

The Influence of the Various PH Value on Structural and Magnetic Properties of Cobalt Ferrite Nanostructure

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ABSTRACT

In this work, CoFe₂O₄ nanostructure was synthesized from Cobalt and ferric nitrates and sodium hydroxide (NaOH) by hydrothermal method. Effect of PH on the structural and magnetic properties of the resulting powder was studied at calcination 400°C and 600°C. The resulted products were characterized by X-ray diffraction (XRD), Field emission scanning electron microscopy (FE-SEM), Fourier transformed infrared (FTIR), and vibrating sample magnetometer (VSM) technique as synthesis, when calcination at 400°C and 600°C with different PH. The reduction in crystallite size that their average crystallite size is found (13.98-22.17) nm crystallite size at PH=9 higher than when PH=3. The FE-SEM images indicated that particles were agglomerated and spherical shape and size between (23.6 - 57.9) nm for PH=9 and (23.3 - 114.6) nm for PH=3. The created CoFe₂O₄ nanostructure have demonstrated ferromagnetic behavior for all the samples behavior which was found in VSM results examination.

Keywords: CoFe₂O₄ Nanoparticle, Hydrothermal Synthesis, Magnetic Properties

1. Introduction

Cobalt ferrite (CoFe₂O₄) is a significant member of the spinel ferrite family, characterized by its inverse spinel structure. In this structure, oxygen atoms form a face-centered cubic (FCC) lattice, while half of the Fe³⁺ ions occupy the tetrahedral sites, and the remaining half, together with Co²⁺ ions, are situated at the octahedral B sites [1].

CoFe₂O₄ nanoparticles have been made using different methods, such as coprecipitation [2], microemulsion synthesis [3], sol-gel synthesis [4], and hydrothermal methods [5]. Typically, the aforementioned approaches necessitate the utilization of a metal salt and a potent base in order to facilitate the formation of the intended oxide. However, it should be noted that the dimensions and structure of the entity are highly susceptible to factors such as pH and temperature [6].

In this work, we have prepared CoFe₂O₄ samples using hydrothermal technique and characterized by XRD, FTIR, and FE-SEM. At room temperature, a vibrating sample magnetometer (VSM) was used to test the magnetic properties of CoFe₂O₄ samples that had been made. The investigation also looked at how pH and temperature affected the structure, morphology, and magnetic properties of the CoFe₂O₄ samples that were made.

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2. Experimental Work

CoFe₂O₄ nanostructure was prepared from Cobalt and ferric nitrates using the hydrothermal method. The aqueous solutions of Cobalt nitrate hexahydrate with 0.2M was dissolved in distilled water and stirred in a magnetic stirrer until the solution appears pink. Ferric nitrate monohydrates (Iron nitrate nanohadrates) with 0.4M was dissolved in distilled water and stirred it until the solution appears orange. Dissolve NaOH with (1, 2, and 3) M in deionized water and stirred at 70oC the resultant solution appears transparent. Then cobalt salt and iron salt were gradually added to NaOH boiled solution, then the pH of the solution was measured and its value was 3 and 9. The resultant solution stirred for (6h) at 70 oC, the solution appears black. The resulting solution was placed in an autoclave and then placed in an oven at 100 oC for (3h). Resulting solution centrifuged for 30 min with 6000 revolutions per minute, then this process was repeated three times. Dried the resulting precipitate at 100 oC for 30 min, the material appeared in the form of a small black mass. The resulting substance was ground well and became a black powder, then annealed at 400oC and 600oC the resulting samples are ready for examined .These same steps were repeated different concentrations of NaOH. In the present study, CoFe₂O₄ nanostructure have been prepared by using the hydrothermal technique and calcined at various temperatures (400, and 600) oC. The purpose of this work is to examine how the PH and calcination temperature affects morphological and structural of CoFe₂O₄ nanostructures synthesized. By using a Shimadzu version 4 (XRD) system, the structure related to the CoFe₂O₄ nanostructures was analyzed, and the intensity was recorded as a Bragg angle function. The radiation source was Cu (K α) with $\lambda= 1.5406 \text{ \AA}$ wavelength was used as the radiation source, while 40kV and 30mA were used for the voltage and current, respectively. The scanning angle 2θ was varied between 10 and 90 degrees step size 0.2 degree, at a speed of 10 degrees per min, with a preset time of 1.2 seconds. For analyzing the morphology regarding samples, a Field emission scanning microscope (FESEM) is used (MIRA3model-TE-SCAN (Dey Petronic Co.)). The Fourier Transform Infrared (FTIR) test is employed to examine the spectra of various substances. This is achieved by utilizing a Bruker FTIR analyzer, specifically the Tensor-27 model, manufactured by Bruker Optics Inc. in Billerica, MA. The test is conducted in an attenuated total reflectance mode. The data were recorded within the 400–4000 cm⁻¹ wave number range. An AGFM-VSM model 117 was used for recording M-H curves regarding the samples during magnetic experiments with the use of a VSM.

3. Results and Discussion

The cobalt ferrite nanostructure fabricated by employing a hydrothermal approach with 1 M of NaOH concentration, **PH is 9** and calcined for 0 °C, 400 °C and 600 °C are confirmed by the peaks observed (Fig. 1). The samples display the recognizable reflection from (220), (311), (222), (400), (422), (511), (440), and (533) planes, which are in good agreement with those of CoFe₂O₄ obtained from JCPDS card no. 96-591-0064. Another finding is that peaks shift to lower 2θ as the temperature of calcination rises, this shows that as thermal energy is added, the rate at which cobalt ions are incorporated into the lattice increases[7]. Due to the fact that the peak shift and lattice constant vary depending on the substituted cations, this makes it possible to discriminate between the diffraction patterns of distinct spinel ferrites[8]. However, when the calcination temperature increased, the peak strength steadily dropped, indicating that the expansion of peaks was hampered by the rise in calcination temperature. The vibrating of the atoms in the sample may be directly connected to the drop in nanoparticle peak intensity as temperature rises[9]. A recent study [10] has provided

evidence regarding the relationship between changes in the distance between adjacent crystal planes (referred to as d) and variations in atomic thermal vibration resulting from temperature increases. According to the study, the diffraction peak's 2 position shifts from its initial position as temperature rises, and the intensity of the diffraction peak decreases. The high purity of the product is supported by the fact that the XRD spectra did not find any further impurity peaks [11].

The determination of crystallite sizes in the ferrite-calcined samples was conducted by employing Debye-Scherrer's formula [12] [13], specifically focusing on the greatest intensity (311) peaks. The obtained results have been documented in Table 1.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

Where k = shape factor, $\lambda = 1.54060 \text{ \AA}$, β = Full Width Half Maximum (FWHM) and θ = diffracting angle. The results showed that when the calcination temperature increased, the crystallite sizes had relatively reduced. The high temperatures of the calcination process may be to blame for the nanoparticles' cracking or fracturing, which results in the reduction in crystallite size[14]. Using Bragg's Law, the lattice parameter was calculated from the XRD pattern [15]. The symbol for interplanar spacing is " d ," and the lattice parameter for the corresponding 2 peak position value is " a ." The integers h , k , and l are commonly referred to as Miller indices [16].

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (2)$$

Lattice constant has expanded from 8.39 to 8.53 \AA . The substituted cations affect how the lattice constant behaves since the radius of Fe^{3+} (0.64 \AA) is lesser than Co^{2+} (0.72 \AA) this answers the rising lattice constant with the introduce of cobalt cation into the lattice. [17]. According to a research [8], the lattice constant increased as the calcination temperature rose.

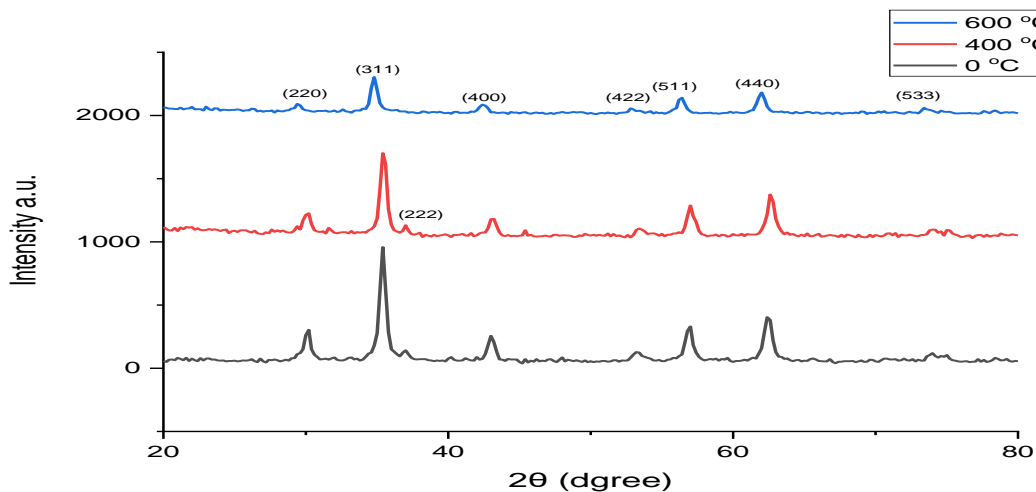


Figure 1: XRD of CoFe_2O_4 with different calcination temperature with pH is 9.

Figure (2) demonstrate the synthesized cobalt ferrite nanostructure by employing a hydrothermal technique with 1 M of NaOH concentration, **PH is 3** and calcined for 0 °C, 400 °C and 600 °C. The samples display the recognizable reflection from (311), (511), (440), and (622) planes, which are in good agreement with those of CoFe_2O_4 obtained from JCPDS card no. 96-591-0064. Peaks at 2θ 24.6°, 33.6°, 49.8°, 54.4° and 72.6° is belong to hematite $\alpha\text{-Fe}_2\text{O}_3$. Quyen *et al*[18] clarified the reason of the most peaks belong to hematite that the creation of iron hydroxide precursors is frequently at low pH solution and therefore the Fe/Co molar ratio in the final outcome increasing causes a rise the formation of $\alpha\text{-Fe}_2\text{O}_3$ phase in the calcined products. Meanwhile Lafta *et al* [19] explain the low concentration of sodium hydroxide (low pH) leads the reaction to produce $\alpha\text{-Fe}_2\text{O}_3$. Huang *et al* [20] also confirmed the with increase pH the hematite phase disappears. However, the peak strength gradually decreased as the calcination temperature rose, demonstrating that the increase in calcination temperature interfered with the growth of peaks. The decrease in nanoparticle peak intensity as temperature rises may be directly related to the atoms in the sample vibrating[9]. Recent research[10] shown how a rise in temperature-induced atomic thermal vibration might alter the distance between the closest crystal surfaces (d), and as a result, the diffraction peak's 2θ position changes from its initial position. The strength of the diffraction peak also reduces. The calculation of crystallite sizes was performed using Debye-Scherrer's formula (1) based on the (311) peaks. This analysis was conducted for all the ferrite-calcined samples, and the obtained results have been documented in Table 1. The findings demonstrated that the crystallite sizes had decreased as the calcination temperature rose. This could be due to the presence of $\alpha\text{-Fe}_2\text{O}_3$ that was obtained by oxidation process[21]. Using Bragg's Law eq. (2), the lattice parameter was calculated from the XRD pattern. Lattice constant has expanded from 8.27 to 8.30 Å. Given that the radius of Fe^{3+} (0.64) is less than that of Co^{2+} (0.72), the substituted cations have an impact on how the lattice constant behaves, this answers the rising lattice constant with the introduce of cobalt cation into the lattice. [17]. In accordance with a study [8], as the calcination temperature climbed, the lattice constant increased.

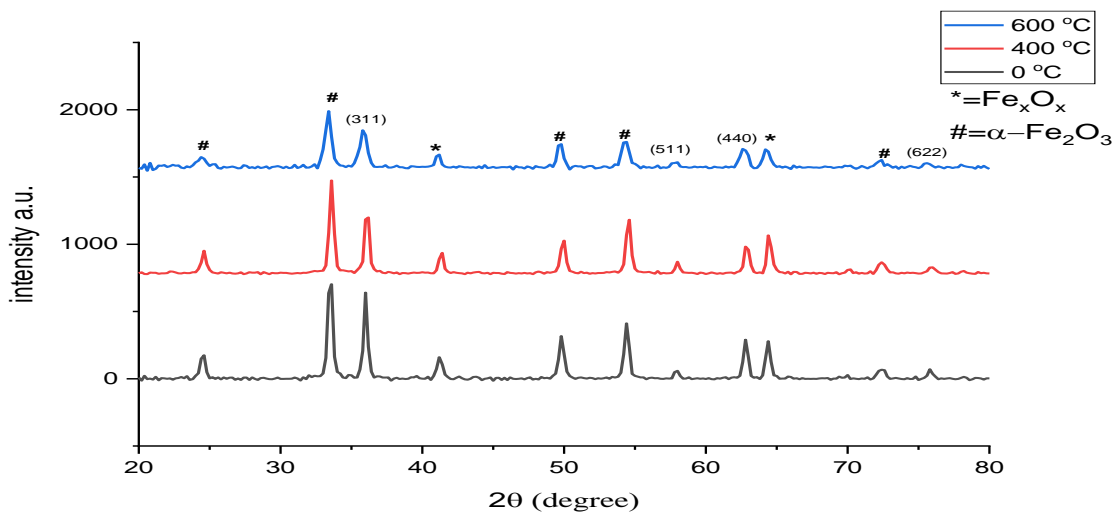


Figure 2: XRD of CoFe_2O_4 with different calcination temperature with pH is 3.

Table 1. Crystallite size and lattice constant of CoFe_2O_4 with different calcination temperature.

Sample Name	2θ (degree)	FWHM β	D(nm)	Lattice Constant
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pH	Calcined Temperature °C		(degree)		(a) Å
9 pH	0	35.4226	0.6173	14.11	8.39
	400	35.468	0.6109	14.26	8.38
	600	34.8149	0.6218	13.98	8.53
3 pH	0	35.995	0.3936	22.17	8.27
	400	36.10637	0.5904	14.78	8.24
	600	35.83659	0.5904	14.77	8.3
JCPDS card		35.632			8.35

Figures (3 and 4) shows calcinated cobalt ferrite nanostructures at (0, 400 and 600) °C for (1 M) of NaOH concentration and pH is (3 and 9) presented in the range 4000–450 cm^{-1} have been investigated by FTIR spectroscopy at room temperature. The popular bands in the range 3779–3426 cm^{-1} , 1637–1616 cm^{-1} and 1385 cm^{-1} are assigned to the O–H stretching vibration demonstrating the presence of water adsorbed at the nanostructures' surface [22]–[29]. The absorption bands at 2922 cm^{-1} were noticed, which are associated with C–H stretches [8], [23]. In addition, the frequency range of 583–562 cm^{-1} is associated with the metal-oxygen stretching vibrations occurring in the tetrahedral sites of spinel ferrites. Similarly, the frequency of 478 cm^{-1} corresponds to the metal-oxygen stretching vibrations in the octahedral sites of spinel ferrites. These findings have been reported in previous studies [15], [26], [30], [31]. The fabrication of cobalt ferrite uses different pH, as illustrated in Figures (3 and 4), which modifies absorption intensity while maintaining the same frequency range. This shows that cobalt ferrite manufacturing has the capacity to change its magnetic properties [32]. FTIR peaks of synthesized cobalt ferrite nanostructures were shown in Table (2).

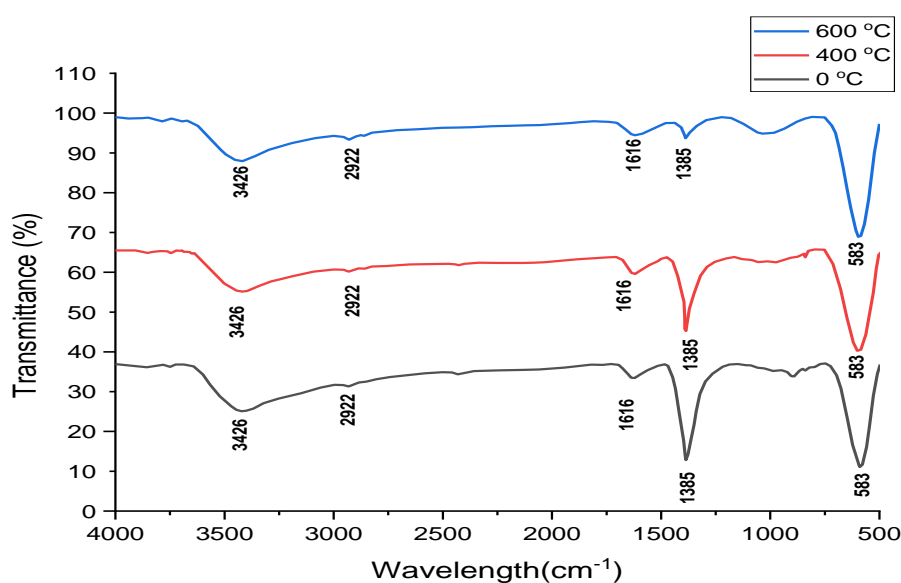


Figure 3: FTIR of CoFe_2O_4 with different calcination temperature with pH is 9.

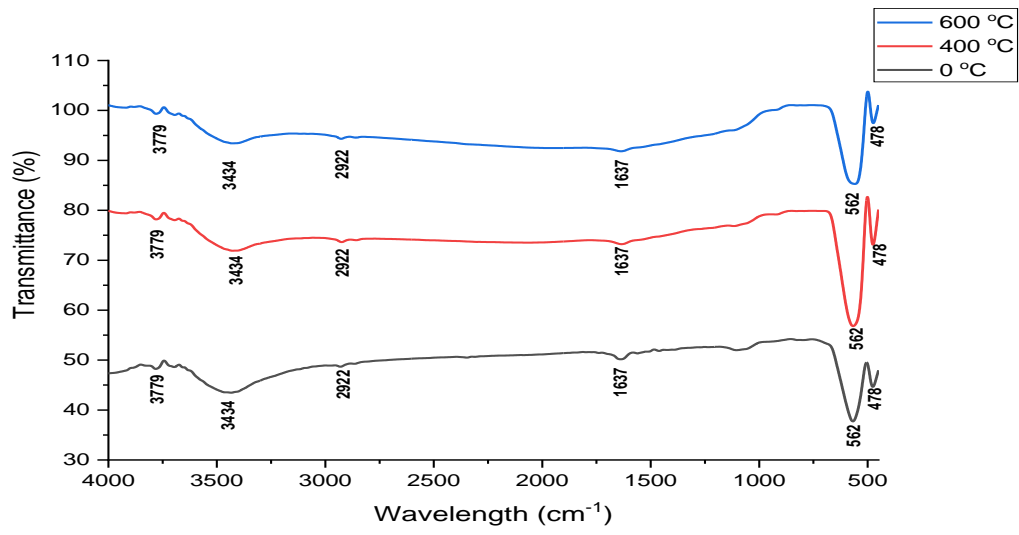
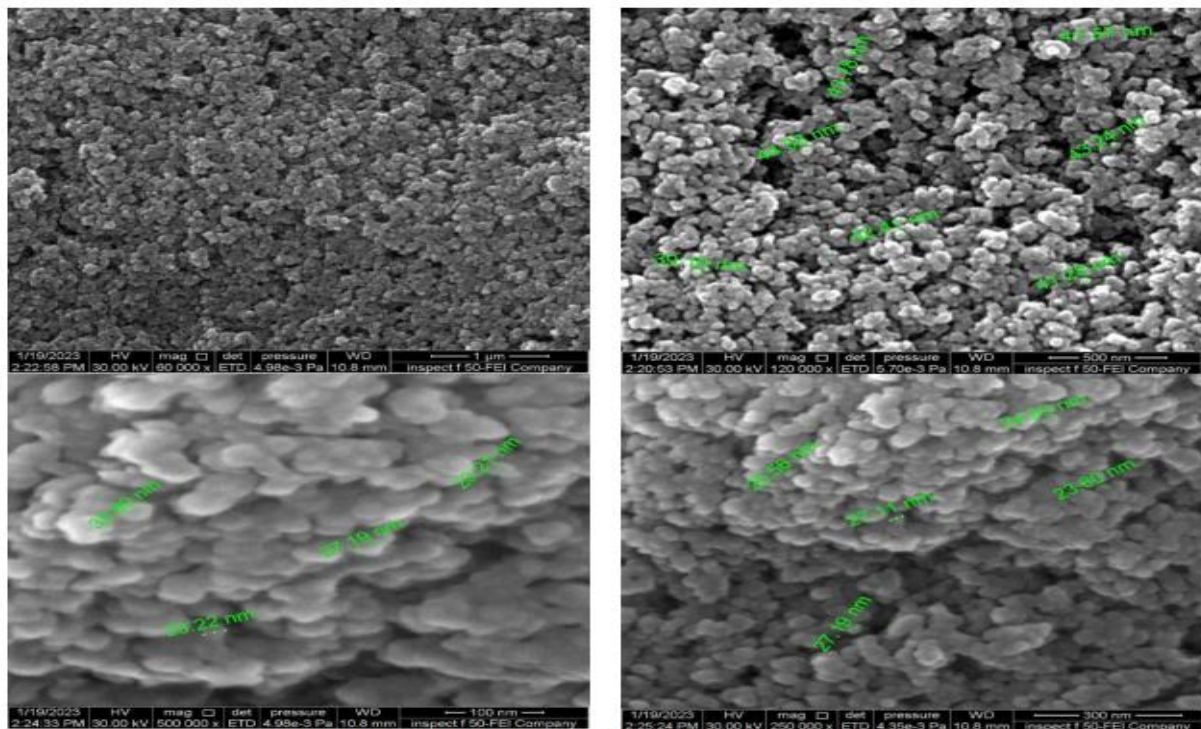


Figure 4: FTIR of CoFe₂O₄ with different calcination temperature with pH is 3.

Table 2: FTIR peaks of synthesized cobalt ferrite nanostructures.

Chemical Bonds	Band Location cm^{-1}	
	9 pH	3 pH
O–H stretching vibration		3779
O–H stretching vibration	3426	3434
C–H stretches	2922	2922
O–H stretching vibration	1616	1637
O–H stretching vibration	1385	
Metal-Oxygen stretching vibrations	583	587
Metal-Oxygen stretching vibrations		478

Figures (5, 6 and 7) shows the FE-SEM micrographs of cobalt ferrite nanostructure synthesized with 9 pH calcinated at different temperatures. The FE-SEM images indicated that particles were agglomerated and spherical shape and size between 23.6 to 57.9 nm. The observed particle size exhibited a greater magnitude compared to the crystallite size ascertained using the X-ray diffraction data. The resulting agglomeration caused multiple crystallites to come together to create a bigger particle due to a higher surface area to volume ratio. Also, due to grain expansion at higher calcination temperatures for the lowering of Gibbs free energy brought on by the reduction of expanded surface area of nanoparticles [15]. In addition, magnetic nanoparticles tend to cluster and stick together due to the interaction of magnetic dipoles[43] Similar results were observed in previous studies [40][39].

**Figure (5):** SEM picture of CoFe_2O_4 with 9 pH without annealing.

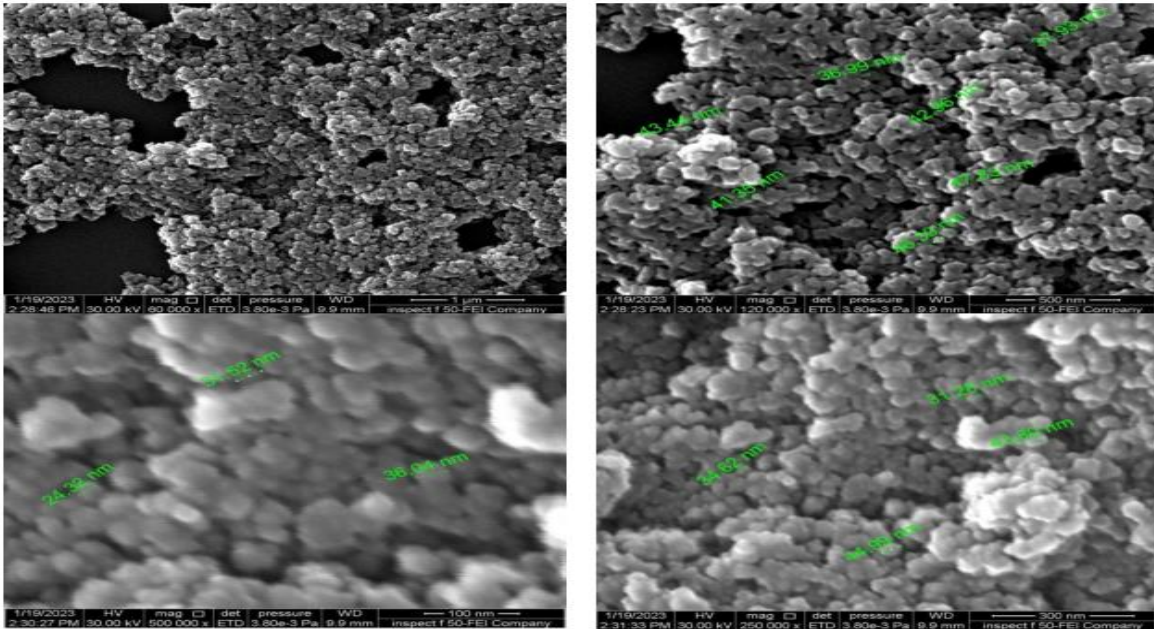


Figure (6): SEM picture of CoFe_2O_4 with 9 pH calcined at $400\text{ }^\circ\text{C}$

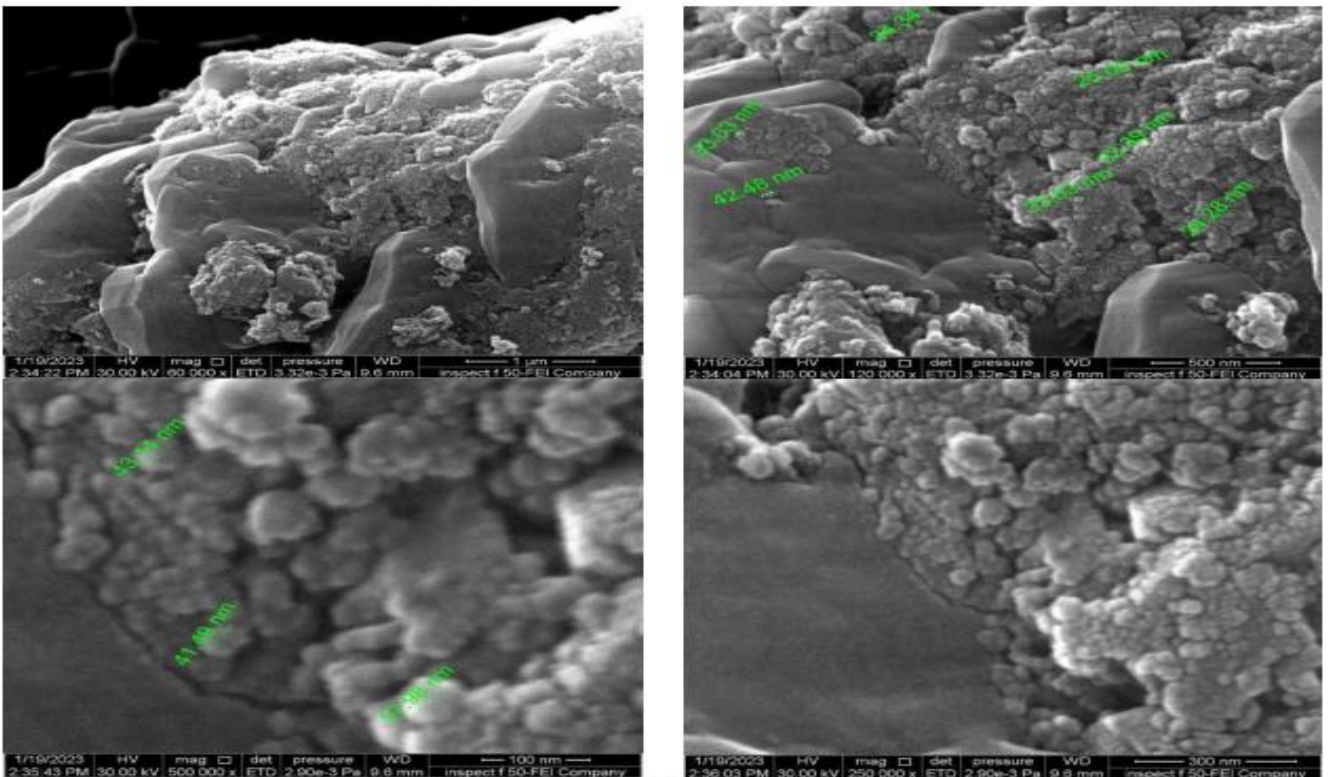


Figure (7): SEM picture of CoFe_2O_4 with 9 pH calcined at $600\text{ }^\circ\text{C}$.

Figures (8, 9 and 10) shows the FE-SEM micrographs of cobalt ferrite nanostructure synthesized with 3 PH calcinated at different temperatures. The FE-SEM images indicated that particles were agglomerated and spherical shape and size between 22.3 to 114.6 nm. The crystallite size determined from the X-ray diffraction data was less than the particle size that was actually observed. The resulting agglomeration caused multiple crystallites to come together to create a bigger particle due to a higher surface area to volume ratio. furthermore

due to the grain expansion at higher calcination temperatures for the minimization of Gibbs free energy as a result of the decrease of extended nanoparticle surface area[15]..Similar results were observed in previous studies[44][45].

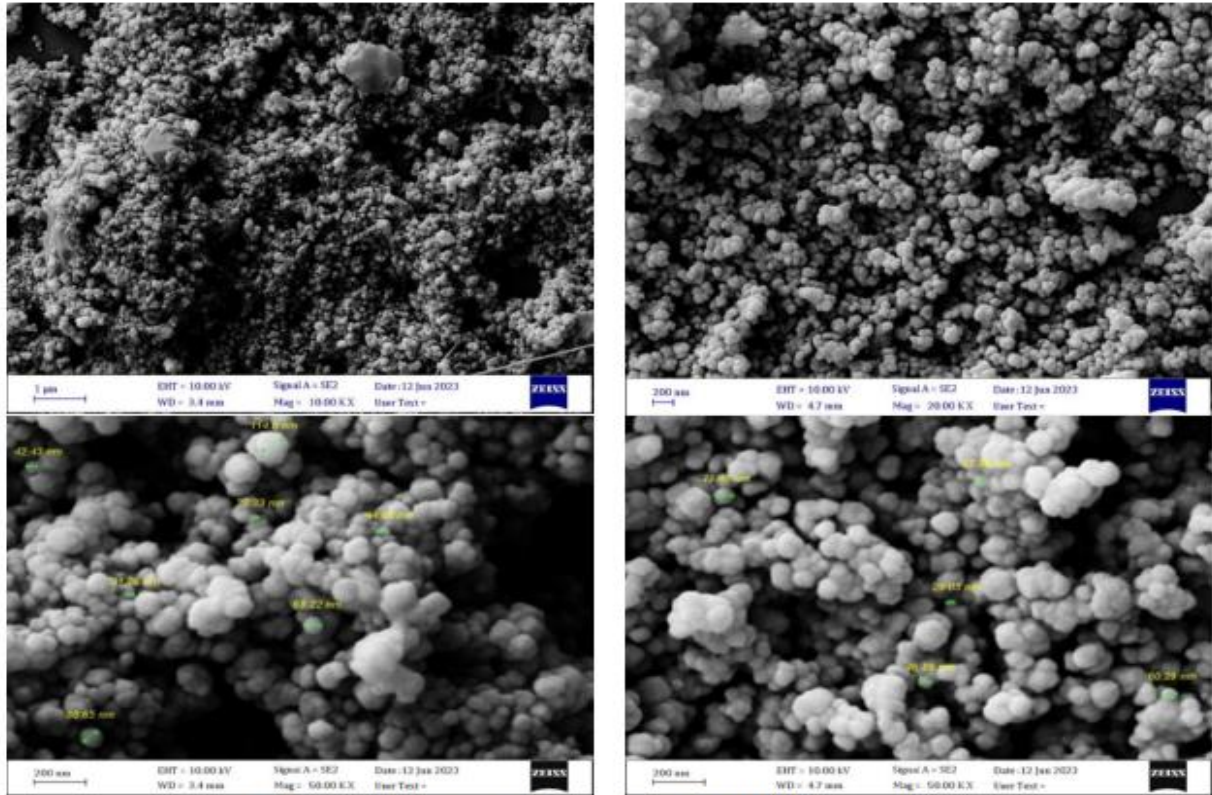


Figure (8): SEM picture of CoFe_2O_4 with 3 pH without annealing.

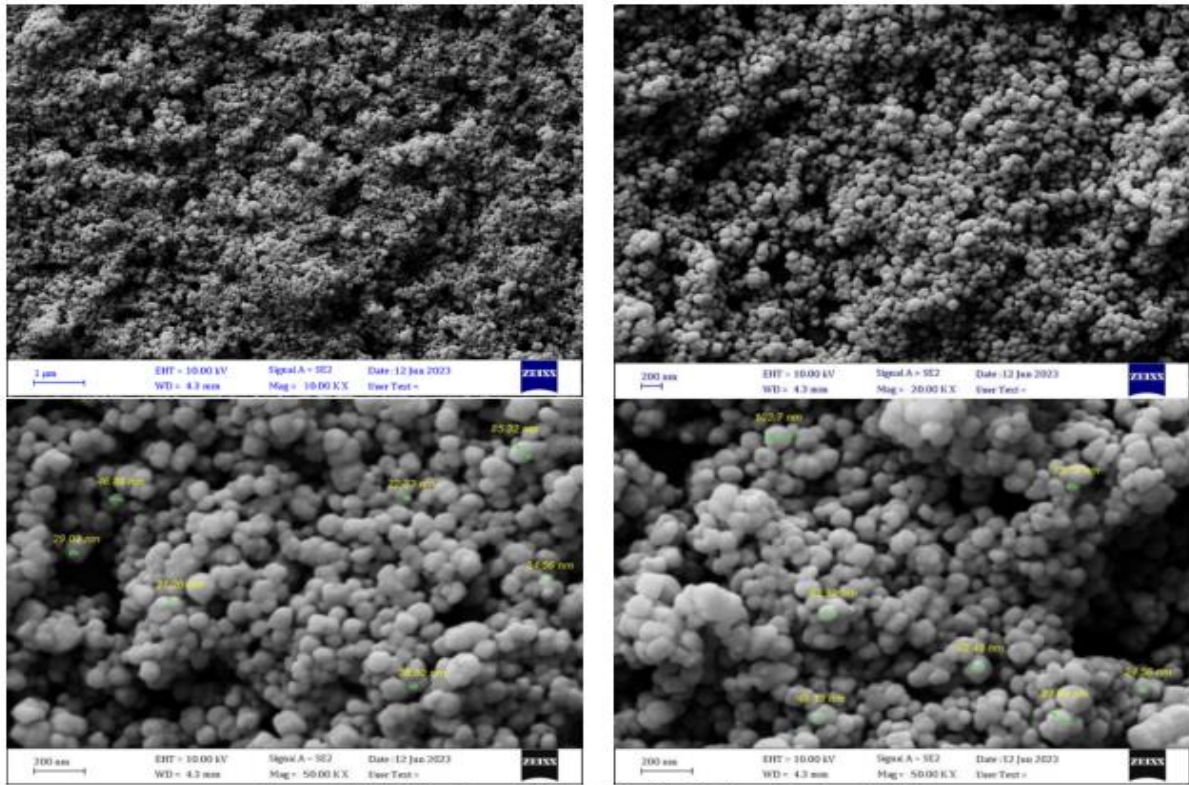


Figure (9): SEM picture of CoFe_2O_4 with 3 pH calcined at 400 °C.

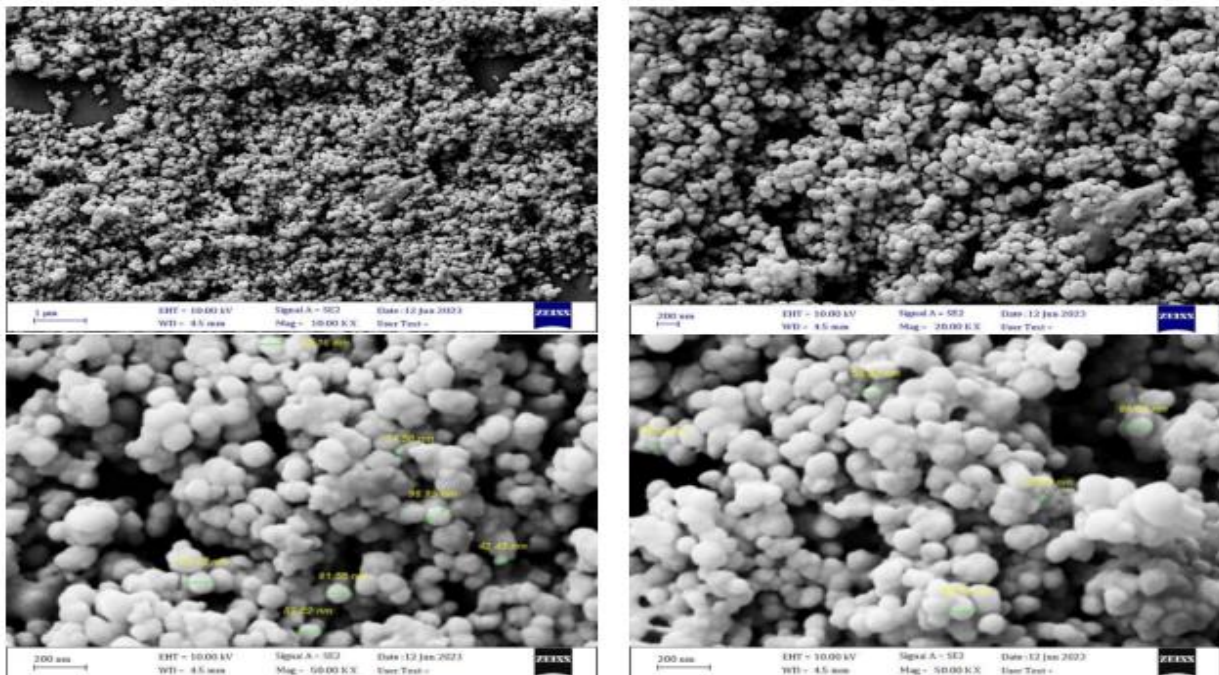


Figure (10): SEM picture of CoFe_2O_4 with 9 pH calcined at 600 °C.

Magnetic characterization of the cobalt ferrite synthesized with 9 pH samples before and after calcination was performed using a vibrating sample magnetometer (VSM) as showed in Figure (11). The discovered magnetic hysteresis at ambient temperature revealed that the

cobalt ferrite nanostructure has ferromagnetic characteristics both before and after calcination. The coercivity H_C of the CoFe_2O_4 nanostructure samples without annealing was 439 Oe, and after calcination for 400 and 600 °C, it increased to 1740 and 1439 Oe respectively. The saturation magnetization (M_S) before calcination was 58 emu/g and after calcination it was 60 and 68 emu/g for 400 and 600 °C respectively. The saturation magnetization values (M_S) are lower than the bulk CoFe_2O_4 (~80 emu/g)[21][46]. The interplay between the magnetic spin moment's surface order and disorder and the disruption to the spinel structural inversion brought on by Laplace pressure may be responsible for this decrease in saturation magnetization[18]. Table 3 illustrates the relationship between calcination temperature and saturation magnetization (M_S) and remanent magnetization (M_R), indicating that both M_S and M_R exhibit an upward trend as the calcination temperature increases. This resulted from the treatment at low temperatures' partial crystallization and tiny particle size. SEM analysis has therefore verified that the particle size grew with increasing calcination temperature. The high number of surface atoms, the presence of unsaturated ligands, and the ability to cause significant thermal disturbances may also play a role. As a result, the crystal's magnetic structure was unstable and disordered, making it difficult to maintain the magnetic moment's consistency with the external field[44]. Additionally, because the amount of the non-magnetic layer on the powder surface dropped as the particle size increased, the saturation magnetization might rise as the particle size does as well [44]. Reduced grain growth is the cause of the rise in coercivity with increased calcining temperature[47].

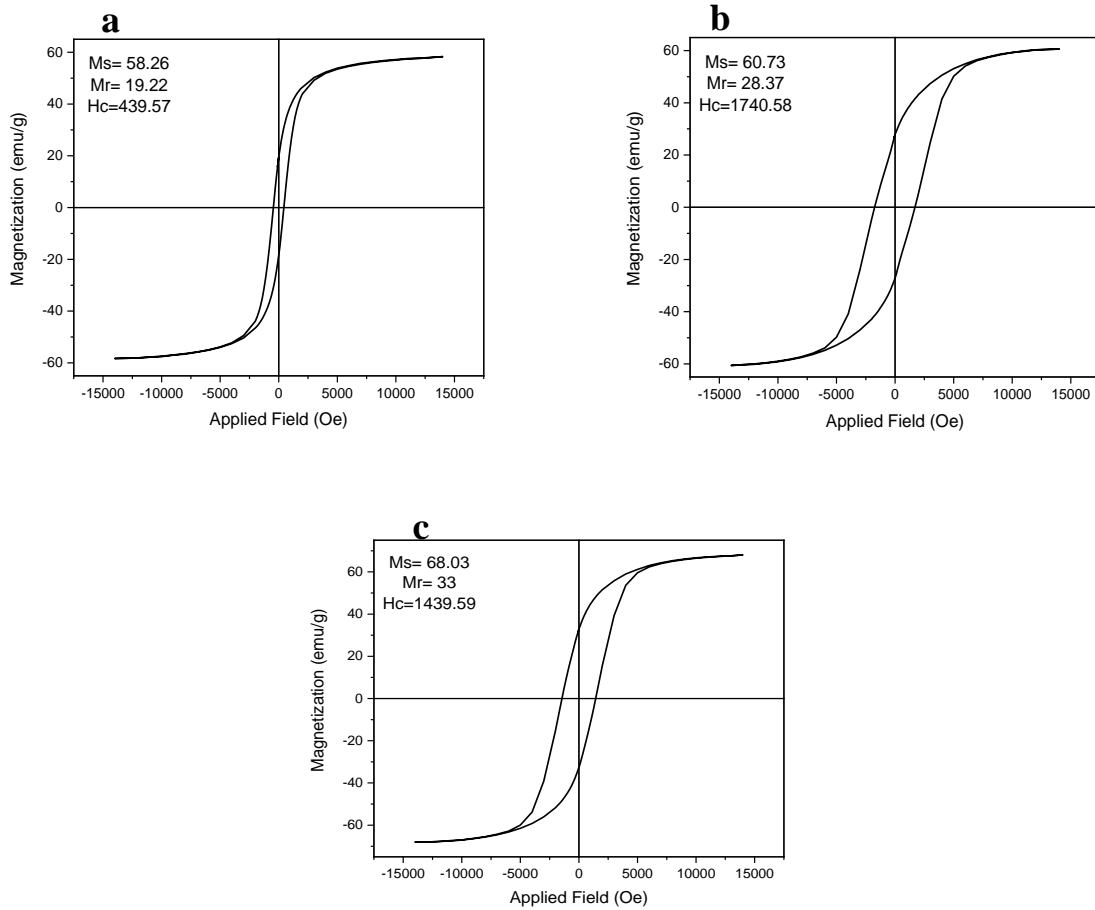


Figure 11: The hysteresis of M–H curve of CoFe₂O₄ with pH is 9 calcined at a: 0 °C, b:400 °C and c:600 °C.

Hysteresis loops were used to calculate the cobalt ferrite's magnetic characteristics. Fig. (12) shows the hysteresis loops of calcinated nanostructure at 0, 400 and 600 °C with 3 pH. Table (3) displays the H_C , M_S , and M_R data with low values. The presence of a spin structure that cancelled the zero magnetization caused by the canted spin order is indicated by a very modest magnetism [48]. Without calcination, the sample's low saturation magnetization value may have resulted from a little amount of Fe–O and O–Fe–O bond formation. However, as the calcination temperature was raised, the bond length and bond angle of the Fe–O and O–Fe–O bonds increased, which led to a drop in the magnetization value [49]. The fact that some grains do not have a spontaneous magnetic moment at higher temperatures means they are unable to contribute to the magnetization, which is another explanation for a drop in the value of magnetic saturation with rising calcination temperature[50] [51]. Due to the thermal variations of the blocked moment across the anisotropy barrier and the impossibility of the moments aligning in an applied field, H_C comparatively reduced with an increase in annealing temperature[21]. Remanent magnetization decreased with increment of calcined temperature due the process of saturation magnetization[52].

Table 3: Magnetic properties of the cobalt ferrite nanostructure.

Sample Name		Ms (emu/g)	Mr (emu/g)	H _C (Oe)
pH	Calcined Temperature °C			
9 pH	0	58.26	19.22	439.57
	400	60.73	28.37	1740.58
	600	68.03	33	1439.59
3 pH	0	0.5	0.108	1225.35
	400	0.38	0.055	914.97
	600	0.37	0.045	610.6

4. Conclusion

CoFe₂O₄ has been prepared through hydrothermal route successfully. After obtaining CoFe₂O₄ powders they were calcined at 400 °C and 500°C. The nanocrystalline nature regarding the synthesized particles is confirmed by XRD data, and it was discovered that the crystallite size decrease at PH=9. Synthesized NPs had a spherical shape and size (23.6 -57.9) nm at PH=9 and (22.3-114.6) nm at PH=3 according to FESEM examination. Magnetic properties of CoFe₂O₄ nanostructure were investigated via VSM analysis. The magnetic saturation, magnetization remnant, and coercivity were all comparatively enhanced with PH=9. The hysteresis curves show normal ferromagnetic behaviour for all the samples.

5. References

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