

Preparation and Studying Magnetic Properties of Cobalt Ferrite (CoFe_2O_4) Material

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Abstract—In this work the Nano-magnetic materials (cobalt ferrite) with formula (CoFe_2O_4) as nano-powder was prepared from (Ferric Nitrate, Cobalt Nitrate and Citric Acid) by utilizing sol-gel combustion method, with different pH (3, 5, and 7) °C, then the powder were calcined at different temperatures (300, 500, and 700) °C, and the samples sintered at different temperatures (1000, and 1100) °C. The magnetic properties (saturation magnetization (M_s), Residual Magnetization (M_r), Remanence Ratio (R), coercivity (H_c) and the Relative permeability (μ_r)) of these samples which formed from this material were studied. The saturation magnetization (M_s), Residual Magnetization (M_r), and Remanence Ratio (R) seem to increase with the increasing calcining and sintering temperatures, at the same pH (7), while the coercivity (H_c) decreases with the decrease in the particle size, because there is a direct relationship between the coercive force and the particle size. The Relative permeability (μ_r) increases with increasing the calcining and sintering temperatures except the samples with pH (5).

Index Terms— Cobalt ferrite, Sol-gel auto-combustion, Spinel ferrite structure, Nano-magnetic material, Magnetic properties, Saturation Magnetization (M_s), Residual Magnetization (M_r), Remanence Ratio (R), Coercivity (H_c), Relative permeability (μ_r), Vibrating Sample Magnetometer (VSM).

1 INTRODUCTION

Nonmagnetic materials are a class of engineered particulate materials of < 100 nm that can be manipulated under the influence of an external magnetic field. They are commonly composed of magnetic elements, such as iron, nickel, cobalt, and their oxides. Like magnetite (Fe_3O_4), are cobalt ferrite (CoFe_2O_4), chromium di-oxide (CrO_2). Nonmagnetic materials display the phenomenon of super-paramagnetism, not keeping magnetizing after the action of magnetic field, offering advantage of reducing risk of particle aggregation [1]. The methods of generation of magnetic nanoparticles in the gas or solid phase using high-energy treatment of the material are usually called physical, while the nanoparticle syntheses, which are often carried out in solutions at moderate temperatures are chemical methods. So that the synthesis methods can be classified into two categories: physical and chemical processes. Several popular methods including co-precipitation, thermal decomposition and/or reduction, micelle synthesis, hydrothermal synthesis, microemulsion, solvothermal, sonochemical, microwave assisted, chemical vapour deposition, combustion synthesis, carbon arc and laser pyrolysis techniques can all be directed at the synthesis of high-quality magnetic nanoparticles [2-5]. They are used in medicine and biotechnology, in ferrofluids, high-density magnetic storage, high-frequency electronics, high-performance permanent magnets, magnetic refrigerants, magnetic cooling systems, as magnetic sensors etc. [2, 5, and 6]. Cobalt ferrite is an attractive material due to its unique optical, electronic, magnetic properties, excellent chemical stability, mechanical hardness, large anisotropy, high coercivity, high Currie temperature and high saturation magnetization [7, 8]. High coercivity gives cobalt ferrite potential in high-capacity

magnetic storage, whereas high magnetic anisotropy forces the particles to relax through the Brownian mechanism, giving them potential applications as sensors [9]. Therefore, it is a promising choice for high density magnetic recording, Ferro fluids technology, bio-molecule separation, biomedical drug delivery, catalysis, magnetic resonance imaging, biocompatible magnetic nano-particles for cancer treatment and magneto-optical devices. It is well known that magnetic properties depend also on the size, morphology and purity of ferrite particles. Therefore, the choice of preparation method plays key role in the observation of fine product with excellent properties; so that there are various methods to obtain cobalt ferrite nanoparticles, such as mechanical, thermal decomposition, sol-gel method, chemical co-precipitation, mechanochemical processing, hydrothermal, sol-gel auto-combustion, and microemulsion method [7].

Normally the magnetization in spinel ferrite arises due to the difference in the magnetic moment of cations which are distributed in the octahedral and tetrahedral interstitial sites.

This directly points to the distribution of the magnetic Fe^{+3} and Co^{+2} ions in the two sublattices along with other factors. The remanence magnitude, (M_r), can be extracted from the hysteresis loop at the inter sections of the loop with the vertical magnetization axis.

The coercivity of a ferro-magnet or ferri-magnet can be reflected by the coercivity field, (H_c), this value refers to the intensity of the magnetic field required to reduce the magnetization of the magnetic sample to zero, after the magnetization of the sample has reached saturation, while the Remanence Ratio (R) $=M_r/M_s$, is an indication of the ease with which the

direction of magnetization reorients to the nearest easy axis magnetization direction after the magnetic field is removed [10,11,12].

The magnetic studies provide important information on the behavior, properties and applications of cobalt ferrite (CoFe_2O_4) such as:

Pham et al. [13]; synthesized Magnetic powder of cobalt ferrite (CoFe_2O_4) using complexometric method in which Ethylene Diamine Tetra Acetic Acid $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8$ (EDTA) acts as a complexing agent, the crystallographic structure, microstructure and magnetic properties of the synthesized powder were characterized by using X-ray Diffraction (XRD), and Vibrating Sample Magnetometry (VSM), they found that the powders calcined at above (850°C) show desired crystallographic structure and good magnetic properties, and the material crystallized in cubic spinel structure is with lattice parameter of about (8.38\AA).

Shun et al. [11]; prepared Cobalt ferrite (CoFe_2O_4) nanopowder by a low-temperature, auto-combustion method. The thermal evolution of the precursor, as well as the microstructure, morphology and magnetic properties of as-synthesized powder were studied. The grains observed in as-burnt powder were proved to be CoFe_2O_4 nanocrystallites with high dispersibility and low agglomeration. Both the saturation magnetization (M_s) and the remnant magnetization (M_r) were found to be highly depending upon the annealing temperature. The highest coercivity (1373 Oe) was achieved by the sample annealed at (400°C).

Zhenfa et al. [10]; prepared CoFe_2O_4 ferrite nanoparticles by a modified chemical co-precipitation route. Structural and magnetic properties were systematically investigated. X-ray diffraction results showed that the sample was in single pure phase. The results of field-emission scanning electronic microscopy show that the grains appear spherical with diameters ranging from (20 to 30) nm. The composition was determined by energy-dispersive spectroscopy with stoichiometry of CoFe_2O_4 . The coercivity of the synthesized CoFe_2O_4 samples was lower than the theoretical values, which could be explained by the mono-domain structure and a transformation from ferromagnetic to superparamagnetic state.

Lunhong et al. [14]; synthesized nano-crystalline cobalt ferrites by a simple, general, one-step sol-gel auto-combustion method. The influence of annealing temperatures on the magnetic properties was investigated. The microstructure was characterized by means of X-Ray Diffractometer (XRD) and Transmission Electron Microscopy (TEM). It was found that the particle size and magnetic properties of the as-prepared ferrite samples showed strong dependence on the annealing temperature. Also they showed the coercivity initially increased and then decreased with increasing annealing temperature whereas the particle size and saturation magnetization continuously increased.

George et al. [15]; synthesized Cobalt ferrite nanoparticles of average size (18) nm by sol-gel method and investigated the magnetic properties. The saturation magnetization value calculated from Vibration Sample Magnetometer (VSM) studies for CoFe_2O_4 is lower than the reported value for the bulk. They showed the magnetization curves demonstrate a trend towards the superparamagnetic behavior of the as-prepared CoFe_2O_4 nanoparticles, also the microwave magnetic parameters showed a decreasing trend with the increase of frequency.

Abedini et al. [7]; compared the magnetic properties of calcined cobalt ferrite at (600°C) for (2 hours) with a heating rate ($10^\circ\text{C}/\text{min}$) formed by nano-crystalline powders by two different methods (co-precipitation and hydrothermal). The structural properties of the produced powders were investigated by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM). The results showed that the formation of cobalt ferrite spinel structures is affected by changing methods. Magnetic hysteresis was measured by using a Vibrating Sample Magnetometer (VSM) in a maximum field of (10) kOe. The saturation of magnetization (M_s), remanence (M_r) and coercivity (H_c) were derived from the hysteresis loops.

Allaiedini et al. [16]; synthesized the magnetic properties of nano-crystalline cobalt ferrite by the hydrothermal method have been investigated. The structural properties of the produced powders were investigated using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The observed XRD pattern confirmed the spinel/cubic structure of the prepared cobalt ferrite. The SEM pictures showed that the simple hydrothermal method produces uniform sphere-shaped nanopowders. Moreover, infrared spectroscopy was used to confirm the formation of cobalt ferrite particles. Magnetic hysteresis was measured using a vibrating sample magnetometer in a maximum field of (10 kOe). The magnetization of the prepared nanoparticles was investigated, and the saturation magnetization (M_s), remanence (M_r), and coercivity (H_c) were derived from the hysteresis loops. The results revealed that the cobalt ferrite nanoparticles synthesized via the simple hydrothermal method exhibits superior magnetic properties.

2 THE EXPERIMENTAL WORK

2.1 Materials

Cobalt (II) nitrate [$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (hexahydrate) 97% purity, India], ferric (III) nitrate [$(\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O})$, 98% purity, India], citric acid [$(\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O})$, 99% purity, Iraq, Baghdad], distilled water and ammonia solution [(NH_3) , India].

2.1 Preparation method

The stoichiometric amounts of cobalt (II) nitrate $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and ferric (III) nitrate $(\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O})$ with ratio 1:2 were dissolved in distilled water under magnetic stirring. Then citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) was dissolved in distilled water under magnetic stirring and it mixed in the metal nitrate solu-

tion to chalet Co^{2+} and Fe^{3+} ions in the solution. A small amount of ammonia was added drop-wise into the solution to stabilize the nitrate-citrate solution, and then the solution was heated to dryness on a hot plate without stirring until it becomes viscous and finally formed a very viscous gel when temperature becomes (85-95) °C. The temperature is further raised up to 115°C so that the ignition of the gel starts. The dried gel burnt completely in a self-propagating combustion manner to form a loose powder, that it occurs during at one hour. Finally the as burnt powders were calcined in a furnace at different temperatures (300, 500,700) °C for 4 hrs with a heating rate of (10°C / min). After that the samples were sintering at different temperatures (1000,1100)°C for (4)hours with raising rate (10 °C/min) by using furnace with type (Nabertherm) , to get the good samples for studying the properties. The samples that used only in permeability test in form small ring with weight (0.5) gm and pressed by force (0.25) Ton to be suitable for this test.

3 MEATUREMENTS

Magnetic properties(Magnetization, Saturation Magnetization (M_s), Residual Magnetization (M_r), Coercivity Force (H_c)and permeability) of powders and samples were done by using vibrating sample magnetometer (VSM) type (CRYOGENIC) in (Middle East University) in Turkey, and in Iran in Kashan university respectively, for powder and samples of cobalt ferrite material that prepared with value of pH (7) , and then calcined , sintered at different temperatures ,and for the powder with values of pH (3,5) , at calcining temperature (700) °C ,which sintered at different temperatures.

4 RESULT AND DISCUSSION

4.1 Hysteresis Loop

To clarify the magnetic properties of CoFe_2O_4 nanopowders, the hysteresis loops of the samples prepared by sol-gel auto-combustion with different pH , which calcined at different temperatures, and sintered at different temperatures , were measured by Vibrating Sample Magnetometer (VSM) at room temperature.

The results of VSM measurements and Magnetization versus the applied field plot at room temperature for the obtained powder are shown in Figs (1, 2, 3, 4, 5, 6) respectively.

It is obvious from these figures that the magnetic properties of CoFe_2O_4 powders show high dependence upon both calcined and sintering temperatures, when the samples were calcined exhibited typical magnetic behaviors, because in the thermal treatment , the heating rate of calcination is one of the most important parameters that can effectively increase or decrease the saturation magnetization , therefore, it is possible that calcination at a slower heating rate would allow the

crystallization to be more complete, and the magnetic phase could also increase, resulting in larger saturation magnetization.

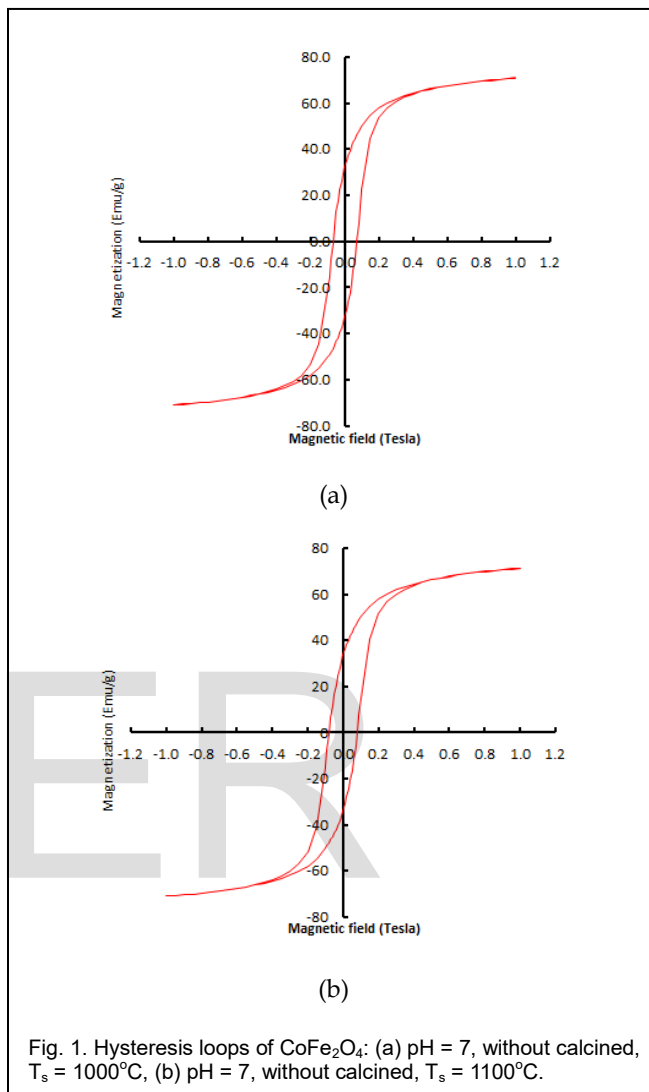
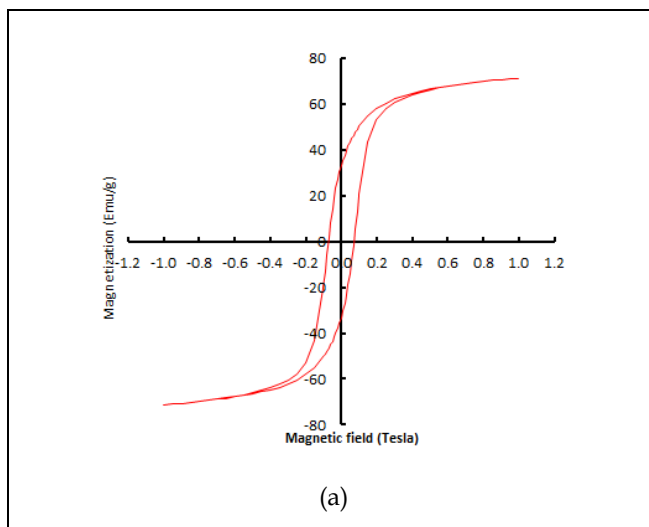
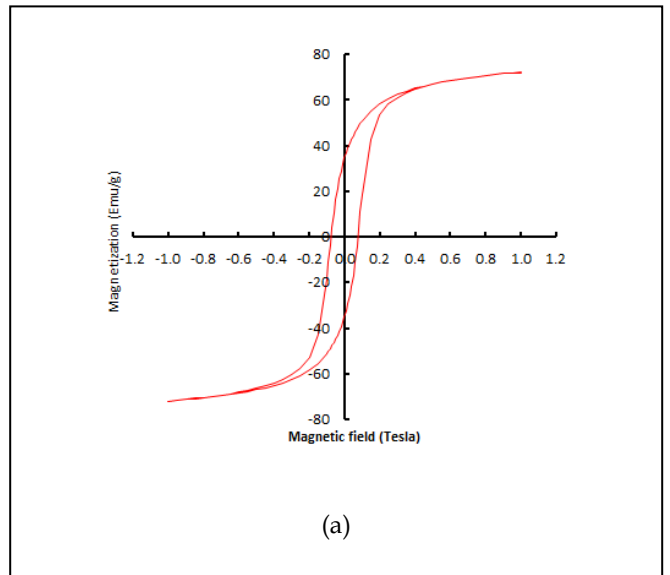
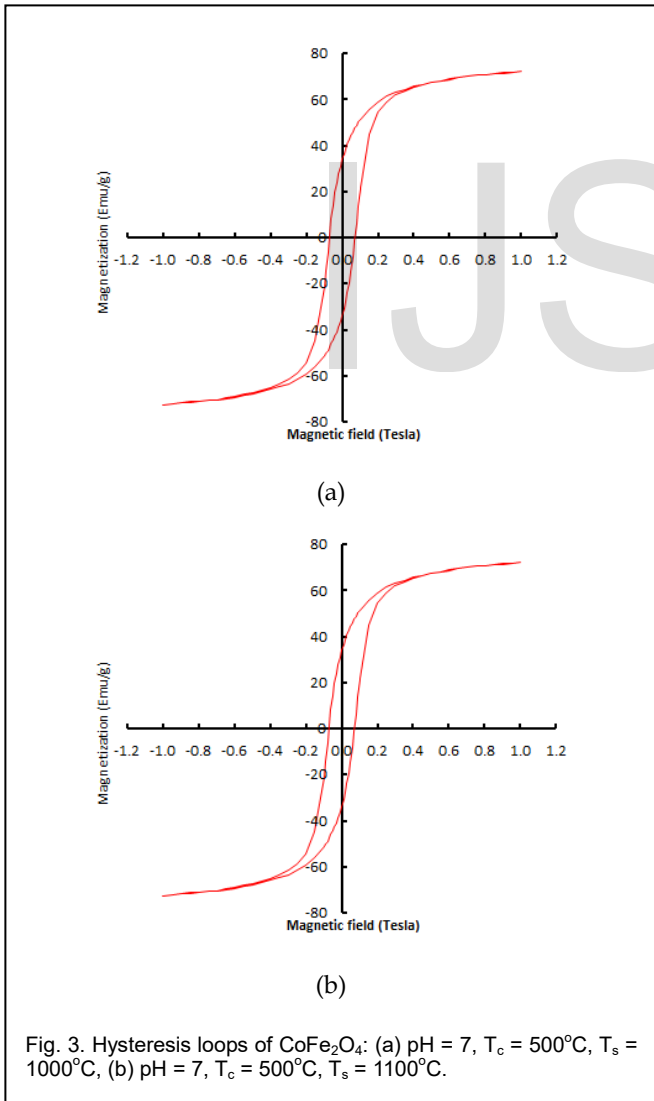
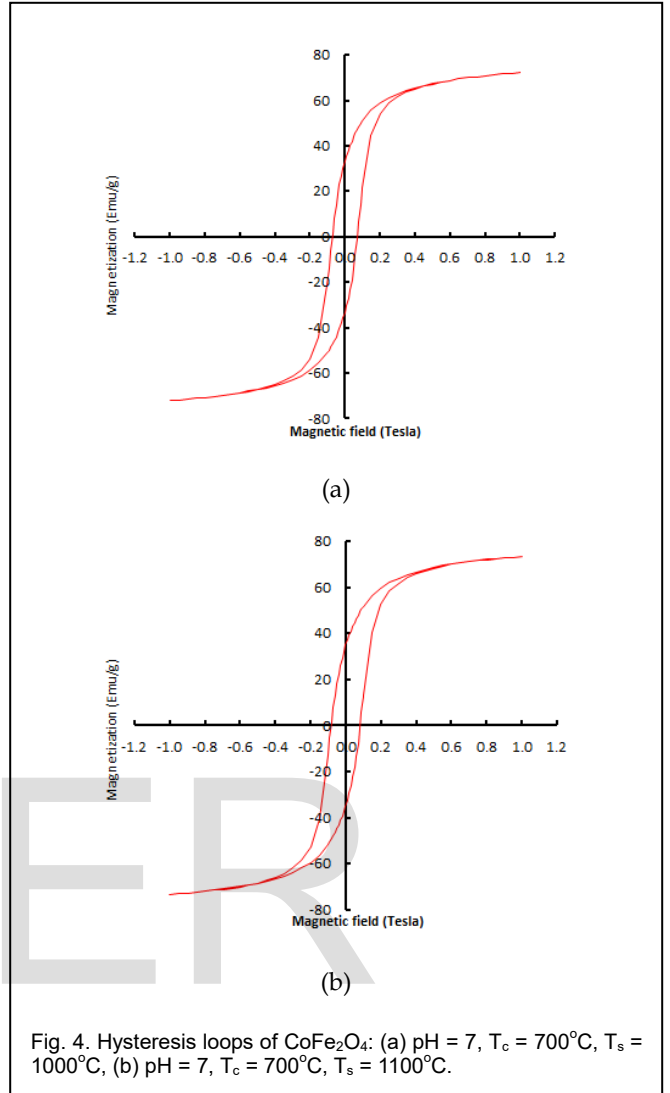
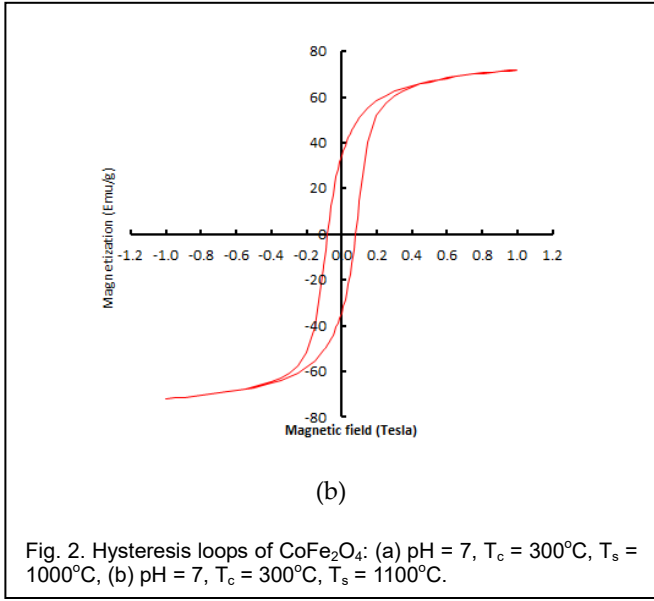
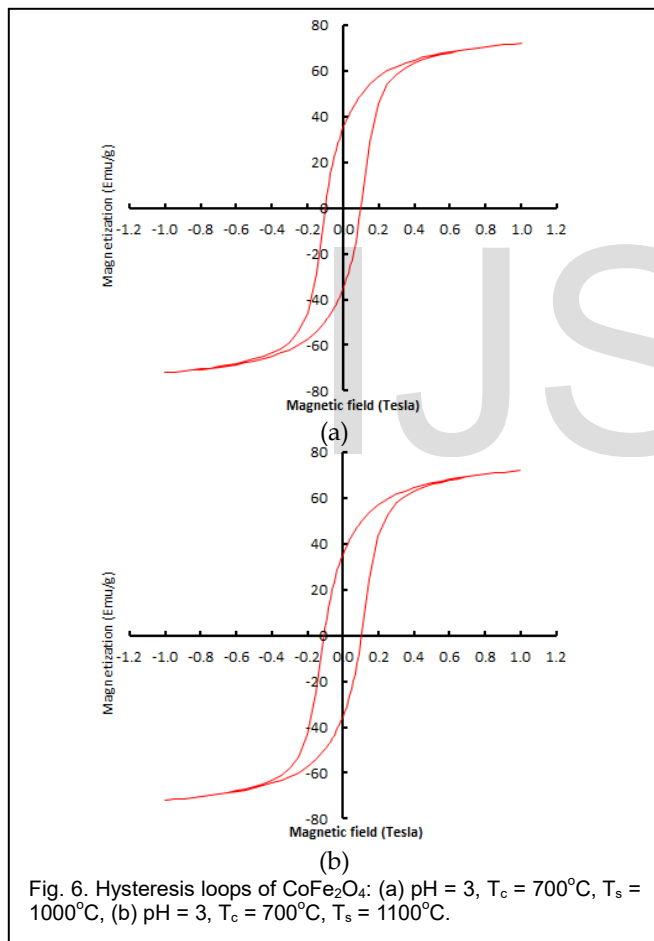
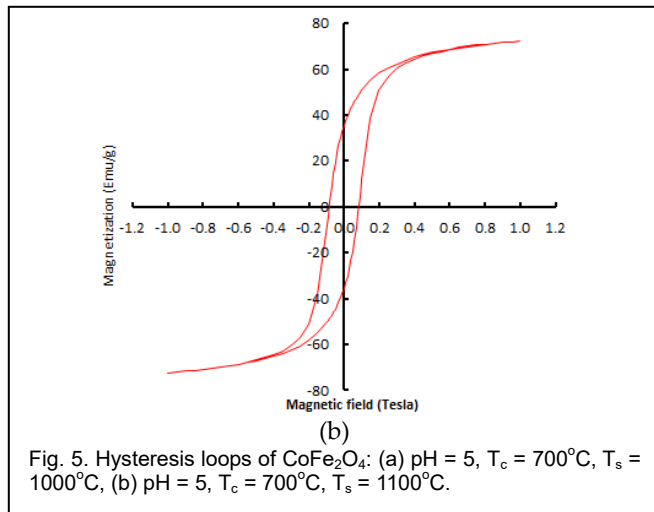


Fig. 1. Hysteresis loops of CoFe_2O_4 : (a) pH = 7, without calcined, $T_s = 1000^\circ\text{C}$, (b) pH = 7, without calcined, $T_s = 1100^\circ\text{C}$.







The saturation magnetization values of the cobalt ferrite samples were found to be lower than the corresponding bulk value of 80.8 emu/g, the low value of saturation magnetization compared with that of the bulk can be understood on the basis of the core-shell model, which explains that the finite size effects of the nanoparticles lead to canting or non-collinearity of spins on their surface, thereby reducing magnetization [14, 16, 17, and 18]. The Saturation Magnetization (M_s), Residual Magnetization (M_r), and Remanence Ratio (R)

increase with the increase in the calcined and sintering temperature, while the Coercivity Force (H_c) decreases with the increase in the calcined and sintering temperature, that is showed in tables (1),(2):

TABLE 1

pH	Temperature (°C)	Saturation Magnetization (M _s) (emu g ⁻¹)	Residual Magnetization (M _r) (emu g ⁻¹)	Coercivity Force (H _c) × 10 ³ (T)	Remanence Ratio (R) =M _r /M _s
7	115	70.7	33.30	10.2	0.466
	300	71.4	33.35	8.6	0.465
	500	71.7	35.35	7.2	0.490
	700	72.4	35.65	7.1	0.492
5	700	72.2	33.95	7.1	0.470
3	700	70.0	32.62	10.4	0.461

The Saturation Magnetization (M_s), Residual Magnetization (M_r), Coercivity Force (H_c) and Remanence Ratio (R) of CoFe₂O₄ nanopowders, which prepared, with different pH, calcined at different temperatures, sintered at temperature (1000°C).

TABLE 2

pH	Temperature (°C)	Saturation Magnetization (M _s) (emu g ⁻¹)	Residual Magnetization (M _r) (emu g ⁻¹)	Coercivity Force (H _c) × 10 ³ (T)	Remanence Ratio (R) =M _r /M _s
7	115	71.3	33.61	8.1	0.471
	300	71.7	33.99	8.0	0.474
	500	72.3	35.05	7.5	0.492
	700	73.3	35.68	7.0	0.494
5	700	71.19	34.40	7.0	0.475
3	700	72.2	35.00	10.2	0.477

The Saturation Magnetization (M_s), Residual Magnetization (M_r), Coercivity Force (H_c) and Remanence Ratio (R) of CoFe₂O₄ nanopowders, which prepared, with different pH, calcined at different temperatures, sintered at temperature (1100°C).

The saturation magnetization (M_s), Residual Magnetization (M_r), and Remanence Ratio (R) seem to increase with the increasing calcining and sintering temperatures, at the same pH (7). This behavior can be attributed to surface effects that are the result of finite-size scaling of nanocrystallites, which in turn lead to a non-collinearity of magnetic moments on their surface and can also be explained by core-shell morphology of the nanoparticles consisting of ferri-magnetically aligned spins core and spin-glass-like surface [16, and 18]. While the coercivity (H_c) decreases with the decrease in the particle size, the coercivity is affected by the factors such as magneto-crystallinity, micro shrinkage, size distribution, anisotropy and the magnetic domain size, also, the (H_c) is closely related to the microstructure, grain size, residual shrinkage and many other complex factors, the decrease in the coercivity with increasing calcining temperature is attributed to the grain growth [19-22]. As the particle size is around the single domain size, the (H_c) will reach a maximum value because of the coherent rotation of spins. Additionally, the saturation magnetization (M_s) is related to coercivity (H_c) through the Brown's relation [13,23,24];

$$H_c = 2K_1 / \mu_0 M_s \quad (1)$$

where, K₁ is the anisotropy constant and μ₀ is the permeability of free space. Also it is observed from above tables and figures

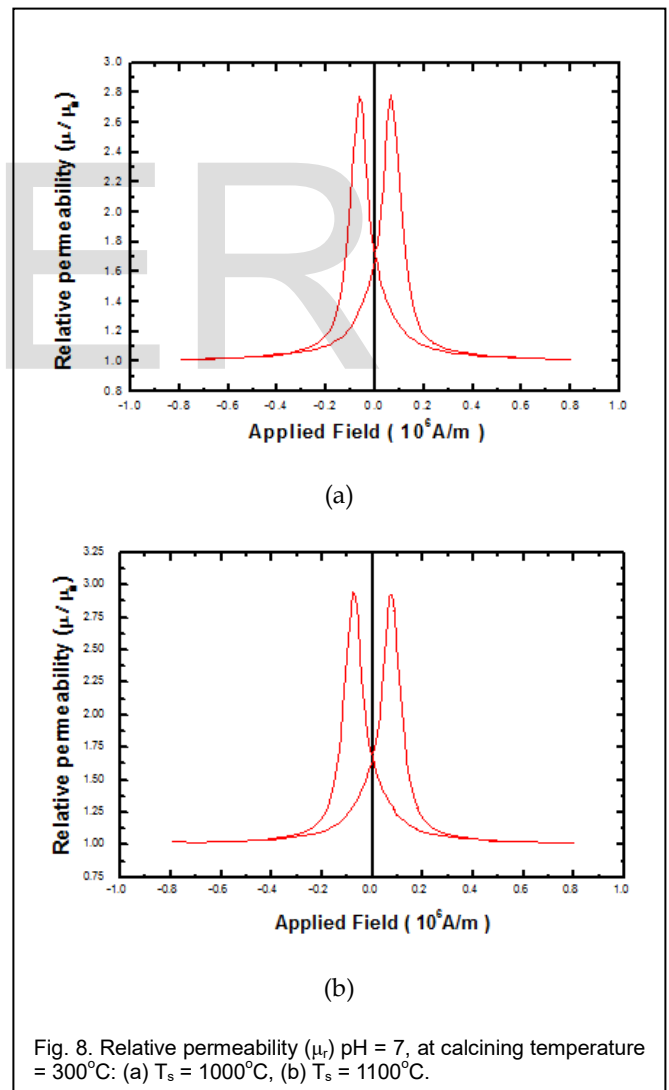
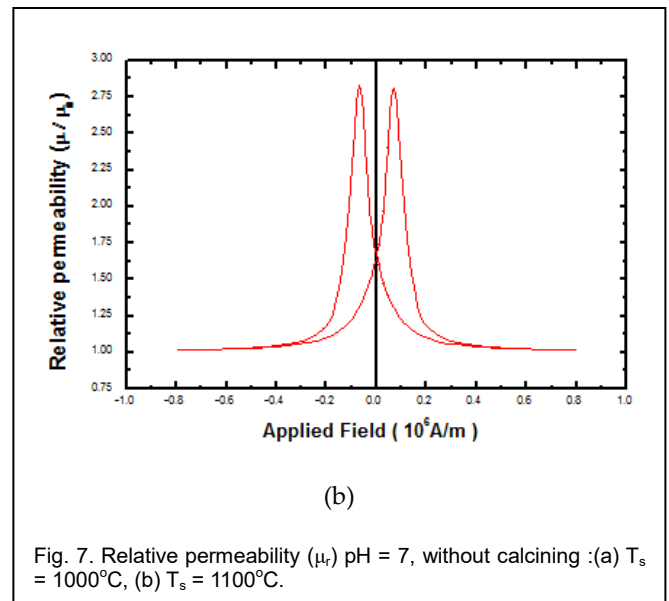
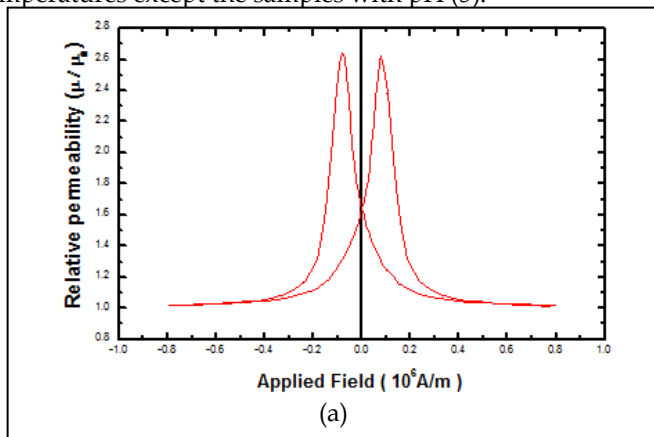
that the values of (M_s , M_r and R) increase when pH increases at the same temperature of the samples, except the behavior of the sample of pH(5), and the low value of R is an indication of the isotropic nature of the material [18,25,26]. The lower (H_c) occurs due to the nano-domain structure and the transformation from ferromagnetic to supermagnetic state.

4.1 Relative Permeability

Permeability (μ) is the measure of the ability of a material to support the formation of a magnetic field within itself. It is measured in henries per meter (H/m or $H \cdot m^{-1}$), or newtons per ampere squared ($N \cdot A^{-2}$). The permeability constant (μ_0), also known as the magnetic constant or the permeability of free space, is a measure of the amount of resistance encountered when forming a magnetic field in a classical vacuum. The magnetic constant has the exact (defined) value ($\mu_0 = 4\pi \times 10^{-7} H \cdot m^{-1}$ or $N \cdot A^{-2}$). Relative permeability (μ_r), sometimes observed by the symbol μ_r , is the ratio of the permeability of a specific medium to the permeability of free space μ_0 :

$$\mu_r = \mu / \mu_0 \quad (2)$$

Relative permeability (μ_r) is a free number without units. When the porosity of ferrite materials decreases the magnetic Permeability increasing. If the samples have smaller and equal grains, the increased density leads to decreasing the porosity, in this case the samples which contain the more magnetic regions, and the area of grain boundary is small, this makes the movement associated with the magnetic regions easier and thus increase the magnetic Permeability. Add to this, the magnetic properties are affected by the porosity and density of the sample, as the increasing porosity adversely affects the promotion (strengthen) of magnetic properties, because they reduce the density of regions of magnetic permeability of lattice for the ferrite body [13,27 and 28]. Figs (7), (8), (9), (10), (11) and (12) show the relative Permeability of samples of $CoFe_2O_4$, prepared by this method, with different pH, which was calcined at different temperatures, sintered at temperatures. Also it showed from these figures that the Relative permeability (μ_r) increases with increasing the calcining and sintering temperatures except the samples with pH (5).



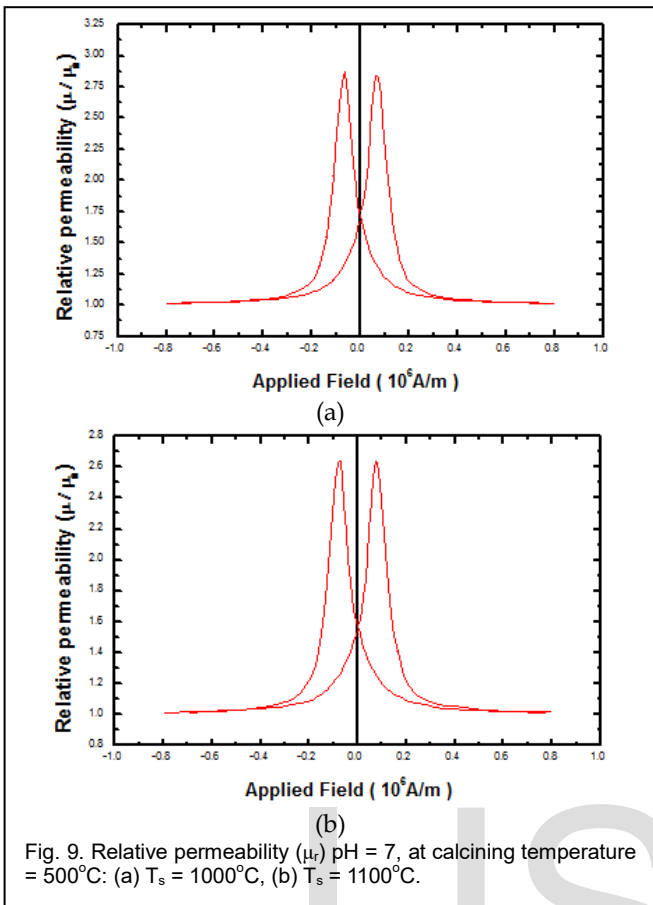


Fig. 9. Relative permeability (μ_r) pH = 7, at calcining temperature = 500°C: (a) $T_s = 1000^\circ\text{C}$, (b) $T_s = 1100^\circ\text{C}$.

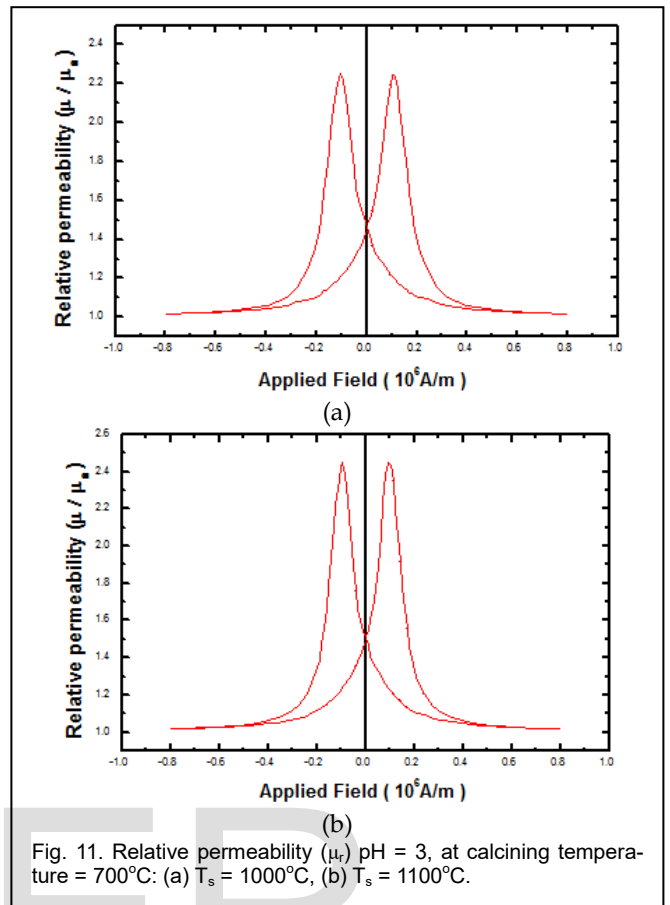


Fig. 11. Relative permeability (μ_r) pH = 3, at calcining temperature = 700°C: (a) $T_s = 1000^\circ\text{C}$, (b) $T_s = 1100^\circ\text{C}$.

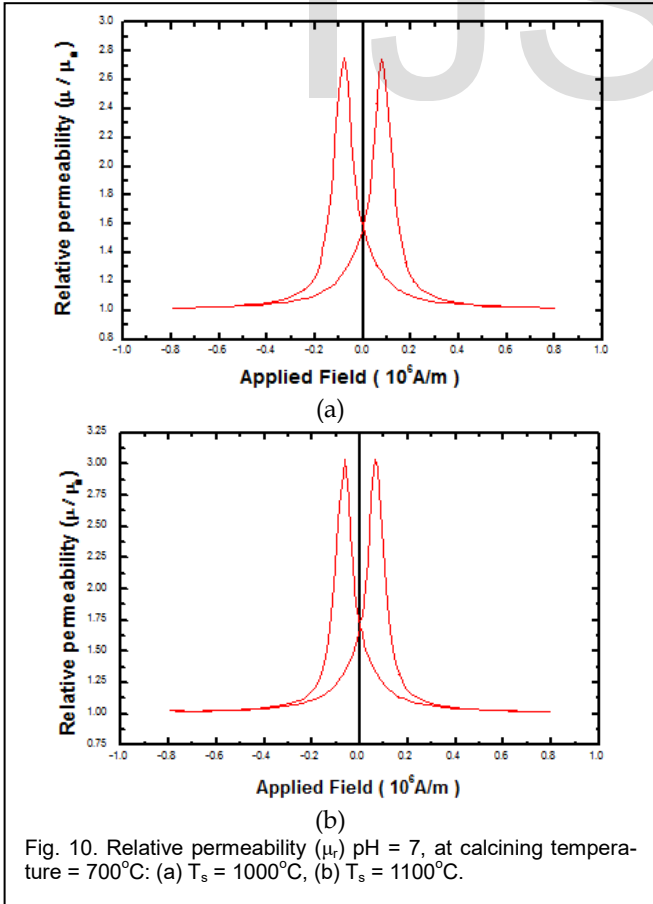


Fig. 10. Relative permeability (μ_r) pH = 7, at calcining temperature = 700°C: (a) $T_s = 1000^\circ\text{C}$, (b) $T_s = 1100^\circ\text{C}$.

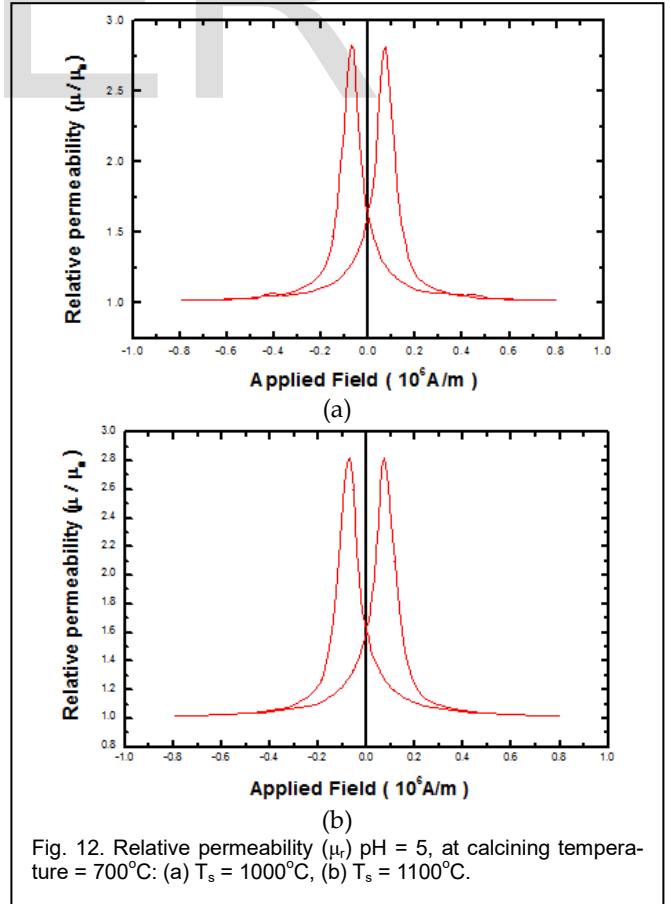


Fig. 12. Relative permeability (μ_r) pH = 5, at calcining temperature = 700°C: (a) $T_s = 1000^\circ\text{C}$, (b) $T_s = 1100^\circ\text{C}$.

5 CONCLUSION

The saturation magnetization (M_s), Residual Magnetization (M_r), and Remanence Ratio (R) seem to increase with the increasing calcining and sintering temperatures, at the same pH(7). The coercivity (H_c) decreases with the decrease in the particle size, because there is a direct relationship between the coercive force and the particle size.

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