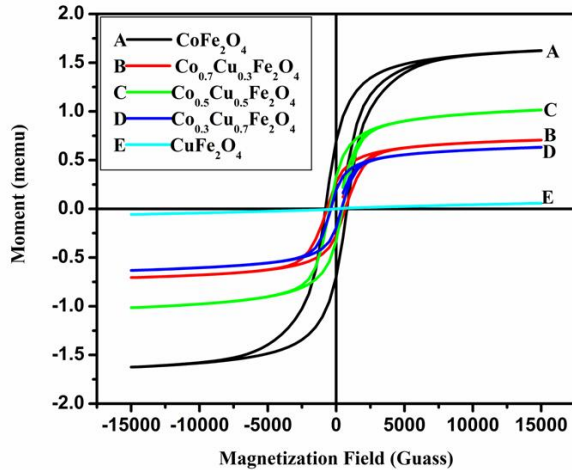


Figure. 2 Magnetisation curves of Cu^{2+} substituted CoFe_2O_4 at room Temperature ($\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$, with $x=0.0, 0.3, 0.5, 0.7$ and 1)

It was shown that the saturation magnetization decrease and increase with Cu^{2+} substitution in CoFe_2O_4 nanoparticles^[16]. The other magnetic properties coercivity and retentivity decrease due to the substitution of non magnetic Cu^{2+} ion in cobalt ferrite chemical composition. The decrement and increment in saturation magnetization can be explained on basis of Neel's theory and super exchange interaction mechanism. The decrement in magnetic parameters was due to the sublattice exchange interaction mechanism takes place among the substituted ions and the existing ions. The electron spins at A and B lattice positions are antiparallel to each other whereas within A and B lattice positions, they are parallel to each other. The total magnetic moment at A and B lattice positions nullify each other and the total magnetization is only due to B lattice position. Thus, B position possesses greater moment than A lattice position. Here Cu^{2+} is a non-magnetic ion replaces the Fe^{3+} ions from tetrahedral positions, the total unpaired electrons at B position increased and are the reason for the increase in saturation magnetization. The reduction in saturation magnetization may be due to existence of large non-magnetic Cu^{2+} ions in B positions which reduces the interaction between A and B positions^[7, 17]. The super exchange interaction mechanism between the sub lattices (A-B) and intra sub lattices (A-A) and (B-B) takes place in the chemical compositions. The preferential occupancy of substituted Copper ion will be in A sub lattice than B sub lattice. Hence the Co ion in A sub lattice will be gradually shifted which results in weakening of B-B interaction. Thus, the saturation magnetization was found to be decrease with Copper ion substitution in Cobalt ferrite nanoparticles^[7, 18].

The observations using scanning electron microscope shows the size, morphology and structural composition of synthesized magnetic nanoparticles. The SEM photographs are shown in Figure. 3A and 3B. From the obtained results it was clear that the synthesized particles are slightly agglomerated in nature due to non addition of surfactant during magnetic nanoparticles synthesis process^[19].

The FTIR spectra of $\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ ($x=0, 0.3, 0.5, 0.7$ and 1) observed in between the frequency range $400\text{-}4000\text{cm}^{-1}$ was shown in Figure 4. The transmittance frequencies observed for the FTIR spectra of $\text{Co}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ are summarized in Table 2.

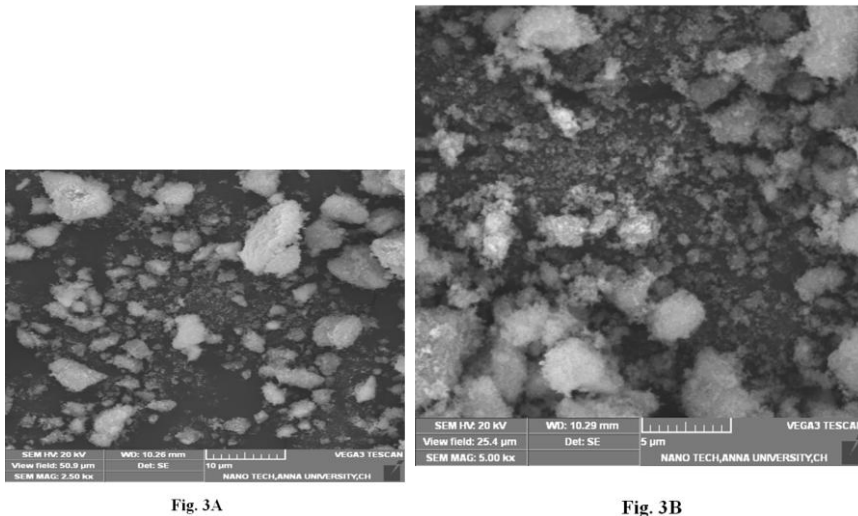


Figure. 3A and 3B SEM Images of Cu²⁺ substituted CoFe₂O₄ Magnetic Nanoparticles

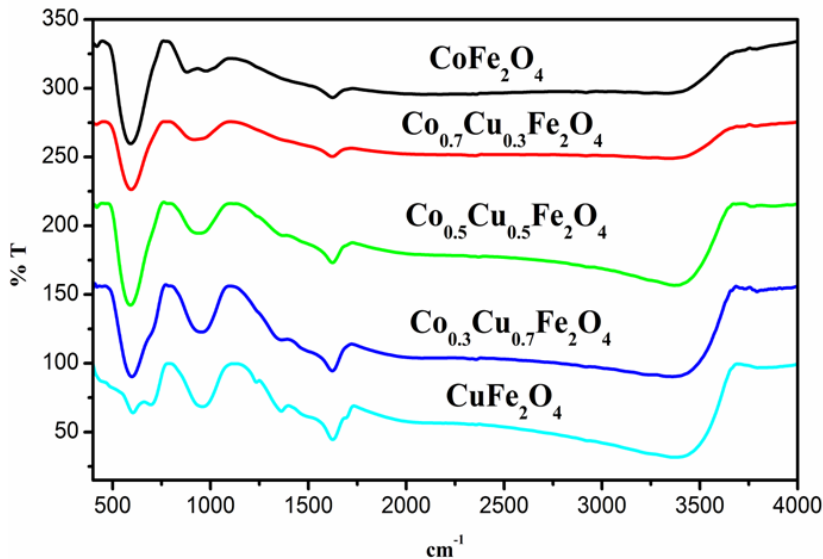


Figure. 4 FTIR Spectra of Cu²⁺ substituted CoFe₂O₄ at room temperature (Co_{1-x}Cu_xFe₂O₄, with x=0, 0.3, 0.5, 0.7 and 1)

Table 2. FTIR transmittance bands for Co_{1-x}Cu_xFe₂O₄ (with x=0, 0.3, 0.5, 0.7, 1)

Samples	IR absorption bands/cm ⁻¹				
	ν_1	ν_2	ν_3	ν_4	
CoFe ₂ O ₄	3327	1623	879	592	
Co _{0.7} Mn _{0.3} Fe ₂ O ₄	3341	1622	918	595	
Co _{0.5} Mn _{0.5} Fe ₂ O ₄	3370	1623	941	591	
Co _{0.3} Mn _{0.7} Fe ₂ O ₄	3359	1622	954	599	
MnFe ₂ O ₄		3377	1625	960	604

The spectra explain that the ferrites could be formed as consistent bonded crystals through ionic, covalent or vander waals forces, to the next lattice positions. The tetrahedral (A sites) and octahedral (B sites) positions in ferrites are occupied by metal ions with respect to the geometrical configuration of nearest oxygen

positions ^[7]. The broad frequency range in between 3796-3788cm⁻¹ and 3377-3327cm⁻¹ represents the O-H stretch which corresponds to hydroxyl group attached to the cobalt oxide surface and it indicates that the water molecules chemically adsorbed by the metal surface during synthesis process ^[20]. Also the frequency range in between 591.96 - 604.33cm⁻¹ represents the existence of metal oxygen bonds. The O-H in plane and out of plane bond appears at 1624.50cm⁻¹ to 1623.92cm⁻¹. The existence of water adsorption bonds, in and out plane and metal oxygen bonds confirms the existence of Co and Cu in the synthesized samples ^[21]. Non metal substituted magnetic nanoparticles are used in various biomedical applications because of its high Curie temperature, magneto crystalline-anisotropy and super paramagnetic behavior at room temperature ^[22]. The adhesion of substratum surface with bacterial cell is being governed by several factors such as physico-chemical properties of substratum, physico-chemical properties of bacterium and suitable environmental conditions in which the adhesion happens ^[23, 24]. Beyond the above mentioned factors, the degree of adhesion of a given bacterium differs with surface properties of the synthesized transition metals substituted nanoparticles. A significant research has been conducted by Sanpo N. et al, to analyse the effects of Cu²⁺ substituted cobalt ferrite nanoparticles on the morphology of *S. aureus* and *E. Coli* bacteria. Typical SEM images indicates that the number of attached bacterial cells on glass surface was decreased than the attachment on Cu²⁺ substituted cobalt ferrite nanoparticles on glass surface. Also, the attached *E. Coli* and its morphology on the surface of Cu²⁺ substituted cobalt ferrite nanoparticles was totally different when compared with the normal glass surface^[23]. Series of studies have been conducted with Cu²⁺ substituted nanoparticles on *E. Coli* shows that the copper ions ruin bacterial cell wall and lysis of the cytoplasm results in cell death. Also high concentrations of Cu²⁺ substituted nanoparticles shows cytotoxicity against *E. Coli*^[25]. The fascinating fact in ferrites is that the synthesized magnetic nanoparticles can also be used in different technological applications such as high density magnetic storage, bubble devices, electronic communication devices, sensors, magnetically guided drug delivery ^[26, 27]. Super lattice magnetic multi layers show excellent giant magneto-resistance (GMR) characteristics and findings in various applications as reading head, magnetic sensors and high density recording media ^[28].

CONCLUSIONS

In this paper, Cu²⁺ substituted cobalt ferrite (Co_{1-x}Cu_xFe₂O₄, x=0, 0.3, 0.5, 0.7 and 1) magnetic nanoparticles were synthesized via chemical co-precipitation method. The obtained powder samples are subjected to various characterization studies. The XRD results confirm the crystalline nature and presence of single phase cubic spinel structure of the obtained magnetic nanoparticles. The VSM results show that the magnetic parameters coercivity (H_c), retentivity (M_r) decrease with increase in Cu²⁺ substitution and saturation magnetization (M_s) shows increment and decrement with Cu²⁺ substitution in CoFe₂O₄ magnetic nanoparticles. The FTIR analyses show the presence of water adsorption bonds and the metal surface which absorbs water during synthesis process; also it clearly shows the existence of metal oxygen bonds which confirms the presence of substituted non-magnetic ions with magnetic nanoparticles. SEM result shows the morphology and composition of synthesized magnetic nanoparticles. Thus, from the basic characteristic techniques the size, structural and magnetic properties of Cu²⁺ substituted CoFe₂O₄ (Co_{1-x}Cu_xFe₂O₄, x=0, 0.3, 0.5, 0.7 and 1) magnetic nanoparticles were investigated. From the reviewed applications of transition metal substituted magnetic nanoparticles, this paper suggests that the Cu²⁺ substituted CoFe₂O₄ magnetic nanoparticles could be

used in drug delivery systems, biotechnology and biomedical applications. In future ferrofluids and nanofluids can be produced from the synthesized Cu²⁺ substituted CoFe₂O₄ magnetic nanoparticles which can be used as transformer oil, engine coolants and as coolant in heat exchangers.

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