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Preparation, Characterization of Cobalt Ferrite Nanoparticls

[1,2] R. A. Hassan, [1]A. A Alharbi, [1]S. H. A. ALLehyani

[1]Department of Physics, Faculty of Applied Sciences, Umm Al Qura University, Makkah, Saudi Arabia
[2]Department of Radiotherapy, National Cancer Institute, Cairo University, Cairo, Egypt

Abstract: **Magnetic nanoparticles (MNPs) possess unique properties which can be applied in nanomedicine: they address targets such as cellular therapy, tissue repair, nanobiosensors, drug delivery, magnetic resonance imaging and magnetic fluid hyperthermia. In the last decades, much research has been devoted to the synthesis of magnetic nanoparticles. Especially during the last few years, many publications have described efficient synthetic routes to shape-controlled, highly stable, and monodisperse magnetic nanoparticles. Most studies suggested that the cobalt ferrites could be a promising materials for various applications. In this work, different sized cobalt ferrite nanoparticls (CFNP) were selectively synthesized with narrow size distribution, by using chemical precipitation methods. The obtained powder was calcined at different temperature (600 °C, 800 °C, 900 °C ,1000 °C). Different characterization like Transmission Electron Microscope (TEM) was done to visualize the morphology and particle size, X-ray Diffraction (XRD) analysis was done to know the phases present, Vibrating Sample Magnetometer (VSM) to get the magnetization and coercivity of the powder, Ultraviolet - Visible Spectroscopy (UV-VIS) and Fourier Transform Infrared Spectroscopy (FTIR) analysis further confirmed the presence of cobalt ferrite nanoparticles. All of the characterization methods confirmed the formation of single-phase CFNP in the range 10–115 nm depending on the annealing temperature.**

Keywords: **Cobalt ferrite nanoparticls, Preparation, Characterization.**

I. INTRODUCTION

Nanotechnology is a science of the future, which began with John Dalton - the father of modern physics. Nanotechnology developing new materials with dimensions on the nano scale called nanoparticles and investigate whether we can directly control matter on the atomic scale[1]. This science has applications in a range of fields such as medicine, electronics, energy production and it satisfies growing demands of society. As the size of nanoparticles is reduced, deviations from bulk magnetic properties appear. The new properties are attributed to surface magnetization effects and to finite-size effects [2].The use of nanoparticle materials provides many unique advantages, due to large volume-surface ratio [3].

Magnetic nanoparticles (as spinel ferrites) offer some attractive possibilities in biomedicine. First, they have controllable sizes ranging from a few nanometres up to tens of nanometres, which places them at dimensions that are smaller than or comparable to those of a cell (10–100μm), a virus (20–450 nm), a protein (5–50 nm) or a gene (2 nm wide and 10–100 nm long). Magnetic nanoparticles of spinel ferrites are of great interest in fundamental science [4].

Ferrites were discovered thousands of years ago. The spinel ferrites are very much important magnetic materials due to their combined electrical and magnetic properties, makes ferrite useful in many technological applications. The basic electrical and magnetic properties of ferrite can be modified so as to suit the desire application [5].

The properties of ferrites have been improved through extensive research. In 1950, ferrites attract worldwide attention because new applications such as microwave devices, electronic media, computer and telephone industry were rapidly expanding [6]. They have potential applications in high-density magnetic recording devices especially those with high coercivity in electronic devices and in medicine. One of the most recent applications studied is the complete

decomposition of CO_2. This decomposition has been significantly improved by developing ultra-fine ferrite particles with high surface area as a catalyst [7].

Ferrites are magnetic ceramics of great importance in the production of electronic components, since they reduce energy losses caused by induced currents acting as electric insulators. Their applications range from simple function device, such as small permanent magnets to sophisticated devices for the electro-electronic industry [8].

Magnetic nanoparticles being subjected to a magnetic AC field may show remarkable heating effects related to losses during, different processes of magnetization reversal in systems of magnetic nanoparticles. There are various theories which explain the reasons for the heating of the MNPs when subjected to an oscillating electromagnetic field. There exist at least four different mechanisms by which magnetic materials can generate heat in an alternating field, which depend in different manners on the applied magnetic AC field strength and frequency. The Magnetic losses to be utilized for heating arise [9,10].

If a magnetic sample is placed in a liquid of low viscosity and exposed to an oscillating field, the sample reacts with oscillating or rotating movements, as a whole (because of the torque exerted on the magnetic moment by the external AC magnetic field), towards the field with the moment locked along the crystal axis under the effect of a thermal force against a viscous drag in a suspending medium, in order to achieve the position of lowest energy, which lead to losses in the surrounding liquid [11,10,12].

The physical and chemical properties of spinel nano-particles are greatly affected by the synthesis route. Therefore a large number of research reports are available concerning the preparation techniques. Quality ferrites continued to be prepared having new properties [6, 7].

Among the various methods for producing nanoparticles, precipitation method are highly interesting, have been widely used, for their versatility, due to their straightforward nature and their potential to produce large quantities of the final product, low temperature for preparation, relatively simple and providing good control over particle properties. Realizable particle sizes range in size from nanometers to micrometers [13,14,15].

Most studies suggested that the cobalt ferrites ($CoFe_2O_4$) could be a promising materials for various applications [16].

$CoFe_2O_4$ nana -particles are of interest because of their unique optical, electronic and magnetic, physical properties such as high Curie temperature, large magnetocrystalline anisotropy, large magnetostrictive coefficient, excellent chemical stability. (Metal & alloys unstable under atmospheric conditions); good thermal stability; high electrical resistivity (high frequency devices, memory cores, recording media), and mechanical hardness, also for its catalytic properties. This kind of ferrite is a spinel (cubic spinel structure) In addition to the precise control on the composition and structure of $CoFe_2O_4$ [17,7,8,15,18,19].

MNP would be of a great advantage to the treatment of human illnesses [20,21]. Magnetic composites can also be used in magnetic bioseparation [22,23], drug delivery [24,25], and hyperthermia [26,27,28] applications.

II. MATERIAL AND METHOD

Cobalt ferrite nanoparticls (CFNP) were selectively synthesized with narrow size distribution, by using chemical precipitation methods, Cobalt nitrate hexhydrate $(Co(NO_3)_2.6H_2O)$, ferric nitrate nonahydrate $(Fe(NO_3)_3.9H_2O)$, was purchased from Sigma-Aldrich Company, and sodium hydroxide (NaOH) was used as the precipitating agents, and deionized water as solvent.

In order to obtain 6 gm weight of $CoFe_2O_4$, an amount of 7.4 g of $Co(NO_3)_2$ was dissolved in 50 ml of distilled H_2O. The solution was mixed and stirred at constant temperature (80 ^0C) in shaking water bath; using a stirring rate of 120 rpm for 15 minute.

An amount of 20.6 g of $Fe(NO_3)_3$ was dissolved in 50 ml of distilled H_2O.

The solution was mixed and stirred at constant temperature (80 ^0C) in shaking water bath; using a stirring rate of 120 rpm for 15 minute. And after that, $Co(NO_3)_2$ solution was mixed with the $Fe(NO_3)_3$ solution at constant temperatures (80 ^0C) in a shaking water bath using a stirring rate of 120 rpm for 30 minute.

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A 10 g of *NaOH* was dissolved in 1/4 liter of distilled H_2O and then added stepwise to the reaction mixture.

When the reaction was completed, precipitate was shown at the bottom of the reaction mixture, and then , 4 or 5 drops of oleic acid solution as a coating agent was mixed with the reaction mixture at constant temperatures (80 ^{0}C) in a shaking water bath using a stirring rate of 120 rpm, which was kept at least for 12 hours. The precipitate separated from the solution, and it was washed for several times with distilled H_2O and ethyl ether, finally the precipitate was dried at 100 ^{0}C for 12 hours.

After this stage, the sample is divided into 4 samples A, B, C, D. The weight of all samples were made similar, the samples were heated at 600 ^{0}C , 800 ^{0}C , 900 ^{0}C , 1000 ^{0}C respectively for 12 hours. Each of these samples is divided into 5 samples for characterization.

Characterization:

Different techniques are available for the characterization of nanomaterials designed with specific properties and applications as requirements. It is necessary a complex analytical system capable to elucidate the mechanism of the reaction, to determine the composition and the properties of the substances. They are: Transmission Electron Microscopy (TEM), X-Ray Diffraction (XRD), FT-IR analyses**,** was done in the King Abdulaziz University, UV-VIS absorbance spectroscopy was done in physics department- Umm Al-Qura University-KSA, and Vibrating sample magnetometer (VSM) was done at national research center Egypt**.** These methods aim at determining the crystal structure, defect structure, chemical analysis, phase identification, crystal or grain size, magnetic properties etc. [1,55]

III. RESULT AND DISCUSSION

1. UV-VIS Spectroscopy Results:

The UV-VIS spectrums Figures 1,2,3,4 show that the cobalt ferrite powder is identified by the charge transfer band at γ_{max} =372, 372, 304 and 300 nm for samples A, B, C and D respectively, where range peak of cobalt ferrite form 300 nm to 372 nm.

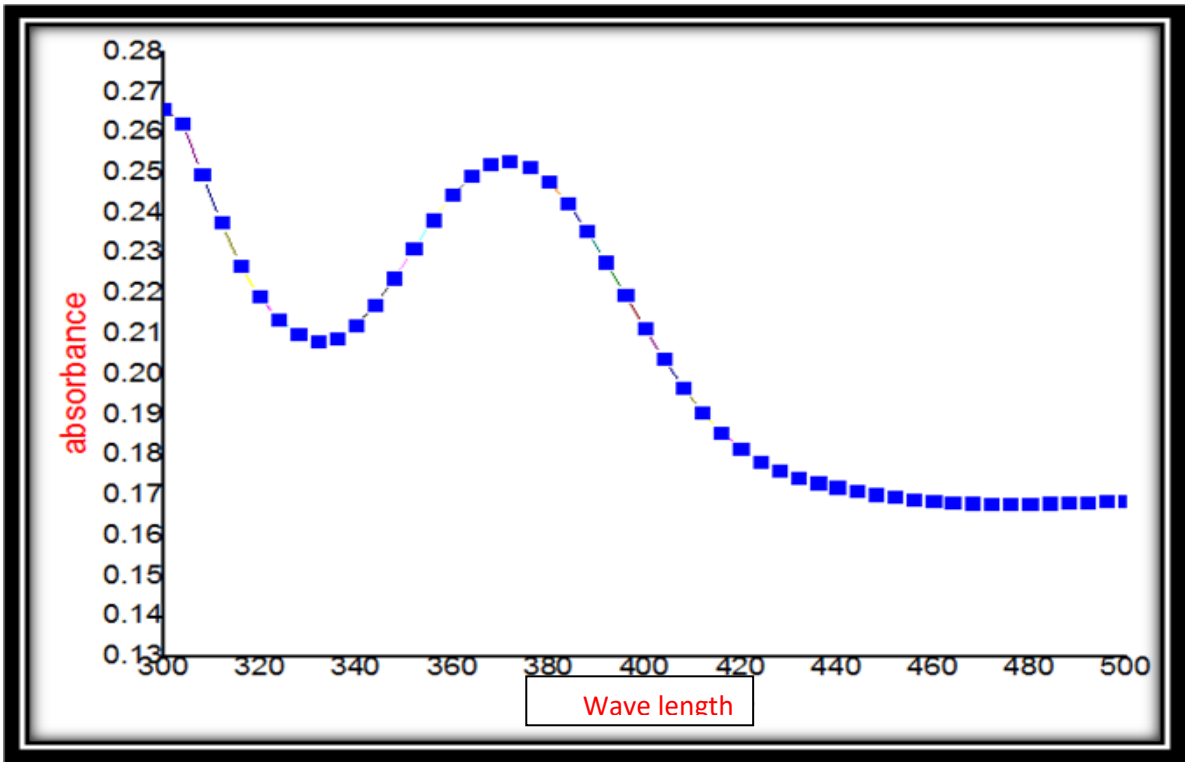

Figure 1: UV-VIS spectrum of $CoFe_2O_4$ powder at 600 ^{0}C

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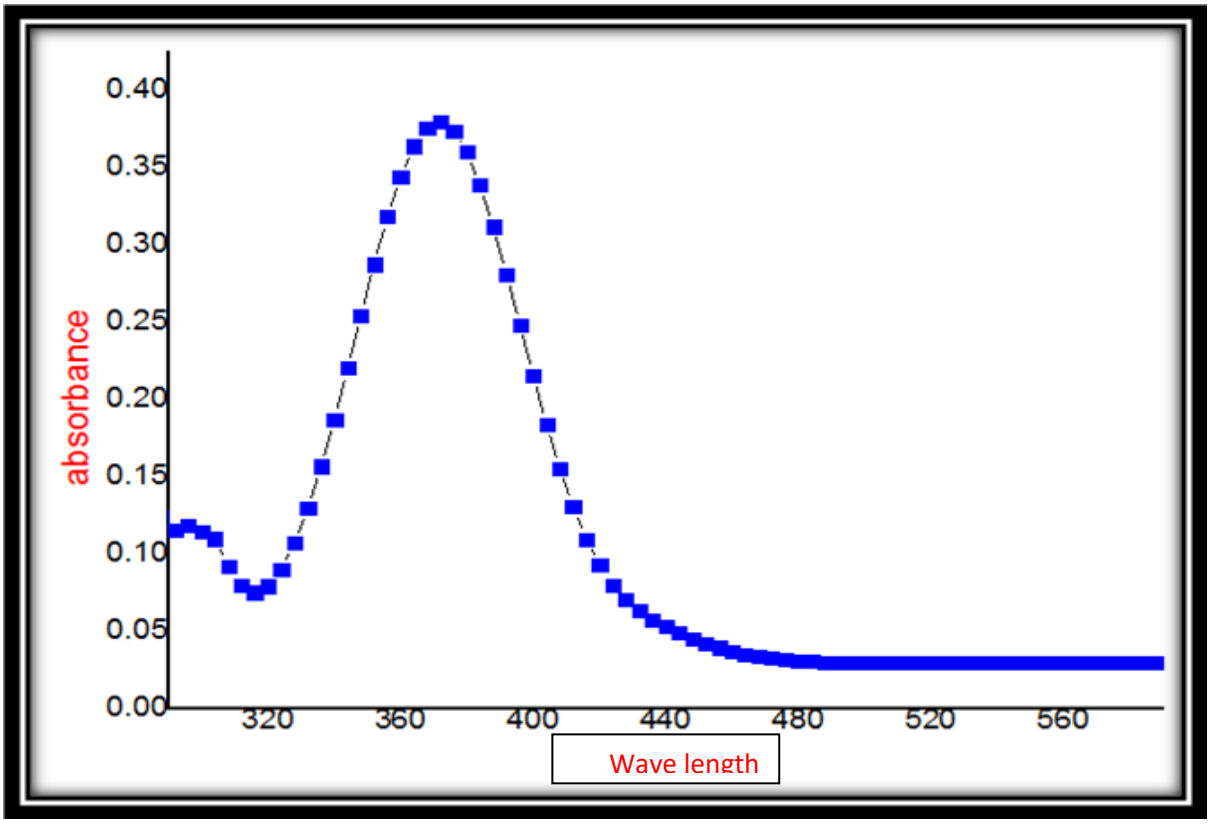

Figure 2: UV-VIS spectrum of $CoFe_2O_4$ powder at 800 ^0C

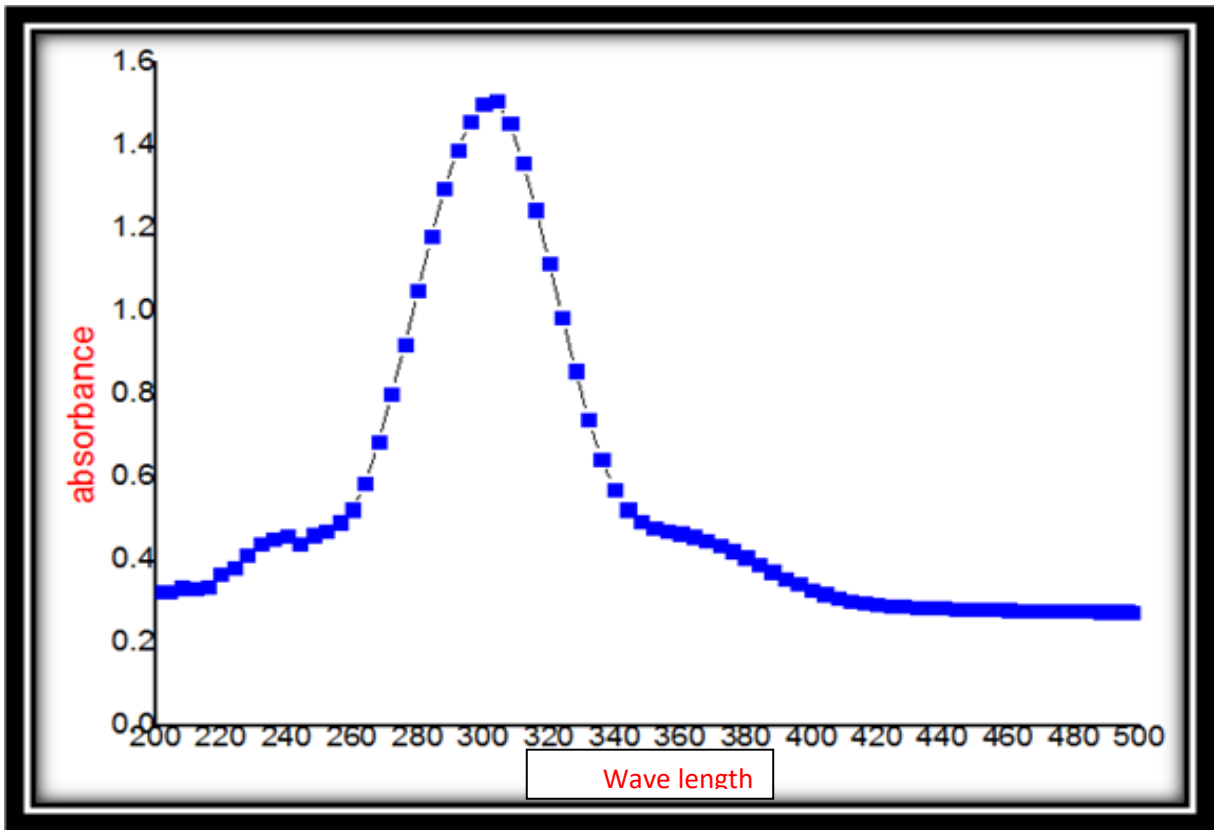

Figure 3: UV-VIS spectrum of $CoFe_2O_4$ powder at 900 ^0C

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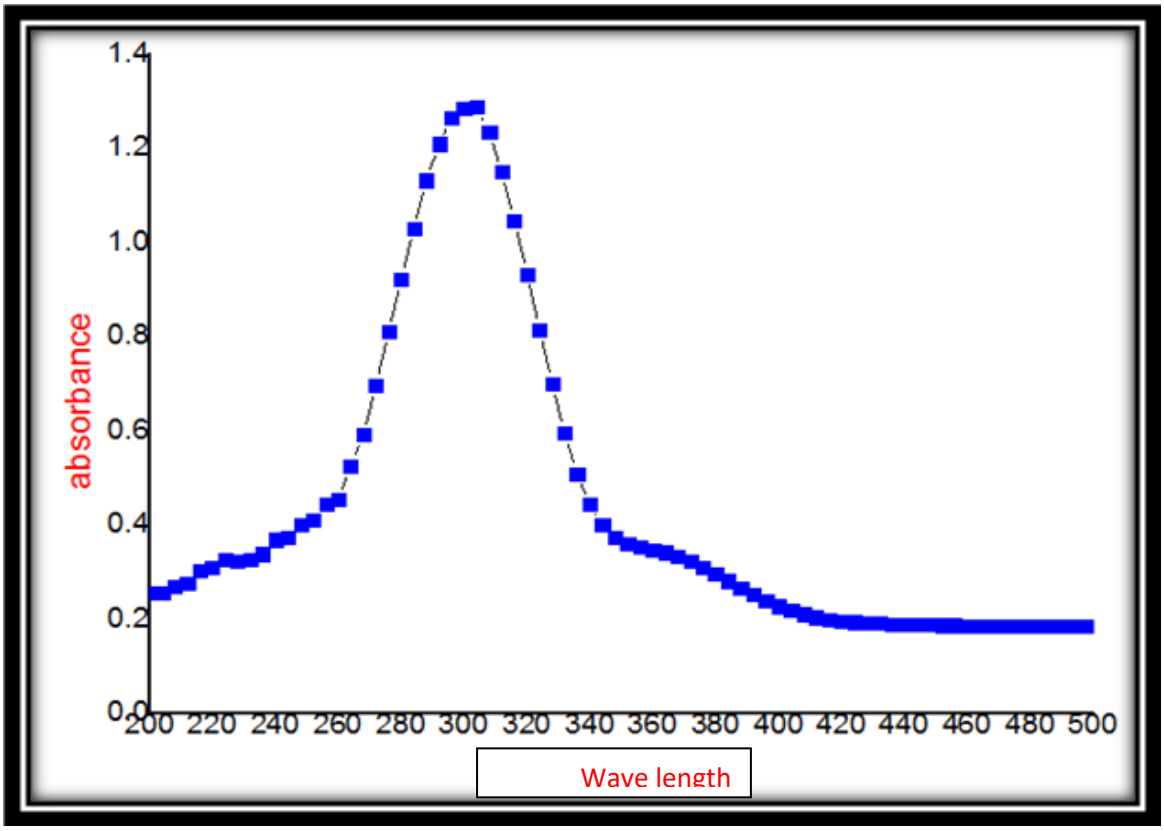

Figure 4: UV-VIS spectrum of $CoFe_2O_4$ powder at 1000 ^0C

2. Transmission Electron Microscope Results:

TEM which we used to analysis cobalt ferrite synthesized by co-precipitation, in order to determine shape and average size of particle.

TEM images display different morphologies and size of cobalt ferrite nanoparticles, where calcination at various temperature (600,800,900,1000 ºC) respectively for 12 hours.

In Figure 5.A&B the substance involves dispersion of nearly spherical particles with narrow size distribution, which spurring their natural of nanoparticle. The size of particle as shown by TEM is including with mean size distribution of 10 to 25 nm.

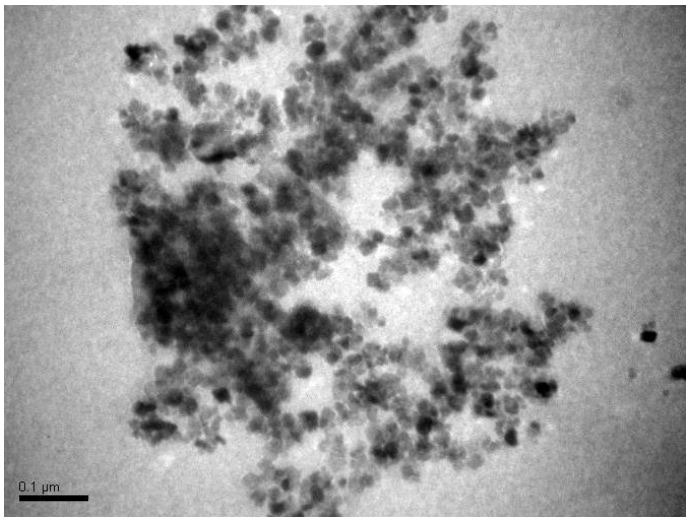

Figure 5.A: TEM micrograph of nanosize $CoFe_2O_4$ particle with an average particle size of 10-25 nm at 600 ^0C

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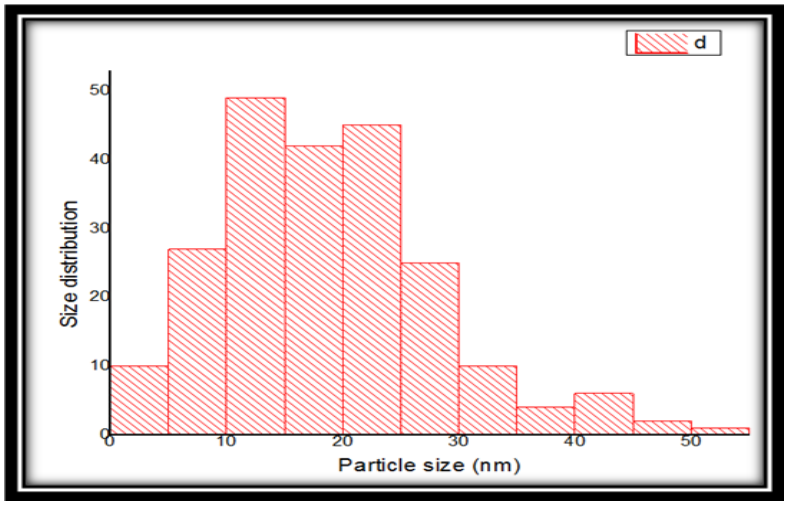

Figure 5.B: Size distribution of nanosize $CoFe_2O_4$ particle with an average particle size of 10-25 nm at 600 ^0C

Figure 6 A&B Shown the existence of large few agglomerates including hundreds of particles. The mean of particle size is about 15-35 nm, as evident in this figure sample involves dispersion of nearly spherical particles.

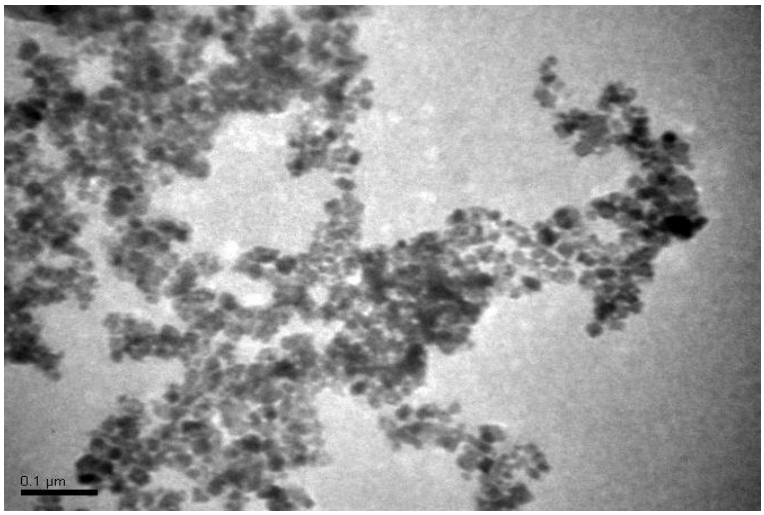

Figure 6.A: TEM micrograph of nanosize $CoFe_2O_4$ particle with an average particle size of 15-35 nm at 800 ^0C .

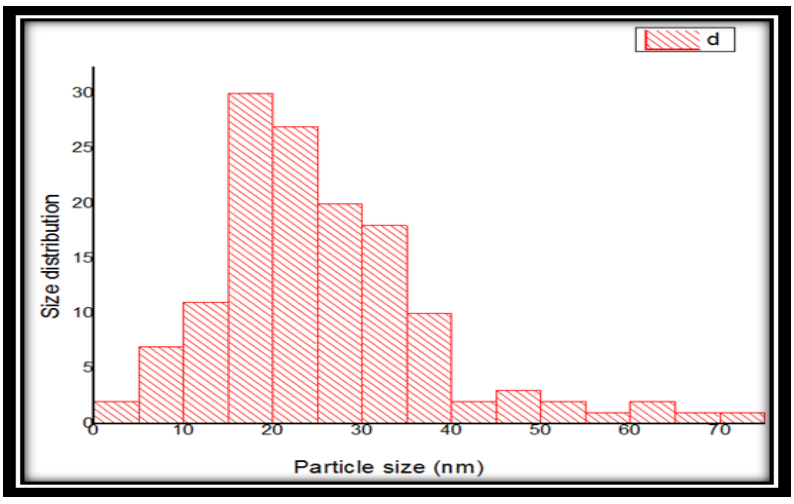

Figure 6.B: Size distribution of nanosize $CoFe_2O_4$ particle with an average particle size of 15-35 nm at 800 ^0C

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Figure 7: Shown the existence of large agglomerates including hundreds of particles. The mean of particle size is about 20-100 nm. As evident in this figure sample involves dispersion of nearly spherical particles.

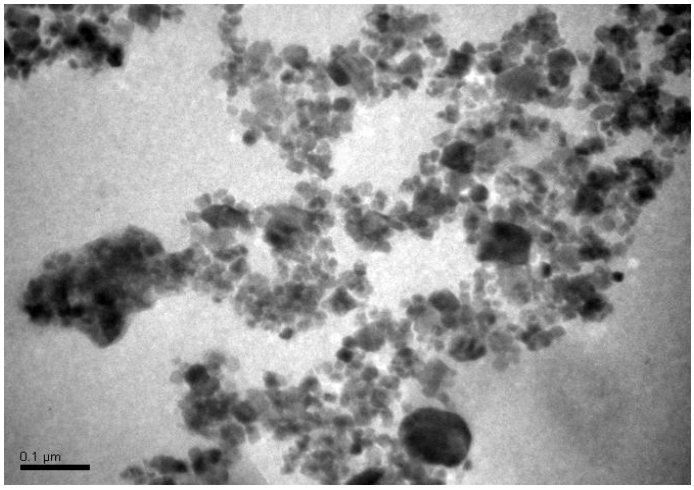

Figure 7.A: TEM micrograph of nanosize $CoFe_2O_4$ particle with an average particle size of 20-100 nm at 900 ^0C

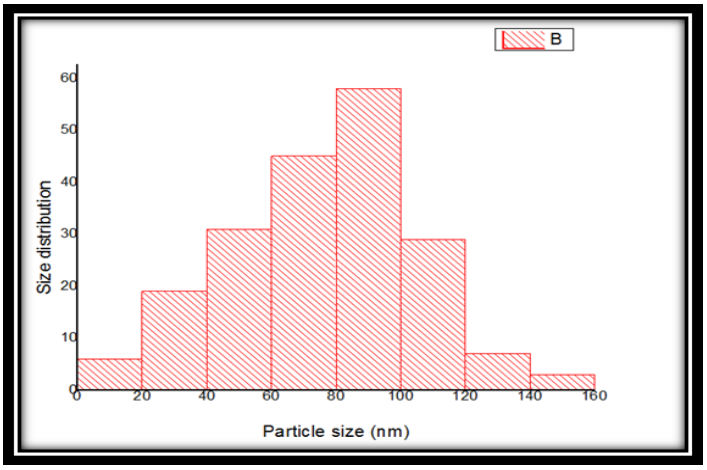

Figure 7.B: Size distribution of nanosize $CoFe_2O_4$ particle with an average particle size of 20-100 nm at 900 ^0C

Figure 8: As evident the existence of largest agglomerates including hundreds of particles. The mean of particle size is about 75-115 nm. There are majority of particles appears spherical in the shape although some elongated particles are also existence.

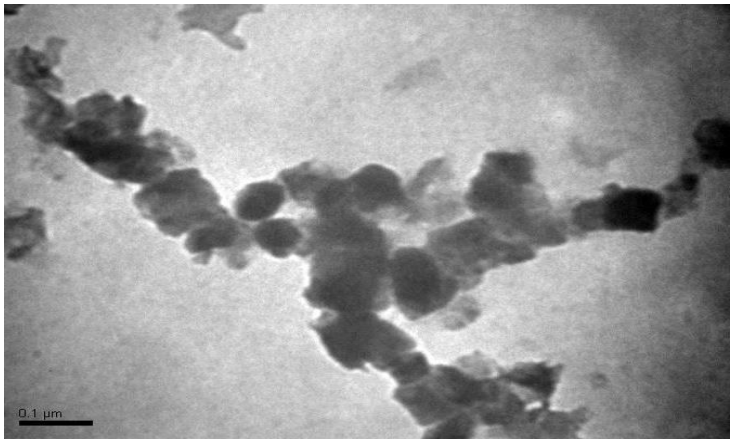

Figure 8.A: TEM micrograph of nano size $CoFe_2O_4$ particle with an average particle size of 75-115 nm at 1000 ^0C

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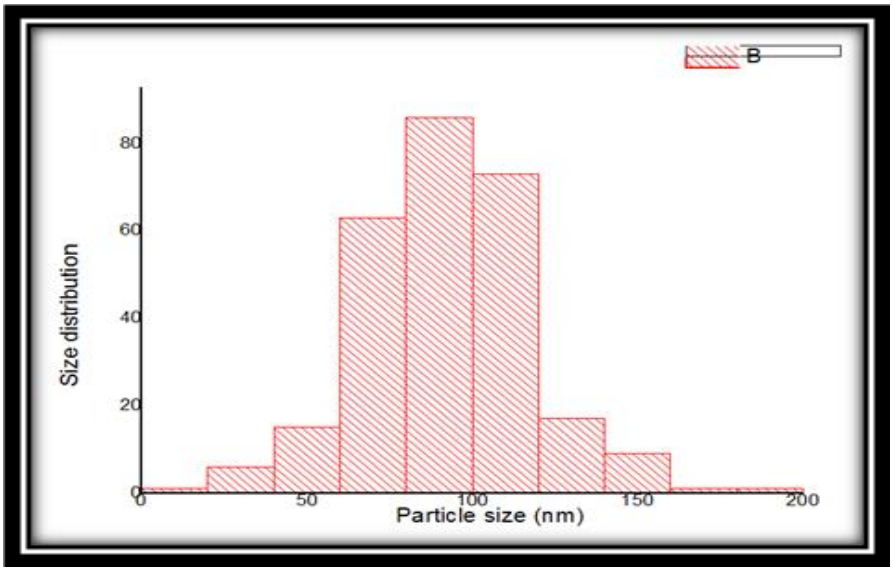

Figure 8.B: size distribution of nanosize $CoFe_2O_4$ particle with an average particle size of 75-115 nm at 1000 ^0C

In general particle size increment with increment temperature of reaction due to ratio of reaction is increment, where these magnetic nanoparticles tend to agglomeration towards each other because magnetic dipole-dipole interaction between particles.

3. Vibrating Sample Magnetometer (VSM) Results:

Vibrating sample magnetometer is used to reorganization magnetic properties of the cobalt ferrite were measured at room temperature.

We can determine saturation magnetization, M_S, remnant magnetization, M_r and coercivity H_C, From obtained hysteresis loops.

In figure 9 the saturation magnetization value of cobalt ferrite M_s, where sample calcination at 600 ^0C is measured about 42.38 emu/g, and remanace magnetization value M_R for this sample are 12.41 emu/g.

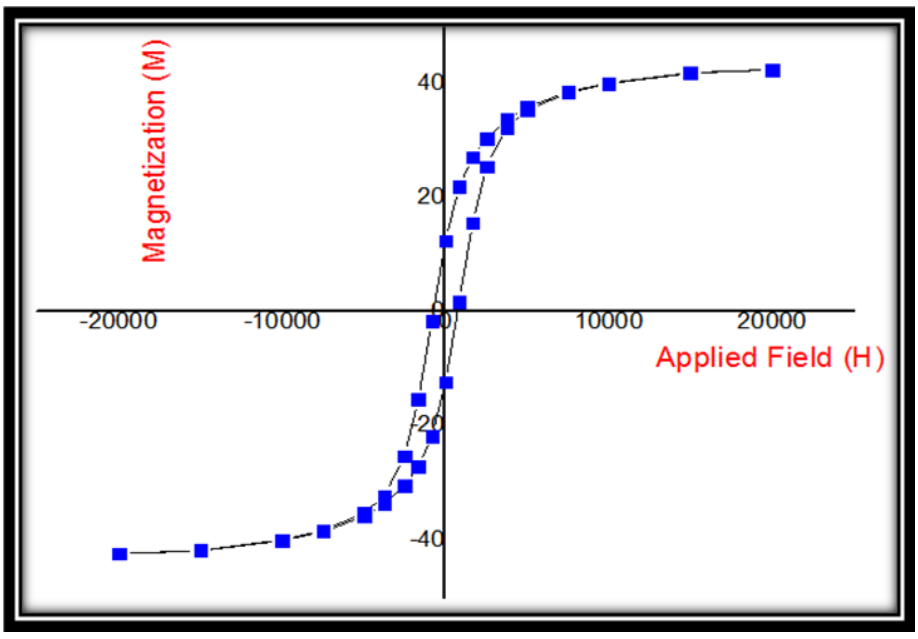

Figure 9: hysteresis loop of cobalt ferrite which calcinated at 600 ^0C

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Also in figure 10 we find the saturation magnetization value of cobalt ferrite M_s ,where sample calcination at 800 ^0C is measured about 26.03 emu/g, and remanace magnetization value M_R for this sample are 8.33 emu/g.

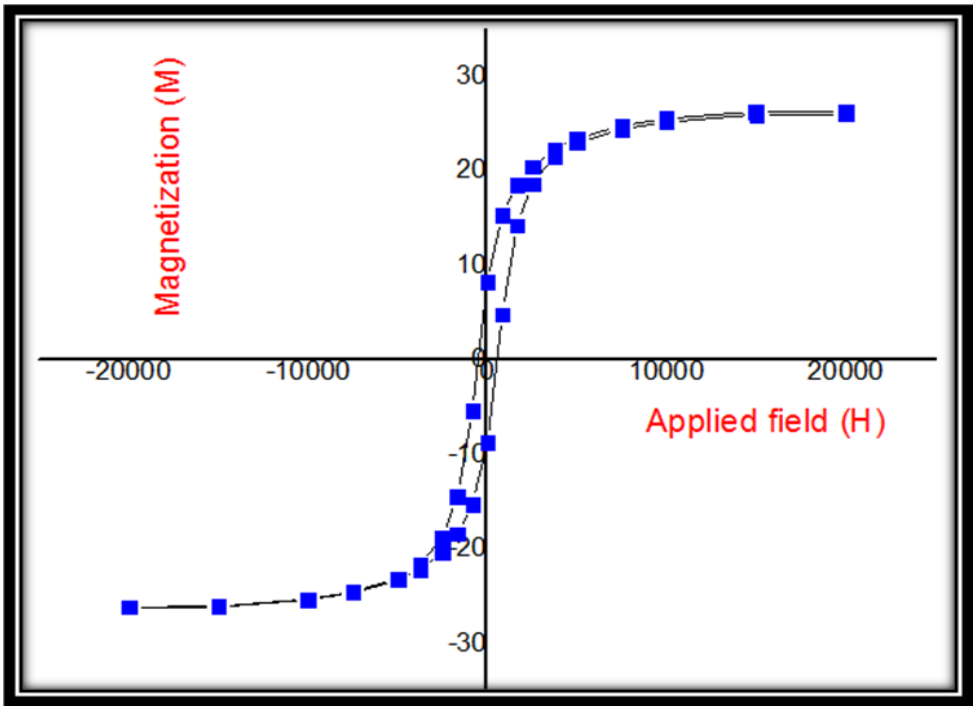

Figure 10: hysteresis loop of cobalt ferrite which calcinated at 800 ^0C

while in the figure 11: we find the saturation magnetization value of cobalt ferrite M_s ,where sample calcination at 900 ^0C is measured about 25.6 emu/g, and remanace magnetization value M_R for this sample are 4.70 emu/g.

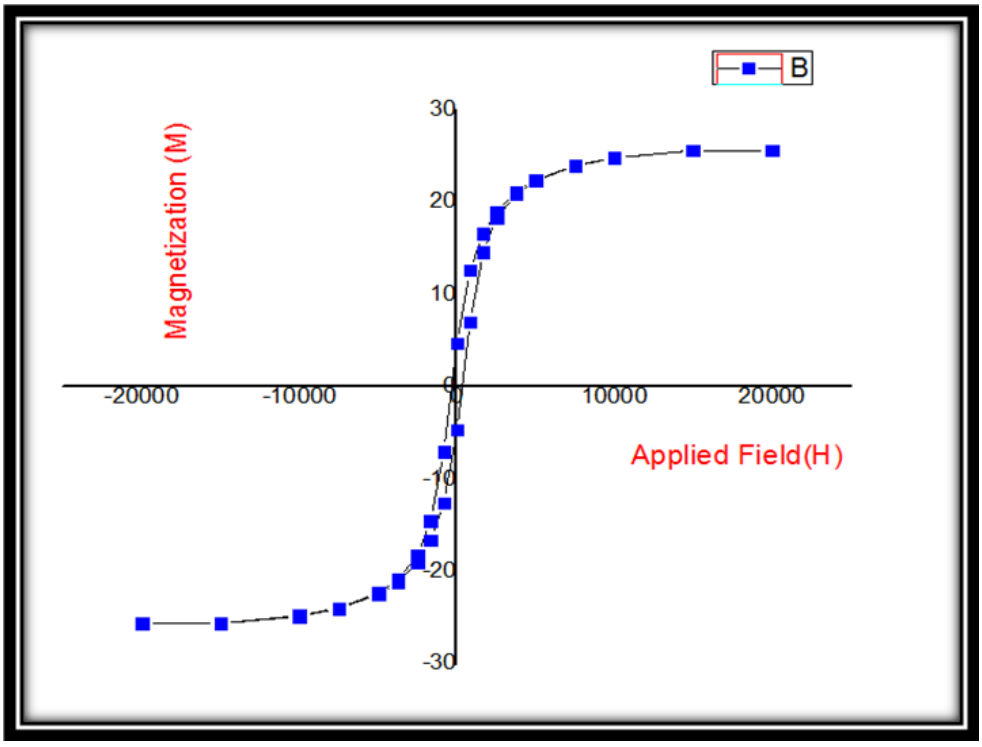

Figure 11: hysteresis loop of cobalt ferrite which calcinated at 900 ^0C

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In the figure 12 we find the saturation magnetization value of cobalt ferrite M_s ,where sample calcination at 1000 ^0C is measured about 18.06 emu/g, and remanace magnetization value M_R for this sample are 2.45 emu/g.

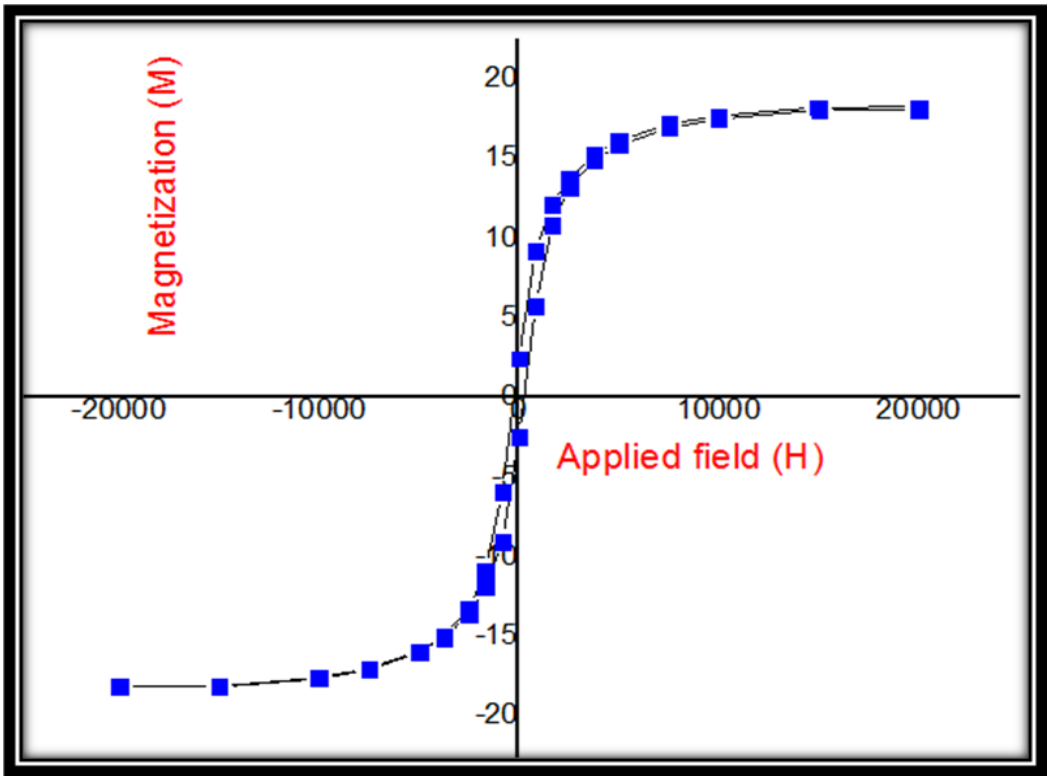

Figure 12: hysteresis loop of cobalt ferrite which calcinated at 1000 ^0C

The size and shape of the hysteresis loops for cobalt ferrite nanoparticles elucidate a kind of thick S-shape. The thickness of the S-shape characterization the amount of hysteresis or the coercivity of the substance where the value of H_C for all sample in curves (A), (B), (C) and (D) are 833.48 O_e, 833.4 O_e, 7.026 O_e and 5.7 O_e, respectively.

The decrease in the coercivity of cobalt ferrite may attribute to magnetic interactions between the $CoFe_2O_4$ nanoparticles.

From this figures we can observed only as particle size reduce, slight increases of the magnetization will be occur.

4. Fourier Transformed Infrared Spectroscopy (FT-IR) Results:

The FT-IR spectra of transmittance versus wave number for samples that is prepared by co-precipitation at temperature 600 C^0, 800 C^0, and 900 are presented in figures below.

In the figure 13, the presence of the OH band (3448 nm) in this spectrum refers to the presence of water, which is reasonable since the functionalization was performed in aqueous solutions as mentioned in the experimental section. However, in the case of the presence of water in the cobalt ferrite, it indicate that the powder were not dry enough. Moreover, the characteristic absorption band of Co (Fe)-O can be seen at 611.324 nm.

Due to the cobalt ferrite is coated with oleic acid, the bands at 1665.5 and 1569.7 nm are related to the vibration of C-H. The band Co-OH is indicated by the vibration at 460.90 nm. The band at 1626 nm corresponds to H-O-H absorption, and that at 846.597 nm is characteristic of the FeOOH. These results clearly indicate the functionalization of the cobalt ferrite nanoparticles with oleic acid.

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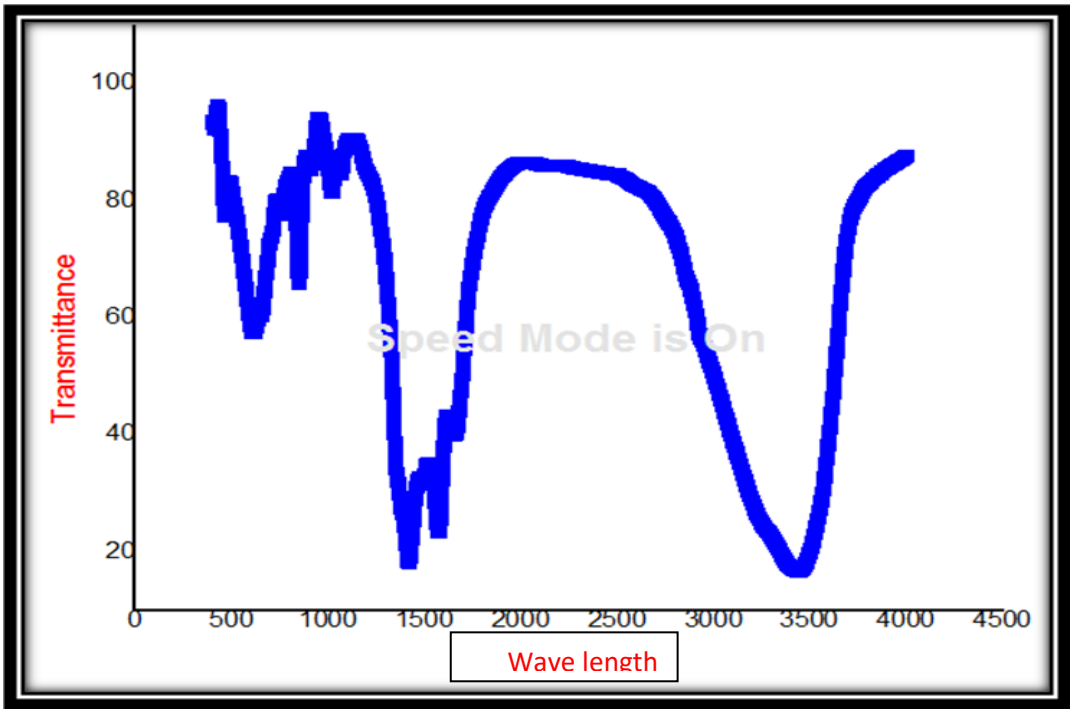

Figure 13: FT-IR spectrum of transmittance versus wave length for sample at 600 ^0C

In the figure 14 the presence of the OH band (3403.7 nm*)* in this spectrum refers to the presence of water. Moreover, the characteristic absorption band of Co (Fe)-O can be seen at 511.0437 nm.

In the case of the cobalt ferrite coated with oleic acid, the bands at 2927.4 and 2852.2 nm are related to the vibration of C-H. The band Co-OH is indicated by the vibration at 474.40 nm. The band at 1634 nm corresponds to H-O-H absorption, and that at 630.60 nm is characteristic of the FeOOH. These results clearly indicate the functionalization of the cobalt ferrite nanoparticles with oleic acid.

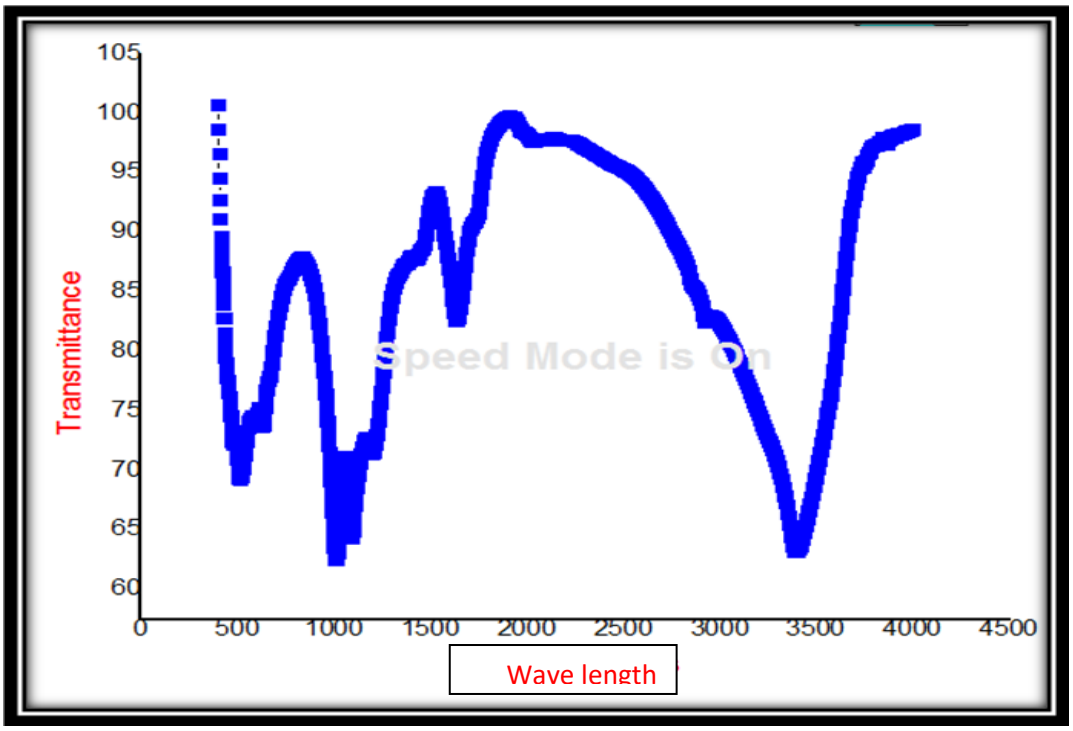

Figure 14: FT-IR spectrum of transmittance versus wave length for sample at 800 ^0C

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In the figure 15, the presence of the OH band (3444.2 nm) in this spectrum refers to the presence of water. Moreover, the characteristic absorption band of Co (Fe)-O can be seen at 568.89 nm.

In the case of the cobalt ferrite coated with oleic acid, the bands at 2360.4 and 2092.3 nm are related to the vibration of C-H. The band Co-OH is indicated by the vibration at 470.54 nm. The band at 1635.3 nm corresponds to H-O-H absorption, and that at 615.60 nm is characteristic of the FeOOH. These results clearly indicate the functionalization of the cobalt ferrite nanoparticles with oleic acid.

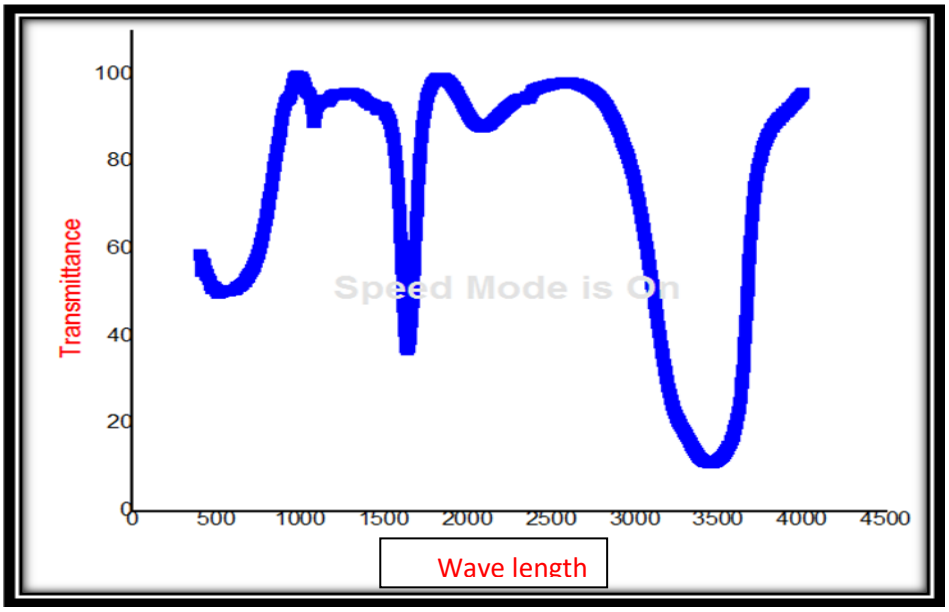

Figure 15: FT-IR spectrum of transmittance versus wave length for sample at 900 ^0C

In the figure 16, the presence of the OH band (3459.6 nm) in this spectra refers to the presence of water. Moreover, the characteristic absorption band of Co (Fe)-O can be seen at 566.96 nm.

In the case of the cobalt ferrite coated with oleic acid, the bands at 2364.3 and 2090.4 nm are related to the vibration of C-H. The band Co-OH is indicated by the vibration at 472.47 nm. The band at 1633.34 nm corresponds to H-O-H absorption, and that at 603.61 nm is characteristic of the FeOOH. These results clearly indicate the functionalization of the cobalt ferrite nanoparticles with oleic acid.

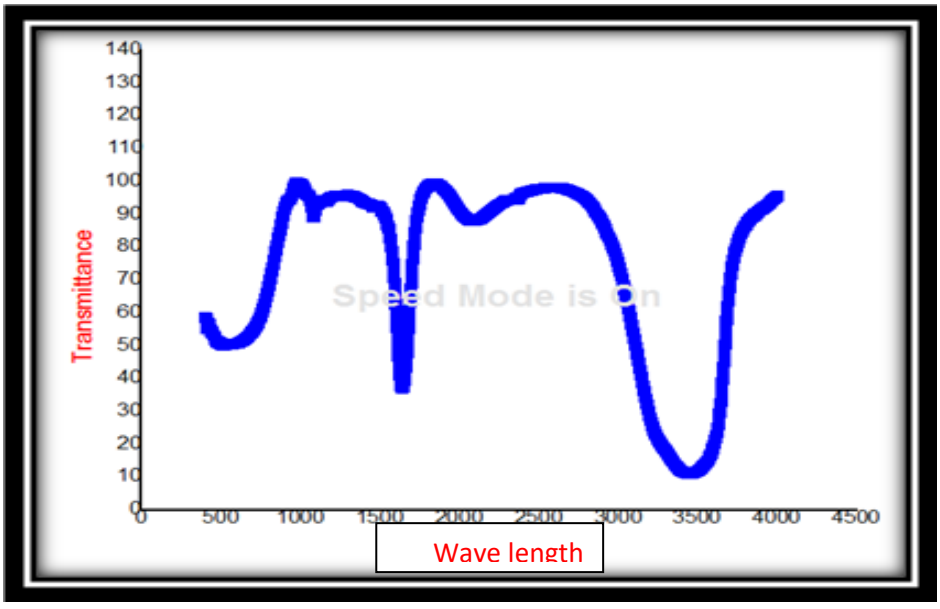

Figure 16: FT-IR spectrum of transmittance versus wave length for sample at 1000 ^0C

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5. X-ray Diffraction (XRD) Results:

XRD analysis was done to know the phase present. XRD diffraction in the figure 17 are characteristic of cubic spinel structure and the absence of extra peaks evidence phase purity, where sample calcinations at 600 ^0C , The sharp diffraction peaks indicate the transparence of the nano-crystals. There are no other detectable traces of extra crystalline or amorphous phases, the strongest peaks at 35.6° phase (622.667)

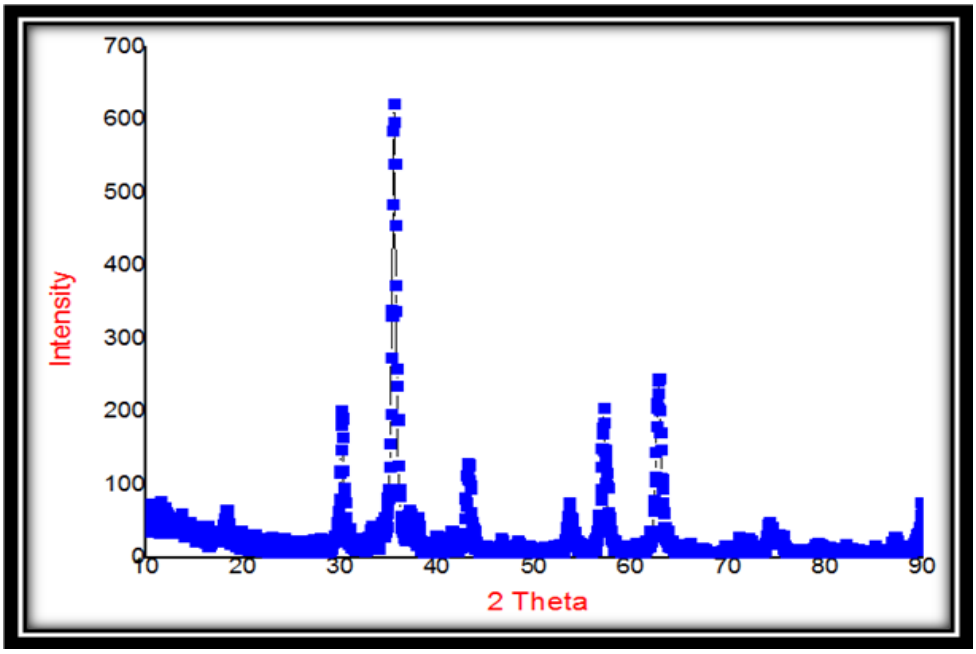

Figure 17: X-ray diffraction spectra for sample at 600 ^0C

The X-ray diffraction shown in figure 18, indicated a pure nano crystalline cobalt ferrite we can see in this figure, the strongest peaks at 35.9° phase (496), sharp diffraction peaks indicate the transparence of the nano-crystals. There are no other detectable traces of extra crystalline or amorphous phases. Where sample calcinations at 800 ^0C .

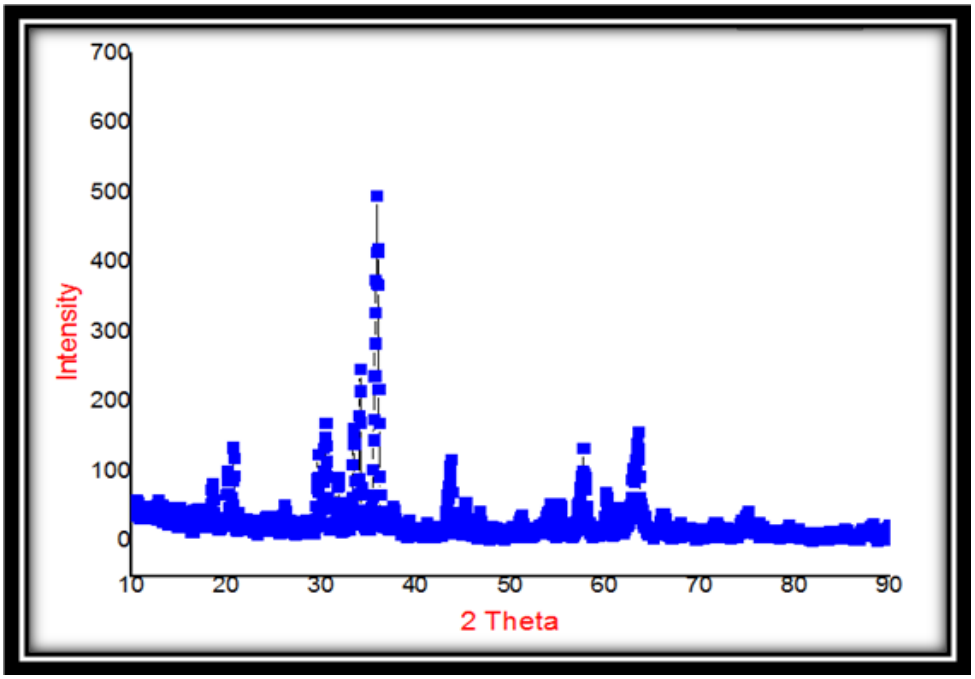

Figure 18: X-ray diffraction spectra for sample at 800 ^0C

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The X-ray diffraction shown in figure 19 and figure 20 there are detectable traces of extra crystallines and phase amorphous and existence extra line in this figure confirms no single phase formation of cobalt ferrite and evidence phase not purity.

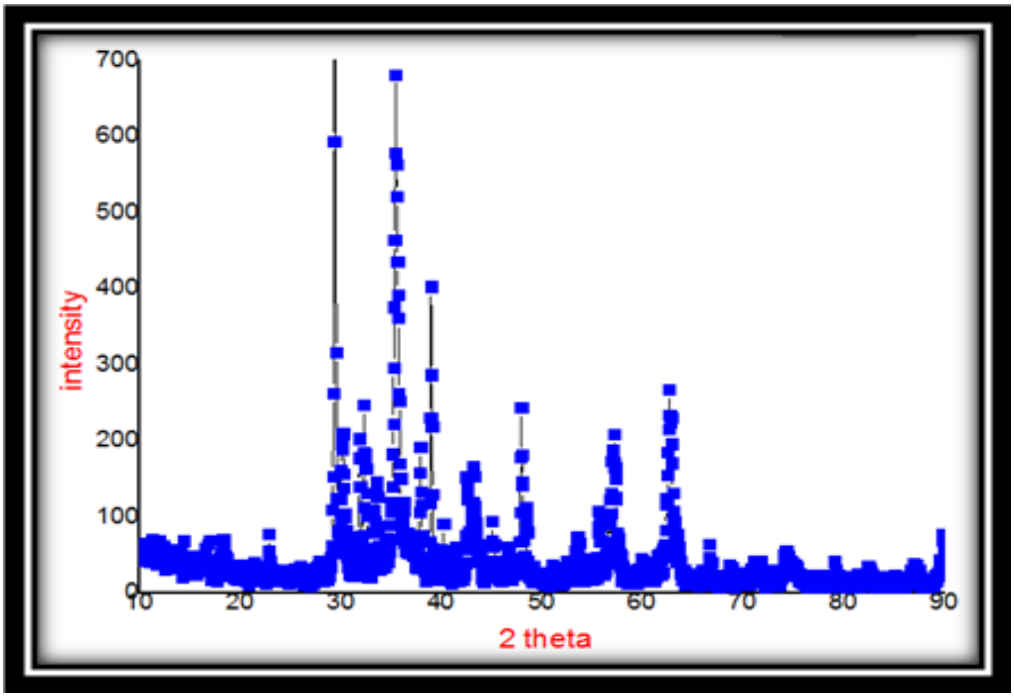

Figure 19: X-ray diffraction spectra for sample at 900 ^0C

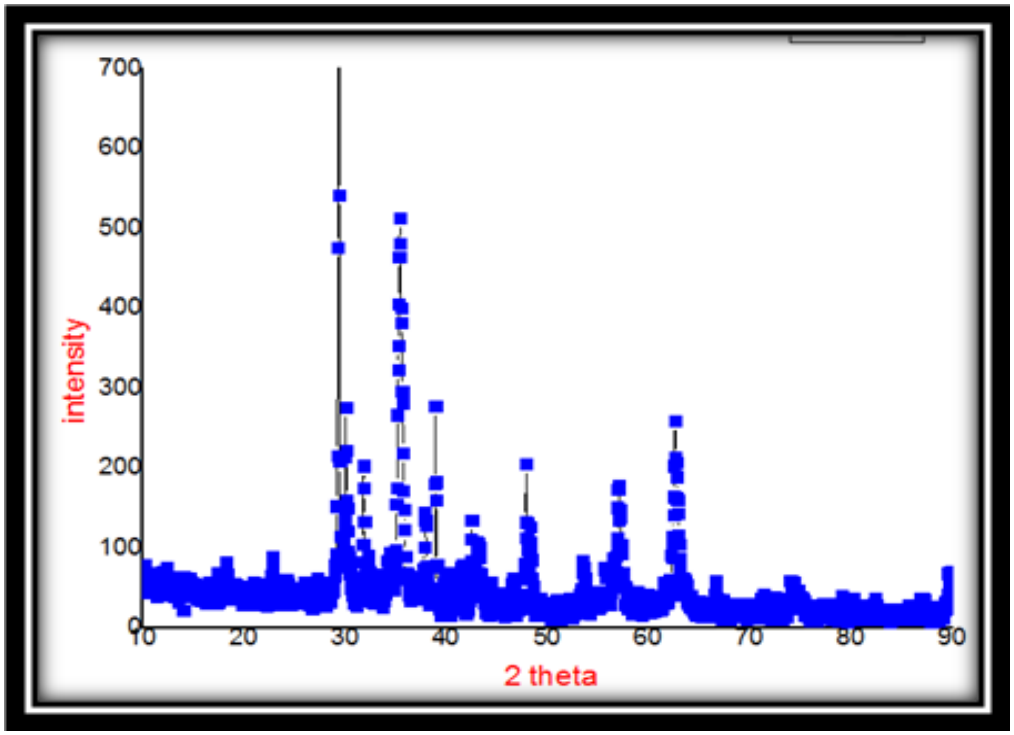

Figure 20: X-ray diffraction spectra for sample at 1000 ^0C

The peak in the figure 17 and figure 18 belongs to cubic spinel structure and the analysis of XRD pattern proves the formation of single phase in the two samples. But in the two figure 19 and figure 20 dose not belongs to cubic spinel structure and the analysis of XRD pattern prove no the formation of single phase in another two samples.

IV. CONCLUSION

In this study, the results of UV-VIS spectroscopy ensure present $CoFe_2O_4$, where γ_{max} is 372 nm. TEM measurement show that size of the samples that were synthesed by Co- precipitation method are in Nano size .The existence of stabilizer encapsulate atoms (oleic acid),which prevent agglomeration and precipitation and thus get a smaller size. The size of particles increases with increase the preparation temperature. We noted that the size of $CoFe_2O_4$ nanoparticles was 10-25 nm ,15-35 nm ,20-100 and nm ,75-115 nm for the samples prepared at 600 ^0C and 800 ^0C ,900 ^0C , 1000 ^0C respectively.

The measured magnetization curve for sample that was synthesed by co-precipitation method display clearly ferrimagnetic behavior & the magnetic property of CFNP depend on the particle size.

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