

SYNTHESIS AND CHARACTERIZATION OF COBALT FERRITE NANOPARTICLES AND THEIR APPLICATIONS

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ABSTRACT: *The aim of this paper focus on the synthesis procedure of magnetic nanoparticles (MNPs) which have potential applications in nanomedicine, including cellular therapy, tissue regeneration, Nano-biosensors, drug delivery, MRI, and MFE. Moreover, due to their unique properties, MNPs may use various of purposes. Numerous of studies have been conducted in the synthesis of magnetic nanoparticles in recent years. In last decade it has been observed that huge number of publications described the efficient approaches for synthesis procedures of monodisperse magnetic nanoparticles, extremely stable and creating shape-controlled. Most studies found that cobalt ferrites could be a promising material for various application. In this research, a narrow size range of cobalt ferrite nanoparticles (CFNP) were synthesized by chemical precipitation procedures. The resulting powder was calcined at temperatures ranging from 600 to 1000 degrees Celsius. Characterization methods such as Transmission Electron Microscopy (TEM) for morphology and size, UV-VIS and FTIR analysis for chemical composition all lent credence to the findings. Each method of characterization agreed that single-phase CFNP develops in the 10-115 nm range, which is consistent with the annealing temperature.*

Keywords: Nanoparticles of Cobalt ferrite, Synthesis, Characterization.

INTRODUCTION

John Dalton, the "father of modern physics," is credited with creating nanotechnology, a cutting-edge field of study. The goal of nanotechnology is to directly manipulate matter at the atomic level, hence it generates new materials with nanoscale dimensions (called nanoparticles). This technology is adaptable and useful in many fields, including the medical, electronic, and power-generating sectors, all of which are growing to meet the demands of modern civilization. As their size reduces to that of nanoparticles, the magnetic properties of nanoparticles begin to depart from those of bulk materials [2]. Nanoparticles have a high volume-surface ratio, making them useful in many novel ways [3].

Spinel ferrite has the formula MFe_2O_4 (M = Co, Ni, Zn, and others) and has cubic, octahedral/tetrahedral positions [4]. Cobalt ferrite ($CoFe_2O_4$) is one of the spinel ferrites; it has an inverted spinel structure with an equal number of Fe^{3+} ions in the A and B sites and all Co^{2+} ions in B sites. Cobalt ferrite has attracted a lot of interest [5-7] because of its mild saturation magnetization, strong coercivity, mechanical hardness, good chemical stability, and impressive electromagnetic performance. Because of these qualities, it might be utilized in electrical components for things like computers, recorders, and magnetic cards. These characteristics, which may be modified during production and synthesis, are dependent on the material's chemical makeup and microscopic structure.

For research and development into the synthesis of cobalt ferrite nanoparticles using a variety of approaches has arisen as a prominent area of study [8-9]. To create cobalt ferrite nanoparticles with controlled size and shape, various precipitation agents, such as polyol methods, sol-gel, organic matrices in the polymeric precursor, and microemulsion methods [12-13] have all been utilized [11]. While several of these methods have been successful in producing sizes in desirable [14, 15] and micro-structures, scaling up their usage has proven difficult due to factors such as cost, complexity, reaction time, the toxicity of reagents and byproducts, and environmental impact [16-19].

Nanoparticles made from $CoFe_2O_4$ are intriguing due to their outstanding chemical stability, large magnetostrictive coefficient, considerable magnetocrystalline anisotropy, high temperature, magnetic properties, and unique optical character. Features like as excellent thermal stability, mechanical hardness (particularly due to its catalytic capabilities), inertness, and low electrical resistivity make it ideal for use in recording media (metal & alloys unstable under air conditions), memory cores, and high-frequency devices.

In the present study, cobalt ferrite nanoparticles were synthesized using a thermal technique from an aqueous solution consisting of deionized water, polyvinyl pyrrolidone, and metal nitrates. This method eliminates the need for the use of harmful chemicals while providing the advantage of the ease of use. Multiple techniques were used to analyze the generated cobalt ferrite nanoparticles' textural and morphological features, confirming the particle size and distribution and uncovering additional aspects of relevance.

MATERIALS AND METHOD

The precursors were metal nitrate reagents, and the solvent was deionized water. Polyvinyl pyrrolidone (PVP) was used for particle aggregation. Across Organics offered for sale cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$) and iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$), both of which had purity levels of more than 99%. By adding 3g of PVP solution in deionized water (100ml) at 343 K the aqueous solution was prepared. Afterward, a magnetic stirrer was used to constantly mix in $Fe:Co=2:1$ for 2 hrs. The result was a clear, colorless solution. As measured by a glass electrode, the solution's pH was in the acidic range of 1 to 2. Before the heat treatment, no significant precipitation had occurred. To remove the water, the mixed solution was baked at 353 K for 24 hours in a glass Petri dish. After 15 minutes of grinding in a mortar, the remaining orange solid was reduced to a powder. The powder was calcined at 673, 723, 823, and 923 K for 3 hours to decompose organic components and crystallize nanoparticles.

Characterization

Nanomaterials engineered for specific properties and uses may be characterized in a number of ways. In order to understand the reaction mechanism and identify the chemical components and their properties, high-tech analytical equipment is necessary. Using XRD the nano-particles CoFe_2O_4 structure was determined. Cobalt ferrite powder crystallinity was evaluated by XRD and FT-IR spectroscopy at a range of calcination temperatures. The Nano crystalline powder was analyzed for its atomic structure and particle size using TEM at room temperature using a JEOL 2010F UHR version electron microscope operating at 200kV accelerating voltage. Select portions of the TEM images were taken to analyze the form and particle size distribution throughout a range of calcination temperatures. Cobalt ferrite nanoparticles' magnetic characteristics were characterized.

RESULTS AND DISCUSSION

In Figure 1, it has a simplified depiction of the interactions between PVP and metal ions (iron and cobalt).

The process of calcination is made easier by the fact that metallic ions are held in place in the cavities of the polymer chain. This makes a solid solution of the metal oxides.

It is clear from Fig 2(a) that the precursor, which is still amorphous and devoid of identifiable diffraction patterns, has a huge peak. Reflection planes (111), (220), (222), (311), (400), (422), (511), (440), and (533) in cobalt ferrite calcined at 673, 723, 823, and 923 K demonstrate the presence of a face-centered cubic structure (ICDD PDF: 22-1086). Our results show that an increase in calcination temperature

results in sharper, narrower diffraction peaks with greater intensity. This means that the nuclei have a higher crystalline volume ratio, which strengthens the crystallinity [22]. The greater particle size is a byproduct of the nuclear fusion process. The impure phase exists in every calcined sample of $-\text{Fe}_2\text{O}_3$. This impure substance occurs in nature as hematite [23].

The average particle size was determined by using the FWHM of the XRD patterns using the well-known Scherer formula [24]:

$$D = 0.9\lambda / \beta \cos\theta, \quad (1)$$

Where λ is the X-ray wavelength of Cu K = 0.154 nm, θ is the Bragg angle [25], and β is the full width at half maximum (FWHM) of the diffraction line in radians. The dimension D of a crystal (nm). Particle sizes were found to rise with calcination temperature as predicted by the Scherer formula (Table 1), a rise that begins at 10 nm at 673 K and continues until it reaches 41 nm at 923 K.

The FT-IR analysis of precursor and calcined materials, showing wave numbers from 280 to 4000 cm^{-1} , is shown in Figure 3. Figure 3 displays all of the precursor absorption peaks that might be attributed to PVP (a). O-H stretching, C=O bending, H-C-H bending, C-H bending, and C-C bending are all represented by the most prominent peaks at 3345, 1638, 1430, 1230, 842, and 670 cm^{-1} [23]. It is important to notice that the absorption maxima at 1230 and 1430 cm^{-1} of the nitrate anion correspond with the C-H vibration.

Table 1: Calcined samples were analysed for their magnetic properties and room temperature XRD and TEM measurements of CoFe_2O_4 nanoparticle size (nm) revealed no significant differences.

CoFe_2O_4 nanoparticles	Calcination temperature (K)	Average particle size XRD (nm)	Average particle size TEM (nm)	Saturation magnetization M_s (emu/g)	Remanent magnetization M_r (emu/g)	Coercivity H_c (Oe)	Remanence ratio
CoFerrite 1	673	10	12.5	1.15	0.10	138	0.087
CoFerrite 2	723	15.5	14	2.14	0.57	823	0.266
CoFerrite 3	823	34	29	18.02	9.02	1791	0.500
CoFerrite 4	923	41	39	28.52	11.61	1002	0.407

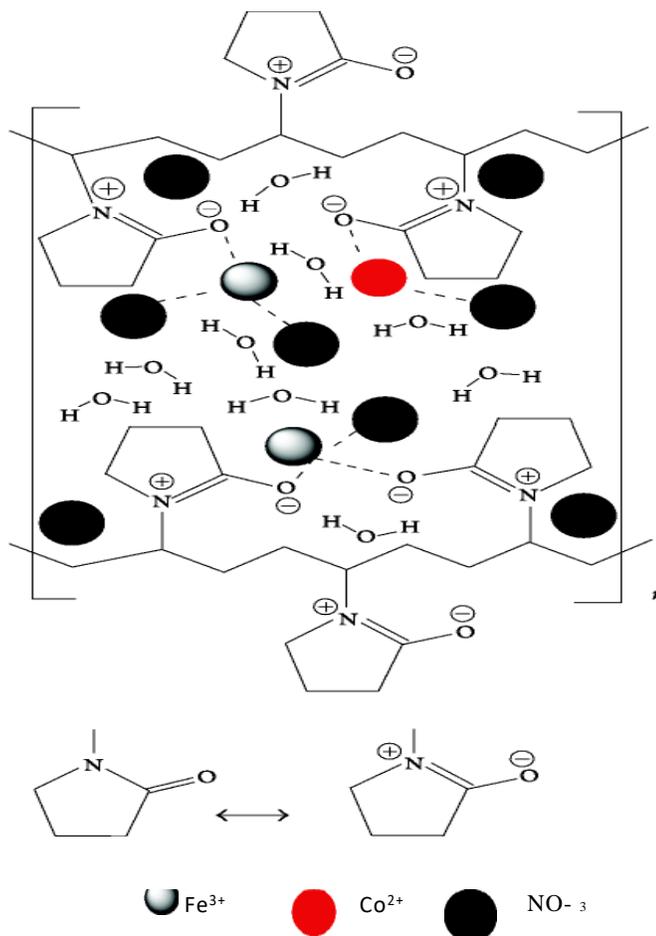


Figure 1: Speculations about the nature of the interactions between PVP and metal ions prior to calcination.

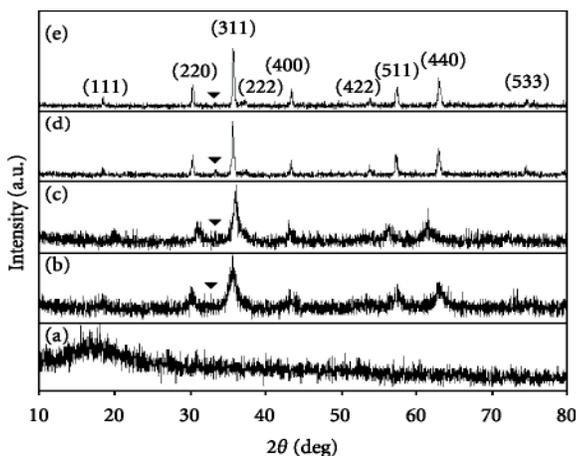


Figure 2: Pattern of XRD (a) the precursor and (b) calcined $CoFe_2O_4$ at 723(c), 823(d) 923(e) K. The PVP matrix and crystalline $CoFe_2O_4$ nanostructure

of cobalt ferrite samples were examined at various calcination temperatures. When calcined at 823 K, pure spinel cobalt ferrite nanoparticles displayed vibrational spectra of the absorption bands at 334 and 545 cm^{-1} , but samples calcined at 923 K showed 331 and 540 cm^{-1} (Fig 3d,e). As a direct result of this, the spinel $CoFe_2O_4$ nanostructure is made more robust [9, 27]. The crystal lattice's ion oscillation causes solids' FT-IR absorption bands [28]. Organic sources are unlikely since samples over 823 K exhibited no C-H stretching mode peaks. FT-IR analysis reduces undesired ions that may distort the crystal lattice during calcination at 823 and 923 K. The O-H stretching vibration-caused peak at 3397 cm^{-1} remains at 823 K (Figure 3(c)).

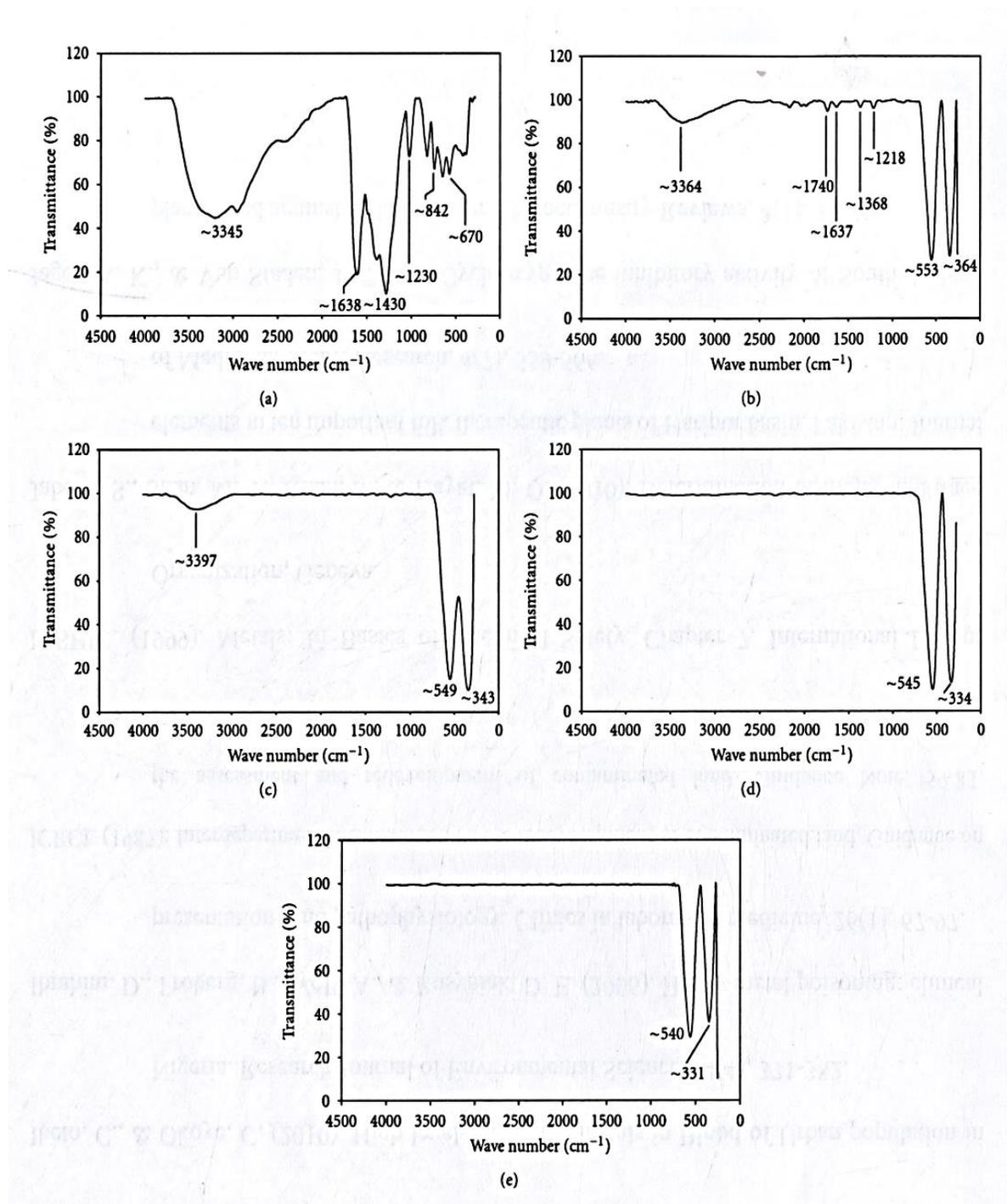


Figure 3: FTIR spectra in the 280-4000 cm⁻¹ region for (a) the precursor and (b) calcined cobalt ferrite nanoparticles

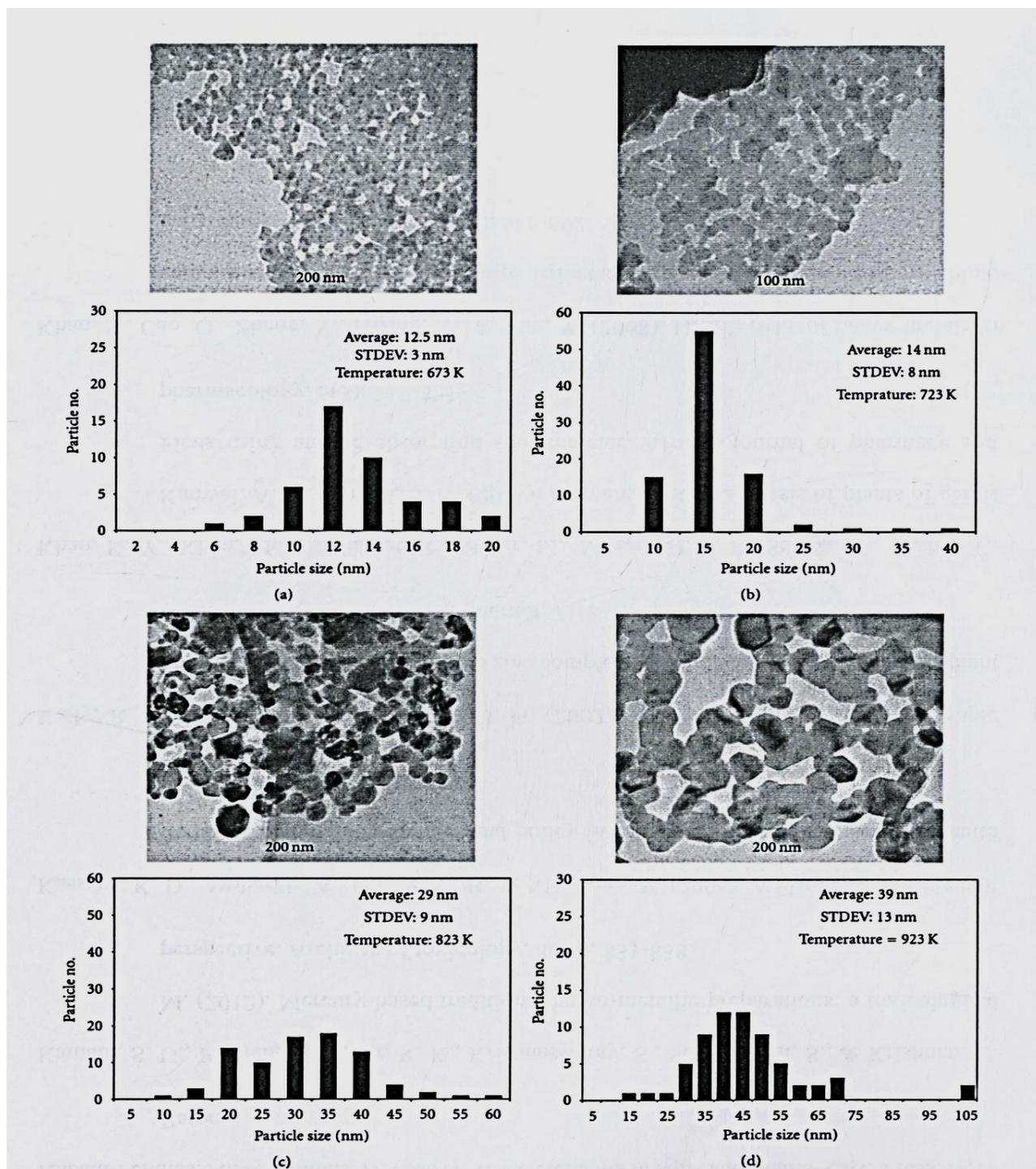


Figure-4 Cobalt ferrite nanoparticle TEM images and particle size distribution histograms at (a), (b), (c), and (d) 673, 723, 823, and 923 K, respectively

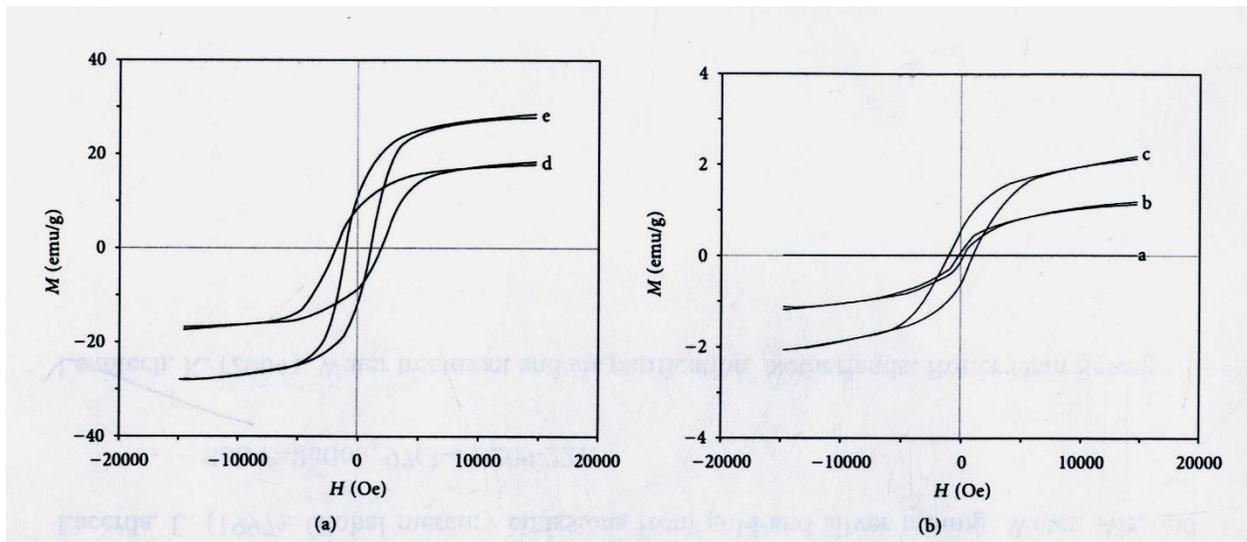


Figure 5: Magnetization curves of (a) uncalcined CoFe_2O_4 and (b-e) calcined cobalt ferrite nanoparticles at (673-e) 723, (823-e), and (923-e) K.

Calcined material had CoO and FeO bond bands at 364 and 553 cm^{-1} . When the calcination temperature was elevated to 923 Kelvin, the CoO and FeO bond absorption bands shifted to new positions at 331 and 540 cm^{-1} , respectively. These connections often strive for nanoparticles made of pure CoFe_2O_4 [9].

Figure 4 shows cobalt ferrite particle sizes and TEM pictures. These findings demonstrate that the heat treatment method yields samples with a constant morphology and particle size distribution. As expected, the XRD data agrees with the hypothesis that particle size increases as a function of calcination temperature (Table 1). The research found that at 673 K, particles were at their smallest (12.5 nm), whereas at 923 K, they were at their biggest (39 nm). This indicates a cluster of particles in close proximity work together to melt their surfaces and grow in size [30]. To get rid of all organic molecules, you need to heat the mixture to 823 K.

Likewise, the particle size of the material that was calcined at a temperature of 923 K is reduced (Figure 4(c)) (Figure 3(c)). It has previously been seen in systems such as zinc ferrite [31] and nickel ferrite [32] that grain formation and an increase in particle size may occur when calcination temperatures are raised.

The $M(H)$ loops tested from -15 to +15 kOe are shown in Figure 5 at standard room temperature. In contrast to the nonmagnetic precursor (Figure 5(a)), all of the calcined materials exhibited typical magnetic properties after heating. A look at Table 1

reveals that raising the calcination temperature from 673 to 923K results in an increase from 1.15 to 28.52 emu/g in saturation magnetization when the sample is kept at room temperature. The greatest saturation magnetization of the 923 K-calcined samples was only 28.52 emu/g , which is much lower compared to the 74.08 emu/g that was reported for multi-domain bulk cobalt ferrite [33]. The mechanisms that cause the magnetization lower are complex. Since it affects saturation magnetization, calcination heating rate is crucial to thermal treatment. Within the scope of this investigation, cobalt ferrite nanoparticles were subjected to a calcining temperature of 10 K per minute. The temperatures that correspond to these are 673, 723, 823, and 923 K respectively. In theory, a larger saturation magnetization might result from an increase in the magnetic phase, and a slower heating rate during the calcination process could result in a more complete crystallization of the material. Both of these outcomes are based on speculation.

The saturation magnetization of electrospun nanostructures of cobalt ferrite at 773 degrees Celsius was raised by Sangmanee and Maensiri [27]. Because Co^{3+} ions are present, as well as a cation distribution that includes cobalt ions on the tetrahedral site [34], the saturation magnetization of the nanoparticles with a mixed spinel structure type is inhibited [36]. This is because of the presence of cobalt ions.

Figure 2 depicts what may be a reduction in saturation magnetization [23, 37] as a result of the formation of a hematite phase that is only weakly magnetic and impure. Figure 6 illustrates how the saturation (M_s) and remanent (M_r) magnetizations of cobalt ferrite nanoparticles fluctuate with particle size. Because of surface effects, the values of saturation magnetization and remanent magnetization in calcined samples of nanoparticles rise as particle size increases.

Saturation and remanent magnetization go up with particle size but go down with particle size [38]. The coercivity field increases to 1791 Oe, while the remanence ratio values ($R = M_r/M_s$) decrease to 1002 Oe and 0.407 for particles with a size of 39 nm (Figure 7).

The value of R , which may vary from 0 to 1, regulates how often certain intergrain group exchanges take place [39]. Consistently rotating, randomly oriented, noninteracting particles have been shown to have an R -value of 0.5 [40, 41], whereas particles engaging through magnetostatic contact have been observed to have an R -value of 0.5. Finally, particles with exchange coupling are shown by $1 > R > 0.5$.

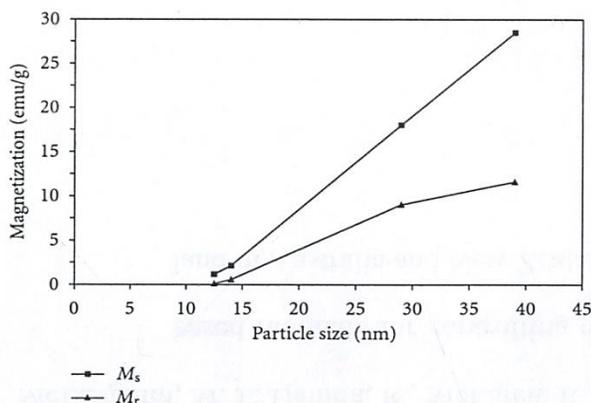


Figure 6: Samples of calcined cobalt ferrite nanoparticles were subjected to temperature changes, and their saturation magnetization and remanent magnetization were measured in relation to particle size.

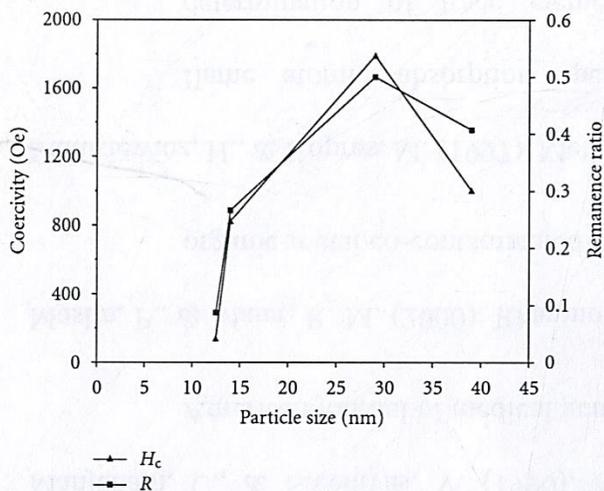


Figure 7: Calcined $CoFe_2O_4$ at varying temperatures show size-dependent changes in remanence ratio/coercivity field.

Size-dependent changes in the remanence ratio and coercivity field may be described in terms of domain structure, critical size, and crystal anisotropy [30, 42-45]. While the polymeric precursor method [14] produces 13 nm cobalt ferrite nanoparticles with values of 67.5 emu/g (M_s), 1842 Oe (H_c), and 0.5 remanence ratio under the same conditions, co-precipitation [30] produces 16 nm cobalt ferrite nanoparticles. Precipitation is one of several methods used to make

nanoparticles, and it stands out for being adaptable, user-friendly, yielding large quantities of the final product at a low preparation temperature, and allowing for precise manipulation of particle properties. Sizes of particles in the range of nanometers to micrometres are more typical [46-48].

CONCLUSION

Cobalt nitrate, iron nitrate, PVP, and deionized water were used to synthesize spinel cobalt ferrite ($CoFe_2O_4$) nanoparticles. TEM and XRD examinations of particles calcined between 623 and 923 K showed diameters from 12.5 to 39 nm. Calcination at 823 and 923 K eliminated all organic components and nitrate ions. Researchers observed cobalt ferrite nanoparticle saturation, remanence ratio, magnetization, and coercivity field. This easy, inexpensive, and ecologically benign approach produces pure nanoparticles of cobalt ferrite. It might make nanotechnology-related spinel ferrite nanoparticles. As shown by the magnetization curve, co-precipitation-synthesized materials are ferrimagnetic. CFNP magnetic properties vary with particle size.

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