

Research Article

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Sadeq H.Lafta*

Effect of pH on Structural, Magnetic and FMR Properties of Hydrothermally Prepared Nano Ni Ferrite

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Abstract: Nano nickel ferrite particles were prepared at pH values 1.5, 4, 7, 10, 13 by a hydrothermal method using metal chlorides and NaOH as an oxidant and solution basicity controller. There is a phase transition from hematite to spinel ferrite that begins when the pH reaches 4. The lowest crystallite size (4 nm) was associated with a highest lattice constant (8.345 Å), at pH=4. Whereas maximum crystallite size 64.5 nm corresponds lattice constant of 8.298 Å at pH=10. The highest magnetization (48 emu/g) value was achieved for the sample prepared at pH=7, which at the same time has a lower coercivity. The samples synthesized at pH ≥ 4 show superparamagnetic behavior owing to its low particle size and to zero field cooling and field cooling measurements. The ferromagnetic resonance (FMR) cavity tests analysis show that the broadened linewidth (770 Oe) and high imaginary permeability or high microwave absorption which is linked to high magnetization and low coercivity of superparamagnetic particles and their aggregation. There was a shift in the resonance field due to internal fields and cation distribution.

Keywords: pH of hydrothermal, Ni-Ferrite, Crystallite size, Magnetization saturation, Coercivity, FMR linewidth.

1 Introduction

Nickel ferrite is one of a group of common magnetic material that has an inverse spinel structure. The spinel ferrites involves two sublattices known as tetrahedral and octahedral which are denoted widely as A and B respectively. The magnetic properties are functions of the cations distribution on these sites. In this case, the

cation distribution is another factor that affects the magnetic properties beside particle size in nanoferrite. Nickel ferrite (bulk or nanostructure) applications get expanded day by day, they include magnetic cores [1], catalysts [2], microwave applications [3], sensors [4], and even antibacterial [5]. At bulk size scale, nickel ferrite has been studied widely from point of preparation conditions. Recently, when nanomaterial began to open a new era in material science, the role of preparation conditions takes up more attention.

Chemical routes are considered as the main methods for nanomaterial synthesizing. The size and shape controlling of nanoferrites is the main factor that affects their properties and subsequently their applications. The previous literature show fluctuations of the pH role in nanoferrite synthesizing, especially influencing the particle size [6, 7]. Sometimes it was found that increasing the pH lead to increasing the particle size [8, 9], while it is decreased in others [10]. Therefore, the pH parameter can be considered as the key factor for controlling ferrites nanostructure and magnetic properties [11]. The saturation magnetization as an example was increased with increasing pH, whereas other researchers find an optimal magnetic saturation and coercivity at pH value of 7.5 [12,13].

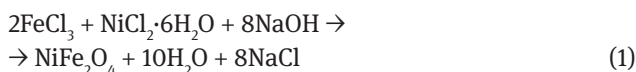
In this study the effect of pH on the structural properties of nano Ni ferrite prepared by hydrothermal method and subsequently on the magnetic properties were investigated. The XRD, SEM, and TEM was utilized to determine the structural properties and particles morphology. The moment-magnetic field hysteresis loop by the SQUID and ferromagnetic resonance FMR test were done.

2 Materials and Methods

Ferric chloride (FeCl_3) and nickel chlorides ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) in addition to NaOH were used as reactant materials to produce the Ni ferrite. It is believed that the ferrite formula

*Corresponding author: Sadeq H.Lafta: University of Technology/
Applied Science Dep., Baghdad-Iraq, E-mail: sadeq000111@yahoo.com

NiFe_2O_4 was satisfied through the following chemical reaction:



The quantity of NaOH in the above reaction is not constant and the mentioned value is just for balancing the equation, which experimentally gives a pH value of 1.5. Table (1) illustrates the calculated weights according to the above reaction to get 0.01 mole of ferrite.

Metal chlorides salts were dissolved in 200 ml distilled water. Then NaOH solution of 2.5 M was adding drop by drop until the required pH is reached under continuous stirring. During this step a dark brown precipitate was formed. This step was followed by transferring the suspension to a Pyrex flask (500 ml), which was placed inside a homemade autoclave and heated by a hotplate at 160 °C for three hours. The stirring process was kept continuous through hydrothermal operation and the pressure was fixed autogenously. It may be pointed out that the suspension before hydrothermal process did not respond to a magnet. After that, the suspension was washed three times to dismiss the produced salts and unreacted materials. Finally, filtering the products by tissue papers and drying them at 80 °C in an oven for four hours were undertaken.

Five samples were prepared at pH equal to 1.5, 4, 7, 10, and 13 with ± 0.2 tolerance in the accuracy of measuring pH. Crystalline phase purity was verified by XRD for each sample under study by using X-pert Panalytical instrument with Cu-K α radiation ($\lambda = 1.5418 \text{ \AA}$) associated with High Score Plus software to analyze the produced patterns. The error in the measuring of 2θ is 0.001° . Depending on the fact that nearly all particles have a spherical shape, the crystallite size (t) was calculated by Scherrer-Debye formula as in Eq.2 [14]:

$$t = 0.9 \lambda / \beta \cos \theta \quad (2)$$

Where (β) is the linewidth at half maximum, (λ) is the X-ray wavelength and θ is the Bragg angle.

SEM and TEM microscopies utilized FEI LEO 1550 SEM and Philips CM 12 TEM instruments to determine the particle size and particle shapes. Composition analyses in Ni ferrite samples were checked by SEM-EDX and TEM-EDX.

The hysteresis loop and magnetization-temperature tests were done by SQUID by Quantum Design MPMS XL SQUID, with specification: field range: 5T, sensitivity: 1×10^{-8} emu at 2,500 Oe, accuracy: approximately 0.1%.

Table 1: Starting material weights for 0.01 mole of Ni ferrites. Where M_w : molecular weight in gm/mole, N: no. of moles. W: weight in grams.

Starting materials	Mw g/mol	N mol.	W g
FeCl_3	162.21	2	3.24
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	237.7	1	2.38
NaOH(theoretical)	39.1	8	8

BRUKER ESR E500 device was utilized to characterize the samples powders around the resonance frequency of 9.7 GHz, it is characterized by: tuning in the range (8-14 GHz), where the cavity resonator shorts the waveguide of microwaves radiation, The quality factors: up to 8000, field modulation: up to 100 kHz, signal-to-noise: 3000:1, The sliding magnetic field: up to 10^4 G with 1G peak to peak modulation amplitude, resolution of field axis: 1024 points. Powders of samples, which have weight of 3mg, were pressed in plastic capsule to carry out these tests.

3 Results

3.1 XRD Test and Morphology Results

The XRD patterns of the five samples at the different pH values are shown in Fig.(1). The pattern of the sample at pH=1.5 show no spinel phase is existing and the pattern is belonging to $\alpha\text{-Fe}_2\text{O}_3$ with JCPDS file No.24-0072. The low concentration of sodium hydroxide (low pH) leads the reaction (1) to produce $\alpha\text{-Fe}_2\text{O}_3$ as:



This behavior may be related to higher ion reactivity [15] and concentration of ferric chloride compared to nickel chloride. As pH value increases to 4, the sample pattern leaves $\alpha\text{-Fe}_2\text{O}_3$ phase and begin to take the spinel phase. The pattern at pH=4 has also no good matching with spinel JCPDS cards. The extra increase in the pH value gives clearer spinel pattern, as a result of improving the reaction (1). The characteristic high intensive peaks at 30.8° , 35.7° , 63.3° which correspond to the planes (222), (310) and (440) of spinel structure began to appear when the pH value exceeds 4. The peaks around 46, 50 and others which are unmarked by miller indices are belonging to the presence of hematite and NaCl (halite phase) which support the occurrence of reaction(1) or (3).

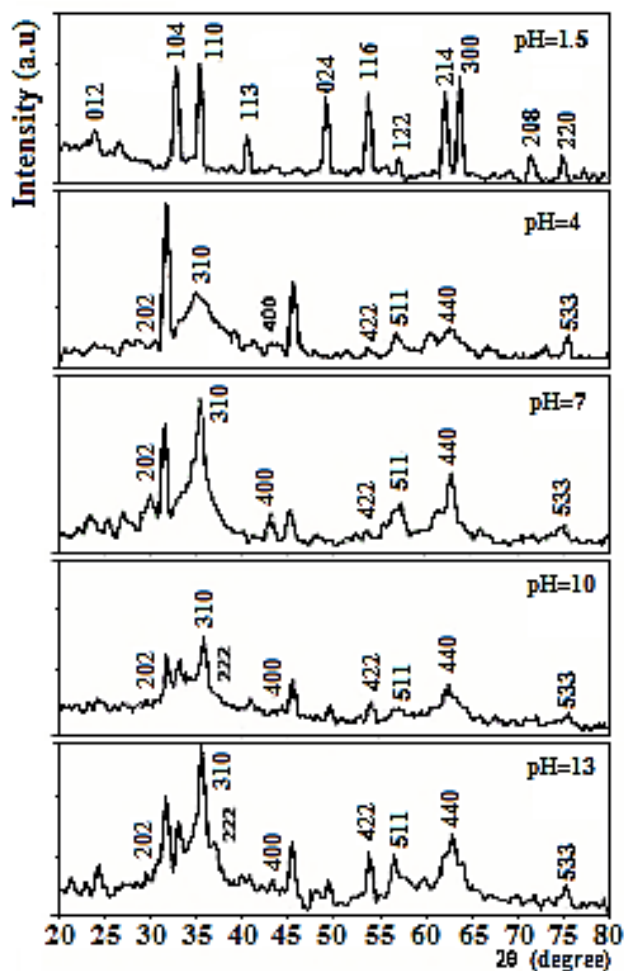


Figure 1: XRD patterns of prepared samples at different pH.

The variance of intensities around 31° and 36° is belongs to cation distribution on A and B sites. This variance in intensities and the low intensities itself are indications of the presence of nanosize especially at pH=4 and the presence of multiphases [16]. The hemetite phase has a shift in peaks positions to higher values by 0.1 degree JCPDS 96-901-5965. The peaks located at 2θ higher than 40° have lower intensities than standard. The Ni ferrite peaks have also a shift to the higher value by 0.23° about the JCPDS 96-591-0065 ones.

The effect of pH on crystal size is linked to crystal growth. Table (2), illustrates the lattice constant (a) and crystallite size (t) variation with corresponding pH values. The lattice constant was minimum at pH=7, while it was maximum at pH=4. It is estimated that the tension stress, which is affected by the crystal size, is the reason behind the variation. The largest crystallite size was (64.5 nm) at pH=10. It is clear from Table (2) that the increase in pH lead to an increase crystallite size except for the case at pH=13.

Table 2: Lattice parameter (a) and crystallite size (t) for spinel samples prepared at different pH. (Sample at pH=1.5 is excluded since it has no spinel phase).

pH	a /Å	t /nm
4	8.345	4.557
7	8.202	28.72
10	8.298	64.5
13	8.315	47.0

This behavior can be explained on the basis of crystal growth, where it thought that at pH=10 the growth rate is the largest which gives maximum crystallite size [17].

The predominant particle morphology was changed from nanorods to nanosemisphears when pH varied from 1.5 to 4 (or larger than 4) as seen in Fig.(2)a and b. The presence of nanorods may be attributed to the existence of $\alpha\text{-Fe}_2\text{O}_3$ phase which is usually constructed under such condition [18], while spinel structure has spherical and low concentration of nanorods.

Besides that the sample of pH=1.5 had nanorods as a general particles, it also had a low concentration of nanosemisphears as shown in Fig.(3)a where an aggregation of nanorods appeared. The sample synthesis at pH values of more than 4 had a very low concentration of nanorods, this can be shown in Fig.(2)b.

The average particle sizes are 6.5 nm, 66.5 nm, 65.8 nm, 51 nm at pH=4, 7, 10, 13 respectively as shown in Fig. (2) and Fig.(3) and there is no large distribution in these sizes. It is considered that pH >10 acts as capping factor besides increasing nucleation centres because of reducing the path of species. Comparing the particle size with the crystallite size in Table (2), gives an indication that each particle contains just one or two crystals.

Nickel, iron and oxygen contents that checked by EDX techniques associated with SEM and TEM devices show good agreement when pH exceeds 7 with stoichiometric composition of samples.

The important benefits of using hydrothermal process for production nanostructures are narrow sized distribution, controlled shape and simplicity [19]. For this study, it is believed that the controlling of size and shape particles is dependant on pH and other conditions such as solution concentration, the ratio of nickel chloride to ferric chloride and temperature.

Synthesizing Ni ferrite particle by solid state reaction [20] reported that the size was 6.16 nm and the lattice parameters was 8.337nm. The particle size is smaller but the lattice constant is nearly larger than the ones of this study, knowing that the bulk Ni ferrite has $a=8.340 \text{ Å}$ [21].

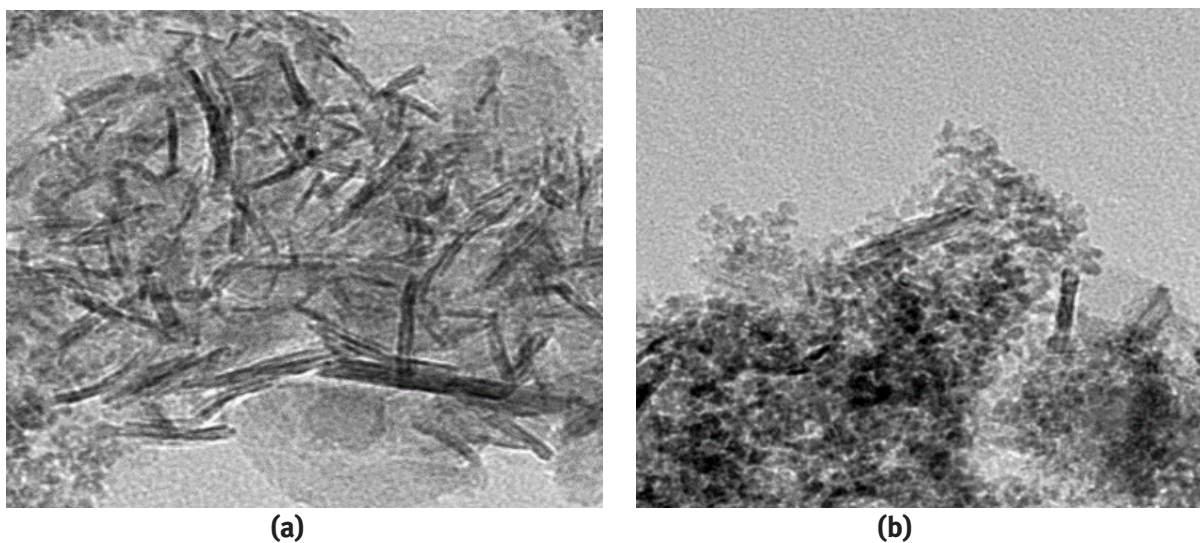


Figure 2: TEM images, (a) at pH=1.5 (image dimensions: 132.5 nm x 132.5nm), (b) at pH=7 (image dimensions 1.1 μm x 1.1 μm).

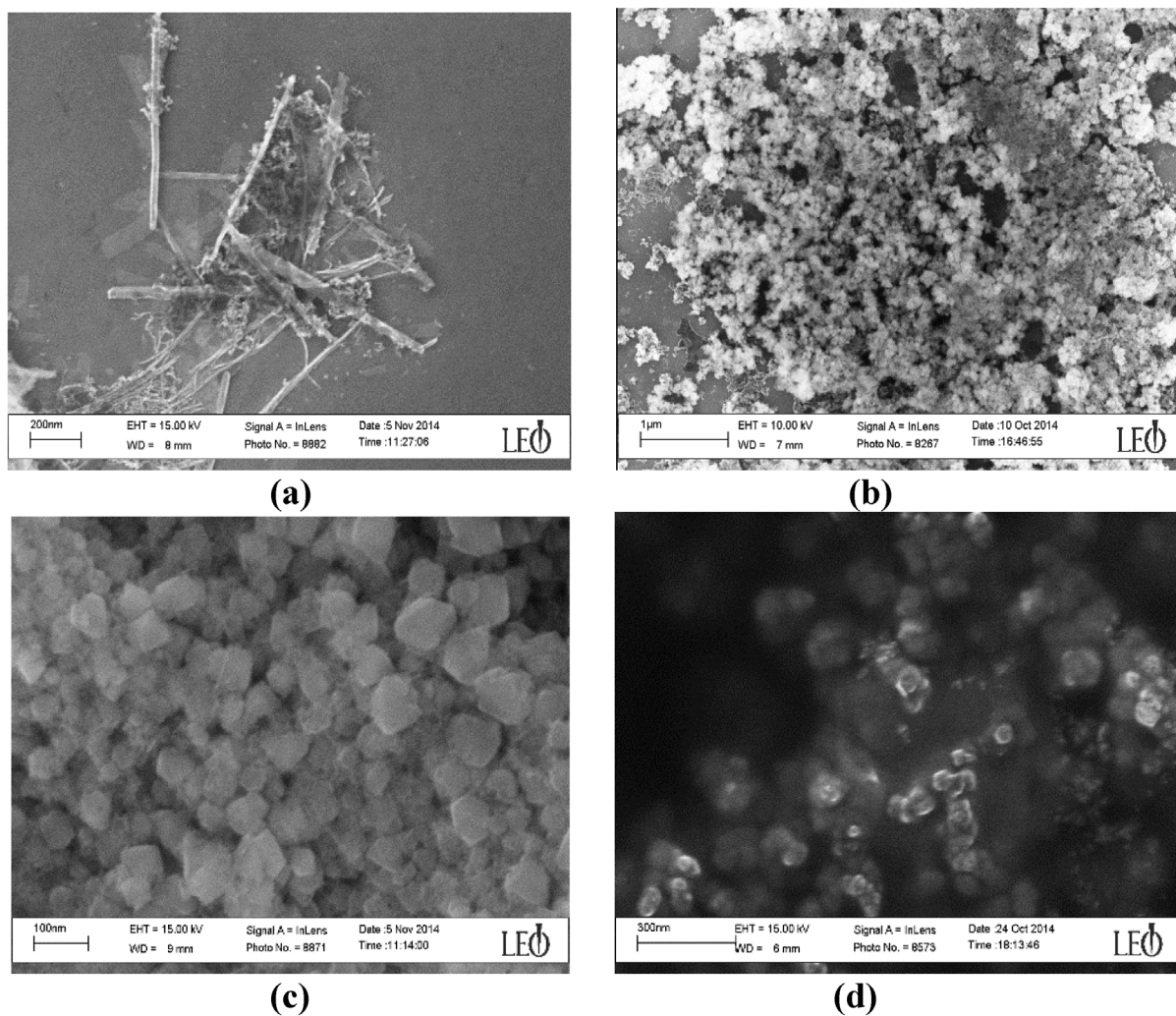


Figure 3: SEM images, (a) aggregation of nanorods at pH=1.5, (b) nanospheres at pH=10, (c) at pH=13. (d) at pH=7.

The crystallite size of the prepared samples in general is lower than that prepared by hydrothermal method [22] but larger than ones synthesized by microwave hydrothermal [23]. Of course these differences are related to preparation conditions which produce compressive or tension stresses in the lattices, as well as the starting material and differences in cations and oxygen concentration.

As in the present study, different nanostructures or particle shapes were also appeared in Zn ferrites and Ni ferrites prepared by co-precipitation [24]. Particle size is the parameter that is very sensitive to preparation conditions and can be controlled by them. However the particles size of current study are asymptotic to that of other researchers [25] and other ferrites [26-29].

3.2 Magnetic Properties Results

Hysteresis loops of the prepared samples are presented in Fig.(4). The sample at pH=1.5 which has dominant $\alpha\text{-Fe}_2\text{O}_3$ structure show paramagnetic like behavior with small hysteresis loop as magnified inside Fig.(4) which is probably because of the presence of a small amount of ferrite phase as shown by the expanded box. The sample at pH=4 shows more ferrimagnetic nature than sample of pH=1.5. When pH=7, the largest moment (or magnetization M) and initial permeability was produced as seen in Table (3). The parameter M reduced to lower values when pH was increased 13. Here, since there was no apparent saturation, the measured magnetization values are determined at 42000 Oe for comparison. The difference between the samples at pH=10 and 13 is that the latter has higher remanence (M_r) than former. Coercivity was increased as pH increased up to 7 then decreased with continuously increasing pH. These results are summarized in Table (3). One can say, pH and subsequently particle size influences both M_r and H_c intensively. The measured initial permeability (μ_{in}) is shown in Table (3). Again the higher value for μ_{in} is at pH=7 which of course is due to high magnetization and low coercivity.

Depending on the evidence of unsaturated curves of all samples Fig.(4) and Table (3) values, one can say that particle size is in the range of superparamagnetic size of ferrite [30, 31].

Samples at pH equal to 7 and 10 had coercivity (H_c) values lower than single crystal Ni ferrite samples prepared by CVD [32] and higher than sol-gel Ni-ferrite [33]. The saturation magnetization M_s cannot be compared to literature since there was no saturation behavior, but it is good to mention that M_s of bulk Ni ferrite is about 50 emu (3200 G)[21].

Table 3: Magnetic parameters for samples prepared at pH=1.5, 4, 7, 10, 13.

pH	H_c (Oe)	M_r (emu/g)	M (at 42000 Oe) (emu/g)	μ_{in} (emu/g.Oe)
1.5	3000	0.5	4	0.02×10^{-2}
4	307	0.8	5.5	0.18×10^{-2}
7	9.5	1.2	48	13.4×10^{-2}
10	19	0.9	36	3.36×10^{-2}
13	75	3.6	36	3.73×10^{-2}

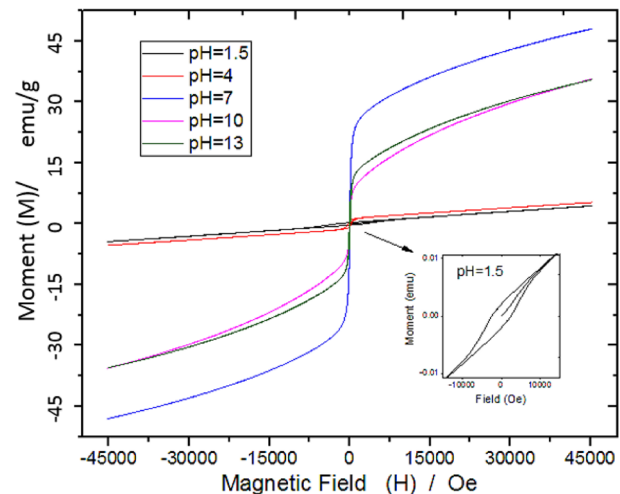


Figure 4: Hysteresis loops of samples prepared at pH=1.5, 4, 7, 10 and 13.

The field cooling (FC) measurements were conducted at a magnetic field of 50 Oe through a temperature range (300 -10 K), whereas the zero field cooling (ZFC) measurements were done from 300 K to 50 K. The curves of FC and ZFC for the samples at pH=10 and 13 are not united below 350 K, as shown in Fig. (5). These curves reflect the superparamagnetic behavior [34,35] for the three samples with blocking temperature (T_b) higher than 350 K for the samples at pH=10 and 13 and around 350 K for the sample at pH=7. The curves uniting at pH=7 at lower temperature is believed to be due to smaller particle size. On the other hand, the reason behind the low decreasing rate in the FC curve is related to particles aggregations due to magnetic attraction and high surface area of nanoparticles. These aggregations are clearly seen in the Fig.(3) images. The particle aggregations promote magnetic interactions among these particles that lead to freezing them and not allowing them to be in the field direction at low field[36]. For the ZFC curve, the particle magnetic moment is going to be zero when temperature is going to 0K.

Ferromagnetic resonance (FMR) analysis is an excellent tool to diagnose material effective magnetization, spin-lattice relaxation, and damping constant via determining linewidth and resonance field especially for microwave device application [37,38]. FMR tests were done for samples prepared at pH=7, 10, 13 as shown in Fig.(6), whereas the other samples (pH=1.5 and 4) did not show noticeable FMR signals which is the derivative (dP/dH) of absorbed microwave power (P) with respect to the magnetic field (H). The most intensive signal relates to pH=7 where the maximum magnetization resides as shown in Fig.(4). The rest signals are fairly less than the previous.

The complex permeability μ_r'' which represents the intensity of absorbed microwave power that proportional to magnetic parameters as in Eq.4 [39]:

$$\mu_r'' = M_s / (2 \mu_0 H_a \alpha_d) \propto M_s / H_c \quad (4)$$

Where μ_0 , K_1 and α_d are magnetic permeability, magnetic anisotropy and damping factor respectively, knowing the anisotropy field $H_a = 2K_1/M_s$. For single domain particle, H_a is equal H_c in certain conditions of measurements, which can be considered here for simplicity [40]. It is clear from Eq.4 that the higher M_s the higher μ_r'' and the higher H_c the lower μ_r'' . The complex permeability result is shown in Fig.(5)b in arbitrary units that is an integration of Fig. (5)a. Where the higher absorption power is occurred for sample prepared under pH=7 and lower power is absorbed at pH=10 and pH=13.

The widest linewidth ΔH_{pp} belongs to sample pH=7. This means that samples have the highest damping factor and the highest microwave resonance absorption. The FMR parameters extracted from Fig.(6) are written down in Table 4.

The resonance field H_{res} is shifted to a higher value when pH increases to 10 and 13. The resonance frequency f_0 (microwave frequency) given by Larmor relation $f_0 = \gamma H_{res}/2\pi$ (γ is the gyromagnetic ratio) must involve the role of the internal field H_i besides the external one H_{ex} , i.e. [41,42]:

$$H_{res} = H_{ex} + H_i \quad (5)$$

This field (H_i) is in turn attributed to: the exchange field, the anisotropy field, the demagnetization field, the porosity field due to the magnetic dipoles on pores. This will result that the internal field is altered somewhat from location to other in the sample. So H_{res} may be shifted a little if the tetrahedral (A site) and octahedral (B site) are not filled homogeneously or the sites occupancy is disorder. Subsequently. This might be happening in $NiFe_2O_4$ if Ni^{2+}

Table 4: FMR parameters for pH values: 7, 10 and 13 at frequency of 9.7GHz.

pH	Linewidth ΔH_{pp} / (G)	Resonance Field H_{res} / (G)	Imaginary Permeability μ_r'' / (a.u)
7	770	2835	22570
10	690	3282	4850
13	678	3282	2440

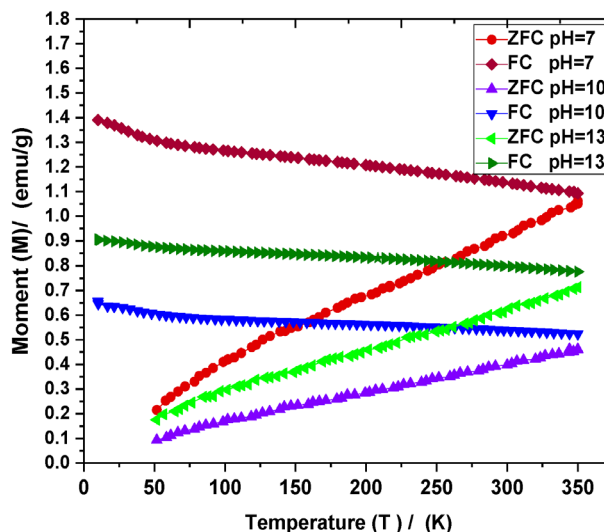


Figure 5: ZFC-FC curves of nano Ni ferrite at pH=7, 10 and 13.

on B sites and Fe^{3+} on both sites are distributed disorderly. These reasons of inhomogeneity will produce also a line broadening in FMR of ferrites. Regarding the line shape, one can see there is asymmetric in the signal at pH=7 which may be related to nonlinear effect at higher field values.

Finally, the increasing linewidth may be related to large particle size distribution which is associated with a slight resonance field shift to higher value. Also particle aggregation with respect to disperse one has a strong effect on the linewidth and resonance field where aggregation leads to broaden the line owing to particles in interaction and that will decrease the resonance field. It is believed that the both latter factors are responsible for the signal behaviors in Fig.(5). Most polycrystalline ferrites possess linewidth in range 10-100 Gauss and grow to several hundred for nanoferrite due to inhomogeneity and spin-orbit interaction [19]. Literature [43-45] showed linewidth (500-1000 Oe) equivalent to samples of current study. The linewidth broadening is mainly related to conductivity of sample, containing relaxing ions (Ni^{2+}), demagnetization and anisotropic broadening.

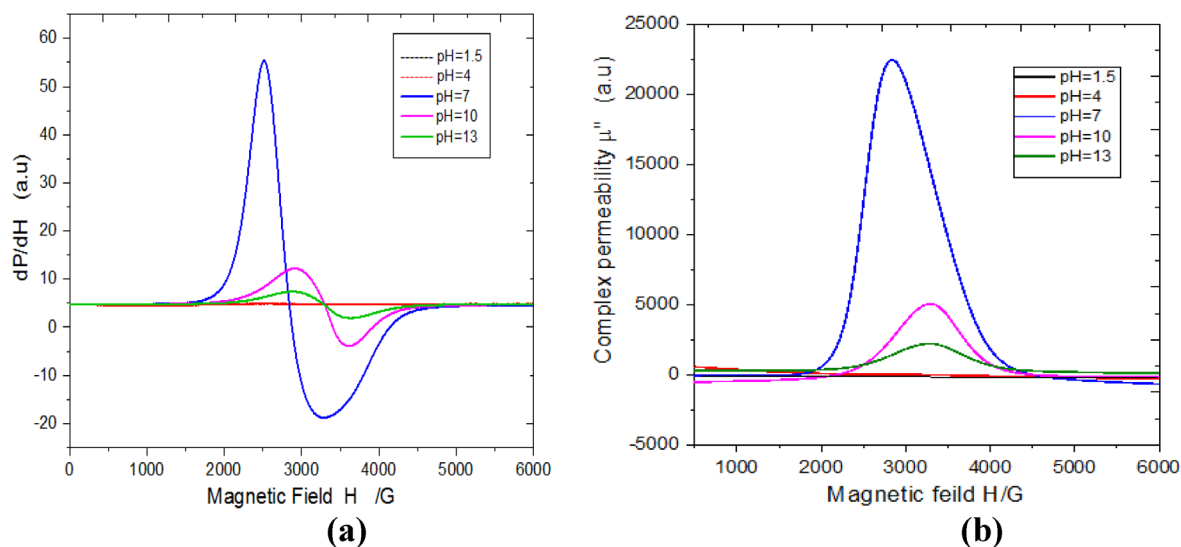


Figure 6: a) FMR signal (dP/dH) versus magnetic field for samples at different pH. b) Complex permeability (absorbed microwave power) for samples at different pH.

4 Conclusion

The role of pH in structural and magnetic properties involving hysteresis and FMR properties of nickel ferrite is affecting the particle size and/or crystallite size which is in turn affecting ferrites properties. The highest particle sizes is confined to pH values from 7 to 10, thus higher magnetization, lower coercivity, higher FMR linewidth exist in this pH range. Superparamagnetic behavior is shown for all samples due to low particle size in general. These parameter values encourage the application of these materials in biomedical applications like magnetic resonance imaging MRI, magneto-resistive sensors, antibacterial and catalyst.

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