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Magnetic Behavior of Virgin and Lithiated NiFe_2O_4 Nanoparticles

Ghadah M. Al-Senani ¹ , Foziah F. Al-Fawzan ¹, Rasmiah S. Almufarrij ¹, Omar H. Abd-Elkader ^{2,3,*}  and Nasrallah M. Deraz ⁴

¹ Department of Chemistry, College of Science, Princess Nourah Bint Abdulrahman University, P.O. Box 84428, Riyadh 11671, Saudi Arabia

² Physics and Astronomy Department, Science College, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia

³ Electron Microscope and Thin Films Department, Physics Research Institute, National Research Centre, P.O. Box 21111, Giza 12622, Egypt

⁴ Physical Chemistry Department, National Research Centre, P.O. Box 21111, Giza 12622, Egypt

* Correspondence: omabdelkader7@ksu.edu.sa

Abstract: A series of virgin and lithia-doped Ni ferrites was synthesized using egg-white-mediated combustion. Characterization of the investigated ferrites was performed using several techniques, specifically, X-ray Powder Diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and High-resolution transmission electron microscopy (HRTEM). XRD-based structural parameters were determined. A closer look at these characteristics reveals that lithia doping enhanced the nickel ferrite lattice constant (a), unit cell volume (V), stress (ϵ), microstrain (σ), and dislocation density (δ). It also enhanced the separation between magnetic ions (L_A and L_B), ionic radii (r_A , r_B), and bond lengths ($A-O$ and $B-O$) between tetrahedral (A) and octahedral (B) locations. Furthermore, it enhanced the X-ray density (D_x) and crystallite size (d) of random spinel nickel ferrite displaying opposing patterns of behavior. FTIR-based functional groups of random spinel nickel ferrite were determined. HRTEM-based morphological properties of the synthesized ferrite were investigated. These characteristics of NiFe_2O_4 particles, such as their size, shape, and crystallinity, demonstrate that these manufactured particles are present at the nanoscale and that lithia doping caused shape modification of the particles. Additionally, the prepared ferrite's surface area and total pore volume marginally increased after being treated with lithia, depending on the visibility of the grain boundaries. Last, but not least, as the dopant content was increased through a variety of methods, the magnetization of virgin nickel ferrite fell with a corresponding increase in coercivity. Uniaxial anisotropy, rather than cubic anisotropy, and antisite and cation excess defects developed in virgin and lithia-doped nickel ferrites because the squareness ratio (M_r/M_s) was less than 0.5. Small squareness values strongly recommend using the assessed ferrites in high-frequency applications.

Keywords: XRD; FTIR; HRTEM; virgin and lithia-doped NiFe_2O_4 ; squareness; antisite defects; magnetization; magneto crystalline anisotropy



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1. Introduction

Most ferrite-based nano particles (NPs) result from the solid-state reaction between the oxides of their constituent elements. The preparation process, which is among the most crucial of these variables, determines the type, structure, and behavior of these ferrites. One of the most crucial techniques for improving and controlling ferrite structures, as well as their optical, morphological, electrical, and magnetic properties, is the doping of ferrites with specific elements.

Doping of ferrites could result in many effects, including the following: (i) alterations in the cation distribution between the ferrite structure's tetrahedral (A) and octahedral (B) locations [1–8]; (ii) Alteration in the energy of the ferrite grain boundaries, which drives

the evolution of the grain [1]; (iii) Alternation in the size of ferrite particle by contraction or expansion of the ferrite lattice; and (iv) Creation of oxygen vacancies with a subsequent change in the ferrite surface. In other words, the control in the microstructures of the ferrites can be achieved by doping of the host matrices with smaller cations [2]. In fact, the magnetic properties of cobalt ferrite were improved by doping it with alumina or zincate [3,4]. The magnetic qualities of zinc ferrite can be controlled by varying the dopant content. The addition of 12–46 weight percent of Li to zinc ferrite when the dopant content was increased inhibited magnetization after being enhanced by Li_2O [5]. However, doping zinc and nickel ferrites with alumina and magnesia led to a reduction in magnetism [6–8].

The preparation method is one of several variables that determine the physicochemical characteristics of ferrites and is expected to be essential for obtaining high-purity, nanoscale ferrites with particular attributes. Nanostructure ferrites can all be prepared using a wide range of techniques, including sol-gel, co-precipitation, refluxing, solvothermal, mechanochemical, ceramic, combustion, microwave plasma, micro emulsion, and mechanical alloying [1,4–10]. The main draw-back of the majority of earlier techniques is the low crystallinity of the resulting ferrite nanoparticles, which can be improved by further heat treatment [11].

Among the preparation routes of nano crystalline ferrites is the auto-combustion method, which has various advantages. Firstly, it is a simple and low-cost process depending upon a short reaction time and low processing temperature with subsequent low energy consumption [12,13]. Secondly, this approach possesses a high degree of purity, a homogeneous chemical composition, and good sinterability [13]. Thirdly, the structure, physicochemical properties, surface properties, and magnetic behavior are all under good control [13]. To put it another way, it demonstrates control over particle growth, reduces agglomeration of nanoparticles, increases their stability, and optimizes their magnetic buildability. Finally, by selecting a suitable aqueous or non-aqueous solvent mixture, by adjusting the kind and content of fuel utilized, it enables the large-scale manufacture of ferrites with controllable size and shape [4–8,14]. In fact, the nitrate salts are frequently used in preparation of spinel ferrites by the combustion method because these salts are water-soluble and low-temperature oxidizing agents [4–8].

In reality, ferrite-based nanoparticles (NPs) constitute a burgeoning technological and economic sector with complete expansion in a wide range of industrial and biomedical application domains. It is possible to attribute the popularity of nano sized ferrites in technological breakthroughs to their tunable physicochemical properties, which include structural, microstructural, surface, electrical and thermal conductivity, catalytic activity, and light absorption and scattering, and result in enhanced performance over their bulk counterparts. Ferrite-based materials are versatile and effective in the industry sector, for example, in magnetic recording media, photoelectric devices, sensors, magnetic pigments, storage devices, and batteries and solar cells. Ferrites can also be applied in the biomedical field, such as in controlled drug delivery, tumor treatment, magnetic resonance imaging, biomagnetic separation, cellular therapy, tissue repair, cell separation, and bio sensing [1–3,9,10,12,15–20].

This study aims the green synthesis of virgin spinel nickel ferrite nano particles (NiFe_2O_4 NPs) and nano particles doped with different amounts of lithia via the egg-white-associated auto-combustion method. This investigation is particularly interesting because there is a dearth of knowledge regarding the effects of lithium doping on the development and various aspects of virgin spinel nickel ferrite. We, therefore, investigated the effects of lithium doping on the formation, structural, morphological, surface, and magnetic characteristics of NiFe_2O_4 NPs.

2. Materials and Methods

2.1. Materials

Lithium nitrate, ferric nitrate hydrate, and nickel (II) nitrate all have linear formulae. The chemical ingredients employed were LiNO_3 , $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$,

respectively. These materials were supplied by the Sigma-Aldrich Company (Darmstadt, Taufkirchen, Germany). These reagents were applied quantitatively and required no further processing. Egg white was sourced from the raw eggs of local hens.

2.2. Preparation Method

One sample (S1) of virgin NiFe_2O_4 and two samples (S2 and S3) were fabricated by using the egg-white-mediated combustion route. Taking the stoichiometric ratio of $\text{Fe}/\text{Ni} = 2$ into consideration, the first sample (S1) was made by completely combining an equimolar combination of ferric nitrate hydrate and nickel nitrate hexahydrate with 6 mL of egg white in a crucible. The resulting liquid was first swirled at 60 °C in order to allow the water to evaporate and to boost viscosity. The mixture was subsequently converted into a gel by raising the temperature to 120 °C. The created precursor gel was calcined at 300 °C for 15 min to raise the temperature to a crucible level. A large amount of foam had already started to form when a spark erupted in one corner and immediately spread throughout the mass; the final product was a dense, fluffy solid. Two samples (S2 and S3) were prepared following the same procedure but in the presence of 6 mole % and 12 mole % lithium nitrate, respectively.

2.3. Characterization Systems

Using a BRUKER D8 advance diffractometer (Karlsruhe, Germany) and X-ray diffraction technology, measurements of diverse nanoparticles were carried out. The patterns were operated with $\text{Cu K}\alpha$ radiation at 40 kV, 40 mA, and a scanning rate of 2° per minute. In order to calculate the mean crystallite size (d), dislocation density (δ), stress (ϵ), and strain (σ) of NiFe_2O_4 involved in the investigated product, and based on calculations using the Scherrer equation and X-ray diffraction line widening, Equations (1) through (4) were used [21,22]:

$$d = (B \lambda) / \beta \cos \theta \quad (1)$$

$$\delta = 1/d^2 \quad (2)$$

$$\epsilon = \beta \cos \theta / 4 \quad (3)$$

$$\sigma = \epsilon Y \quad (4)$$

where d is the average crystallite size of the phase being studied, B is the Scherrer constant (0.89), λ is the employed X-ray beam wavelength, β is the full-width half maximum (FWHM) of diffraction, θ is the Bragg's angle, and Y is the Young's modulus.

Fourier-transmission infrared spectra (FTIR) of different materials were measured using a Perkin-Elmer Spectrophotometer (type 1430). Two milligrams of each solid sample were mixed with 200 mg of vacuum-dried IR-grade KBr. A range of 1000–4000 cm^{-1} was used to measure the FTIR spectra. A steel die with a 13 mm diameter was used to scatter the mixture after it had been processed in a vibrating ball mill for three minutes. The identical disks were placed in the double grating FTIR spectrophotometer holder.

Utilizing JEOL JAX-840A and JEOL Model 1230 transmission electron microscopes (TEM) operating at 100 Kev, the specimens were placed in a specific solvent, and a deep coating of copper grid with carbon film was applied before the grid was allowed to completely evaporate the solvent before examination. The sample was briefly ultrasonically dispersed to spread individual particles over the mount arrangement and copper grids.

Utilizing both standard volumetric instruments (Brunauer–Emmett–Teller method) and surface area analyzers from the Micromeritics Gemini VII 2390 V1.03 series, the specific surface area (S_{BET}), total pore volume (V_{P}), and mean pore radius ($\bar{r}$) of various materials were evaluated (Microtrac, Alpharetta, GA, USA). Prior to the measurements, each sample was out-gassed for two hours at the lower pressure of 10^{-5} Torr at a temperature of 200 °C. A vibrating sample magnetometer (VSM; 9600-1 LDJ, USA) with a 20 kG maximum applied field was used to investigate the magnetic properties of the under-recognized solids.

3. Results

3.1. XRD Analysis

XRD analysis as a structural technique enabled us to study the purity and formation phase with subsequent investigation of different structural properties for the undoped and Li-doped NiFe_2O_4 studied. Figure 1 displays XRD patterns of virgin NiFe_2O_4 NPs (S1) and those doped by (S2 and S3) at two lithium concentrations (3 and 6 mole%, respectively). Table 1 lists the Miller indices, d spacing, and values of 2θ for the crystalline phases that are present in the as-synthesized solids. In contrast, the whole spectrum of diffraction peaks of the virgin sample is indexed as (220), (311), (222), (400), (422), (511), (440), (620), (533), (522), and (444) crystal planes at $2\theta = 30.33^\circ, 35.40^\circ, 37.18^\circ, 43.10^\circ, 53.80^\circ, 57.46^\circ, 63.01^\circ, 71.55^\circ, 74.55^\circ$, and 75° (NiFe_2O_4 -PDF: 44-1485). From this figure, it is evident that Li-doped samples do not show peaks related to any phases other than those belonging to the NiFe_2O_4 -based spinel type structure. Pure sample lithia doping resulted in a shift in all diffraction peaks, as well as a reduction in the height and FWHM of various peaks. For all diffraction peaks, an increase in lithia concentration would, in actuality, lead to a greater in shift and a reduction in both the intensity and FWHM. The cation distribution on the tetrahedral and octahedral sites included in the spinel structure could be estimated from the literature by tracing the peak height at the (220) and (440) reflection planes [8]. The cations on the tetrahedral, octahedral, and oxygen ion parameters, respectively, have a strength and durability on the intensities of the (220), (440), and (511) planes. Therefore, the comparison of the peak height at these planes is very necessary and important, especially after doping of virgin NiFe_2O_4 with lithia. It was found that the peak height at the (440) reflection plane was greater than that at the (220) reflection plane with an increase in the content of lithium.

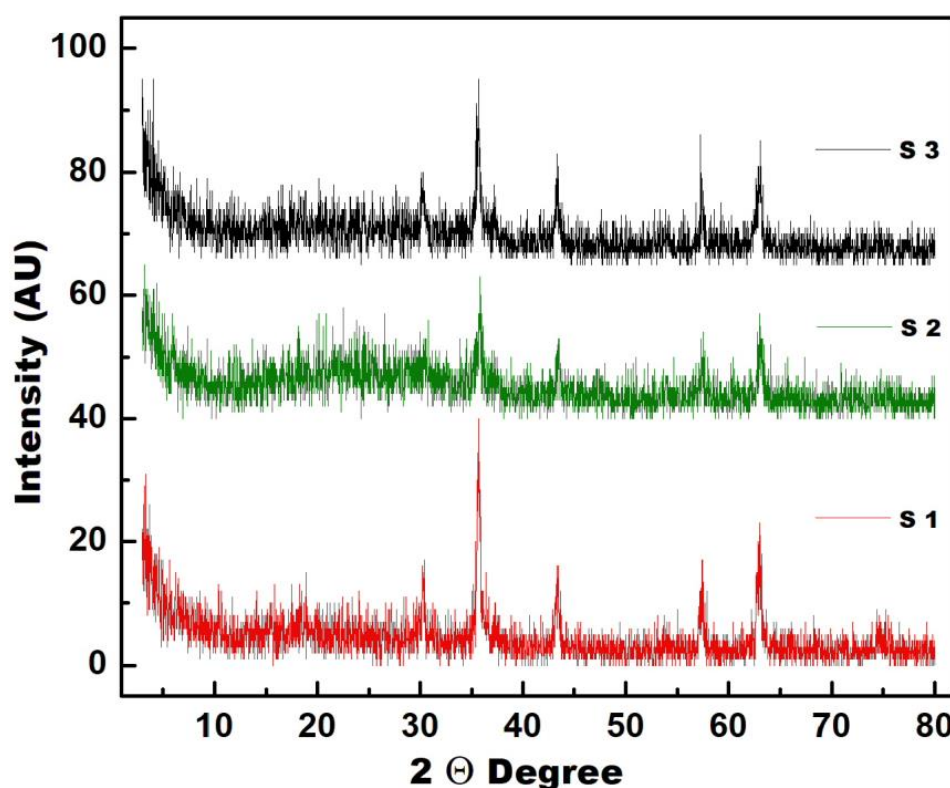


Figure 1. XRD patterns of S1, S2 and S3 samples.

Table 1. Miller indices, and the values of 2θ for the crystalline phases (NiFe_2O_4) in the as-synthesized solids.

Sample	h	k	l	2θ Obs.	2θ Calc.	Difference	Error %	Phase	Space Group
+0% Li_2O	1	1	1	18.4700	18.5433	−0.0733	−0.39686	NiFe_2O_4	FD3M
	0	2	2	30.4300	30.5082	−0.0782	−0.25698	NiFe_2O_4	FD3M
	1	1	3	35.8100	35.9392	−0.1292	−0.36079	NiFe_2O_4	FD3M
	2	2	2	37.3500	37.5958	−0.2458	−0.6581	NiFe_2O_4	FD3M
	0	0	4	43.5300	43.6879	−0.1579	−0.36274	NiFe_2O_4	FD3M
	3	1	3	48.1000	47.8405	0.2595	0.5395	NiFe_2O_4	FD3M
	2	2	4	54.0800	54.2204	−0.1404	−0.25962	NiFe_2O_4	FD3M
	3	3	3	57.5800	57.8084	−0.2284	−0.39667	NiFe_2O_4	FD3M
	0	4	4	63.0200	63.4982	−0.4782	−0.75881	NiFe_2O_4	FD3M
	3	1	5	67.0490	66.7771	0.2719	0.40552	NiFe_2O_4	FD3M
	2	4	4	68.1290	67.8518	0.2772	0.40688	NiFe_2O_4	FD3M
	2	0	6	72.3750	72.0744	0.3006	0.41534	NiFe_2O_4	FD3M
	5	3	3	75.4930	75.1751	0.3179	0.4211	NiFe_2O_4	FD3M
	2	2	6	76.5220	76.1984	0.3236	0.42288	NiFe_2O_4	FD3M
	4	4	4	79.6200	80.2507	−0.6307	−0.79214	NiFe_2O_4	FD3M
+3% Li_2O	0	2	2	30.4300	30.4727	−0.0427	−0.14032	NiFe_2O_4	FD3M
	1	1	3	35.8100	35.8970	−0.0870	−0.24295	NiFe_2O_4	FD3M
	2	2	2	37.6500	37.5514	0.0986	0.26189	NiFe_2O_4	FD3M
	0	0	4	43.5800	43.6357	−0.0557	−0.12781	NiFe_2O_4	FD3M
	3	1	3	47.8700	47.7828	0.0872	0.18216	NiFe_2O_4	FD3M
	2	2	4	54.1900	54.1537	0.0363	0.06699	NiFe_2O_4	FD3M
	1	1	5	57.5200	57.7364	−0.2164	−0.37622	NiFe_2O_4	FD3M
	0	4	4	63.3700	63.4176	−0.0476	−0.07511	NiFe_2O_4	FD3M
	3	1	5	66.7800	66.6913	0.0887	0.13282	NiFe_2O_4	FD3M
	2	4	4	67.9800	67.7642	0.2158	0.31745	NiFe_2O_4	FD3M
	2	0	6	72.1600	71.9796	0.1804	0.25	NiFe_2O_4	FD3M
	3	3	5	75.2400	75.0749	0.1651	0.21943	NiFe_2O_4	FD3M
	2	2	6	76.3400	76.0963	0.2437	0.31923	NiFe_2O_4	FD3M
	4	4	4	79.7100	80.1409	−0.4309	−0.54058	NiFe_2O_4	FD3M
+6% Li_2O	0	2	2	30.4300	30.4999	−0.0699	−0.22971	NiFe_2O_4	FD3M
	1	1	3	35.7300	35.9294	−0.1994	−0.55807	NiFe_2O_4	FD3M
	2	2	2	37.2600	37.5854	−0.3254	−0.87332	NiFe_2O_4	FD3M
	4	0	0	43.3100	43.6757	−0.3657	−0.84438	NiFe_2O_4	FD3M
	3	1	3	47.8700	47.8270	0.0430	0.08983	NiFe_2O_4	FD3M
	4	2	2	54.1900	54.2048	−0.0148	−0.02731	NiFe_2O_4	FD3M
	1	1	5	57.5200	57.7916	−0.2716	−0.47218	NiFe_2O_4	FD3M
	0	4	4	63.0900	63.4794	−0.3894	−0.61721	NiFe_2O_4	FD3M
	3	1	5	66.7800	66.7571	0.0229	0.03429	NiFe_2O_4	FD3M
	2	4	4	67.9800	67.8313	0.1487	0.21874	NiFe_2O_4	FD3M
	6	0	2	72.4000	72.0523	0.3477	0.48025	NiFe_2O_4	FD3M
	5	3	3	75.2200	75.1517	0.0683	0.0908	NiFe_2O_4	FD3M
	2	2	6	76.6600	76.1746	0.4854	0.63319	NiFe_2O_4	FD3M
	4	4	4	80.0700	80.2250	−0.1550	−0.19358	NiFe_2O_4	FD3M

It was essential to calculate various parameters that depended on the XRD data in order to complete the majority of the information regarding the structural properties of the analyzed NiFe_2O_4 . The following were some of these criteria: crystallite size (d), lattice constant (a), unit cell volume (V), X-ray density (D_x), stress (ϵ), microstrain (σ), and dislocation density (δ), which are the variables listed in Table 2. Additional criteria were the separation between the ionic radii (r_A , r_B), bond lengths (A-O and B-O), and magnetic ions (L_A and L_B) on the tetrahedral (A) and octahedral (B) sites, whereas based on the XRD data, Table 2 already included all the calculated values for these parameters, this table shows that the values of δ , ϵ , σ , a , L_A , L_B , r_A , r_B , A-O, and B-O of NiFe_2O_4 NPs grew noticeably with the increase in the dopant content. Although the behavior varied

depending on the values of “d” and “Dx”, crystallinity declined, and the crystal grain size increased as the dopant content rose. The production of an intrinsic microstrain and a consequent reduction in crystallite size may be responsible for the broadening of the XRD peak. Due to changes in the lattice parameter imposed by crystal defects and dislocations, the microstrain was established.

Table 2. Lattice parameters of NiFe₂O₄ nanoparticles included in the S1, S2 and S3 samples.

Parameters	S1	Error %	S2	Error %	S3	Error %
d, nm	49.09	0.203	47.96	0.083	46.92	0.170
a, nm	0.82797	0.128	0.82948	0.183	0.8310	0.192
V, nm ³	0.5676	−0.546	0.5707	−0.557	0.5739	−0.565
D _x , g/cm ³	5.4853	0.541	5.4556	0.547	5.4256	0.549
L _A , nm	0.3585	−0.195	0.3592	−0.055	0.3590	0.139
L _B , nm	0.2926	−0.170	0.2931	0.034	0.2930	0.136
A-O, nm	0.1893	−0.158	0.1896	0.052	0.1895	0.101
B-O, nm	0.2136	−0.187	0.2140	0.046	0.2139	0.140
r _A , nm	0.0573	−0.523	0.0576	0.173	0.0575	0.347
r _B , nm	0.0816	−0.490	0.0820	0.121	0.0819	0.366
σ (N/m ²)	4.79×10^7	0.542	5.05×10^7	0.415	5.26×10^7	0.409
ε	7.06×10^{-4}	0.481	7.26×10^{-4}	0.547	7.3×10^{-4}	0.507
δ, Lines/nm	4.1×10^{-4}	0.536	4.32×10^{-4}	0.416	4.5×10^{-4}	0.317

3.2. FTIR Analysis

The majority of the features of spinel materials are based on their cation distribution; understanding this is, therefore, a requirement of this study. By using infrared (IR) analysis, the cation distribution of cubic spinel structures can be investigated. This method frequently reveals two fundamental vibration modes at 600 cm^{−1} and 400 cm^{−1}, respectively, which are related to the distribution of cations at tetrahedral (A-) and octahedral (B-) sites. The S1, S2, and S3 sample room-temperature FTIR spectra, in the 4000–400 cm^{−1} region, are shown in Figure 2. Study of this figure reveals that the virgin sample exhibited two main absorption bands (ν_1 and ν_2) at 867–595 cm^{−1} and 550–400 cm^{−1}. These bands confirm the formation of the spinel-type structure, as shown as in Table 3. However, these bands could be attributed to the metal–oxygen (M–O) bond-stretching vibrations, i.e., Fe–O and Ni–O vibrations at both A- and B-sites involved in the spinel ferrite. In other words, these bands confirm that Ni and Fe cations can be distributed between A- and B-sites. In this regard, the authors expect that two small bands (ν_1^* and ν_2^*) located at 867 cm^{−1} and 550 cm^{−1}, may be related to Ni–O vibrations at both A- and B-sites, indicating the formation of random spinel structures. These findings, therefore, refer to the fabrication of random spinel structures. Furthermore, the position of these bands shifted when lithium, in varying amounts, was added to the virgin sample, showing that lithium ions were incorporated into the nickel ferrite lattice. Due to the doping with 3 and 6 mol% Li₂O, respectively, two shoulder bands (ν_1^{**}) were observed at 760 cm^{−1} and 767 cm^{−1}. These bands may indicate Li–O vibration. In addition, the stretching and bending vibrations of the hydroxyl groups (O–H) for the adsorbed water molecules on the sample surfaces may be responsible for the bands at 3433.6–3428.8 cm^{−1} and 1636.3–1633.4 cm^{−1}. The blending of the samples with KBr media causes the adsorption of water molecules on their surfaces [22]. Nevertheless, the bands at 2924.6–2923.52, 1385.60–1384.64, and 1049.10–1044.26 cm^{−1} could be attributable to the stretching vibrations of hydrogen carbon and hydroxyl carbon (C–H and C–O–H). These bands are dependent on the existence of carbon traces that originated from the egg-white internal combustion process [23].

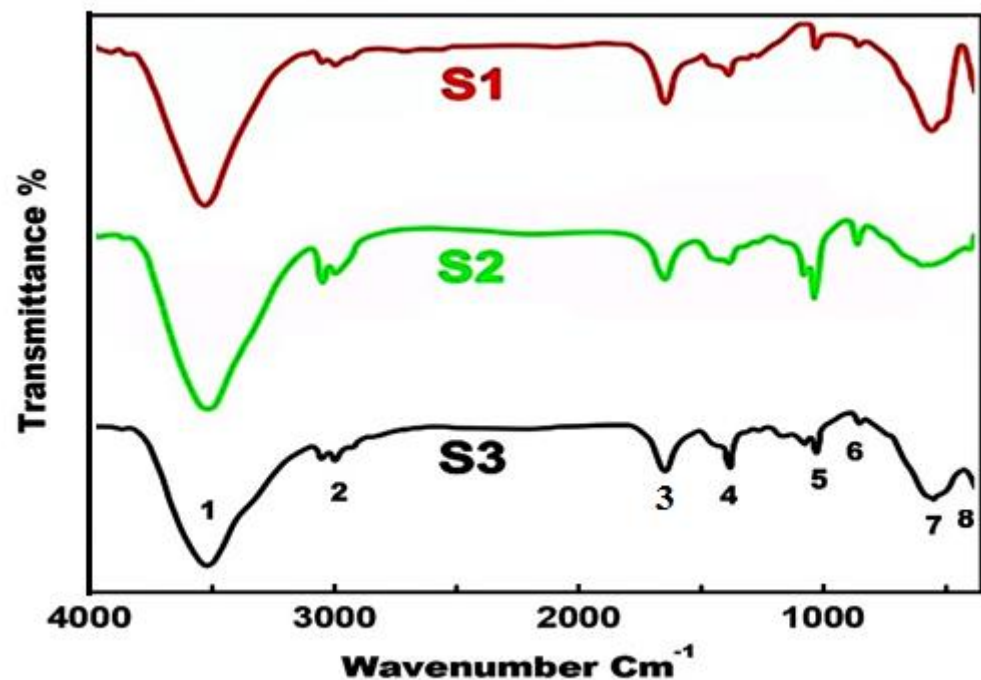


Figure 2. FTIR of the S1 and S2 and S3 samples.

Table 3. FTIR bands of NiFe_2O_4 nanoparticles included in the S1, S2 and S3 samples.

	S1			S2			S3		
	$\nu \text{ cm}^{-1}$	T %		$\nu \text{ cm}^{-1}$	T %		$\nu \text{ cm}^{-1}$	T %	
Peak No 1	3433.64	84.97		3423.99	60.64		3428.81	68.37	
Peak No 2	2923.56	96.17		2925.48	88.23		2924.52	90.21	
Peak No 3	1633.41	93.18		1636.30	87.24		1636.31	87.81	
Peak No 4	1385.60	95.45		1385.60	90.43		1384.64	88.32	
Peak No 5	1044.26	97.58		1049.10	83.13		1046.19	91.93	
Peak No 6	890.20	96.20		880.34	94.53		875.21	97.20	
Peak No 7	640.13	92.01		620.09	90.13		582.39	82.20	
Peak No 8	400.21	86.11		450.34	85.53		413.65	83.99	

3.3. TEM Analysis

Transmission electron microscopy, TEM, must be employed to establish the impact of lithia doping on the microstructure of virgin nickel ferrite. The particle-size distribution of pure (S1) and 6 mol% Li_2O -doped NiFe_2O_4 particles (S2) are shown in TEM, high resolution (HR)-TEM, and selected area electron diffraction (SAED) pictures, respectively. Analysis of these graphs show that the morphology of the S3 sample changed significantly compared with that of the pristine S1 sample, as received (TEM photographs, Figures 3a and 4a). The microstructural analysis of the S1 sample shows that as-received particles are almost spherical with some agglomerations. On the other hand, irregular spherically shaped particles wholly disappeared, and instead, irregularly cubic particles emerged as shown in the S3 sample. This observation confirms significant changes in the microstructure of NiFe_2O_4 as a result of the doping process. Generally, the TEM micrographs of the investigated samples indicate that the distribution of grains is of nonuniform size. Different factors, including the diffusion coefficient and the concentration of dissimilar ions, might be responsible for this fluctuation in grain diameter [24]. Furthermore, using HR-TEM images obtained from one of numerous crystallites, it is possible to estimate the interplanar spacing of 0.25 nm between adjacent planes, which corresponds to the NiFe_2O_4 (311) lattice plane as illustrated in Figures 3b and 4b. Moreover, Figures 3c and 4c show the SAED pattern which consists entirely of concentric rings containing many differently oriented crystallites. The

diffractogram depicts a regular pattern of bright spots indicating polycrystalline samples, powders or nanoparticles. Indeed, the S1 sample is characteristic, as demonstrated by the uniform intensity distribution along the ring circumference yielding smooth rings, as shown in Figure 3c. However, despite having sufficient crystallinity to produce smooth rings, as seen in Figure 4c, the S3 sample exhibits a non-uniform intensity distribution along the produced rings. Finally, Figures 3d and 4d, respectively, show the histograms of the crystallite size statistical distribution (CSD) for the S1 and S3 samples. It can be seen from these figures that the grain diameter lies in the range of 3 nm–31 nm for the S1 sample and 35 nm–100 nm for the S3 sample, with average particles sizes located at 17 nm and 45 nm for these samples, respectively. In fact, there are differences in size between the XRD and TEM results, particularly in the case of the S1 sample, because the crystallite size is assumed to represent the size of a coherently diffracting domain and is not precisely the same as the particle size. However, due to several agglomerations present, the crystallite size of the S1 sample as determined by XRD patterns is significantly larger than the particle diameter detected by TEM. On the other hand, the S3 sample lithia doping caused the grain boundaries to be visible, indicating processes of redistribution and recrystallization. The high porosity and abundance of pores which formed on the S3 sample grain boundary may be responsible for this behavior.

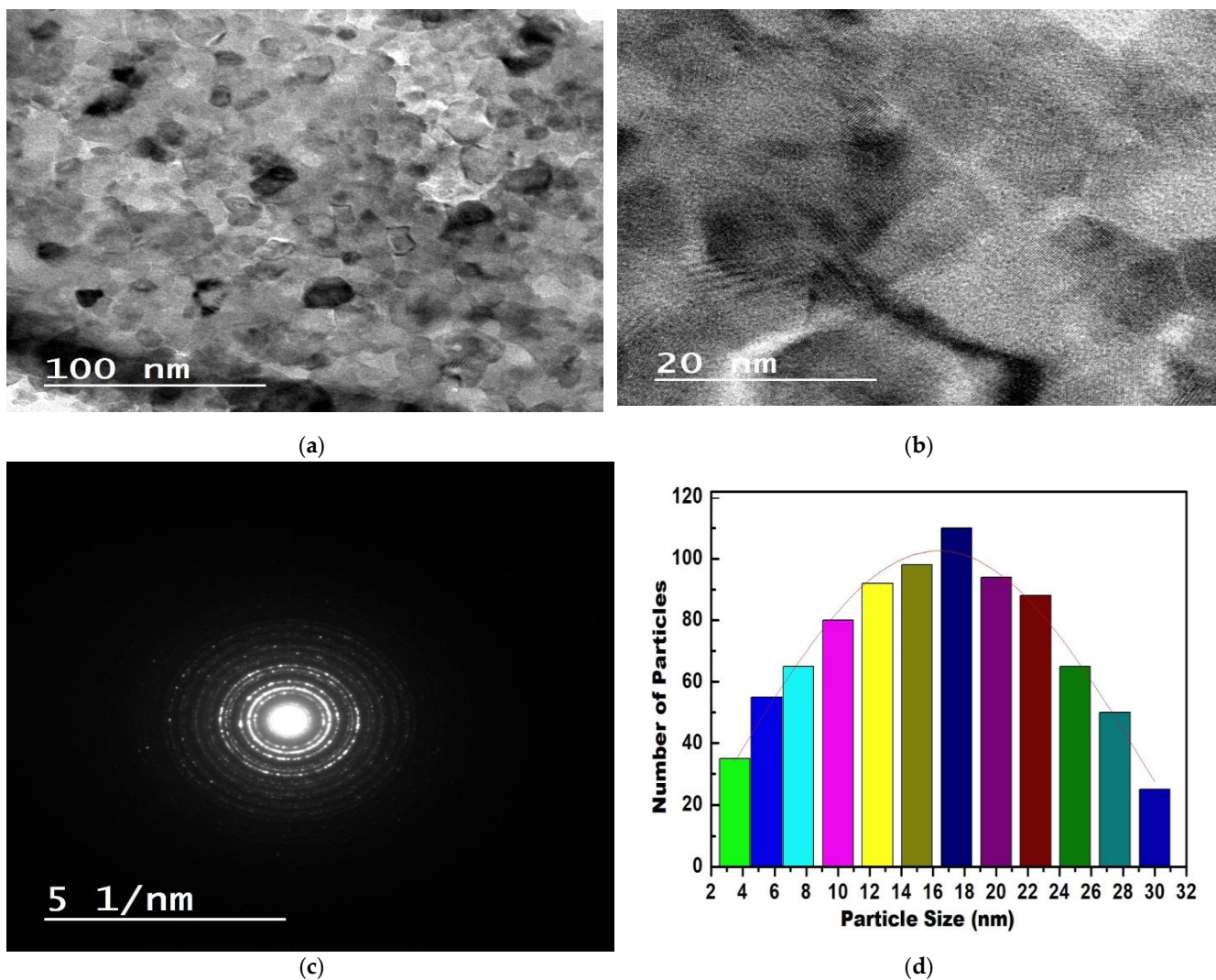


Figure 3. (a) TEM, (b) HRTEM, (c) SAED, and (d) particle size distribution of the S1 sample.

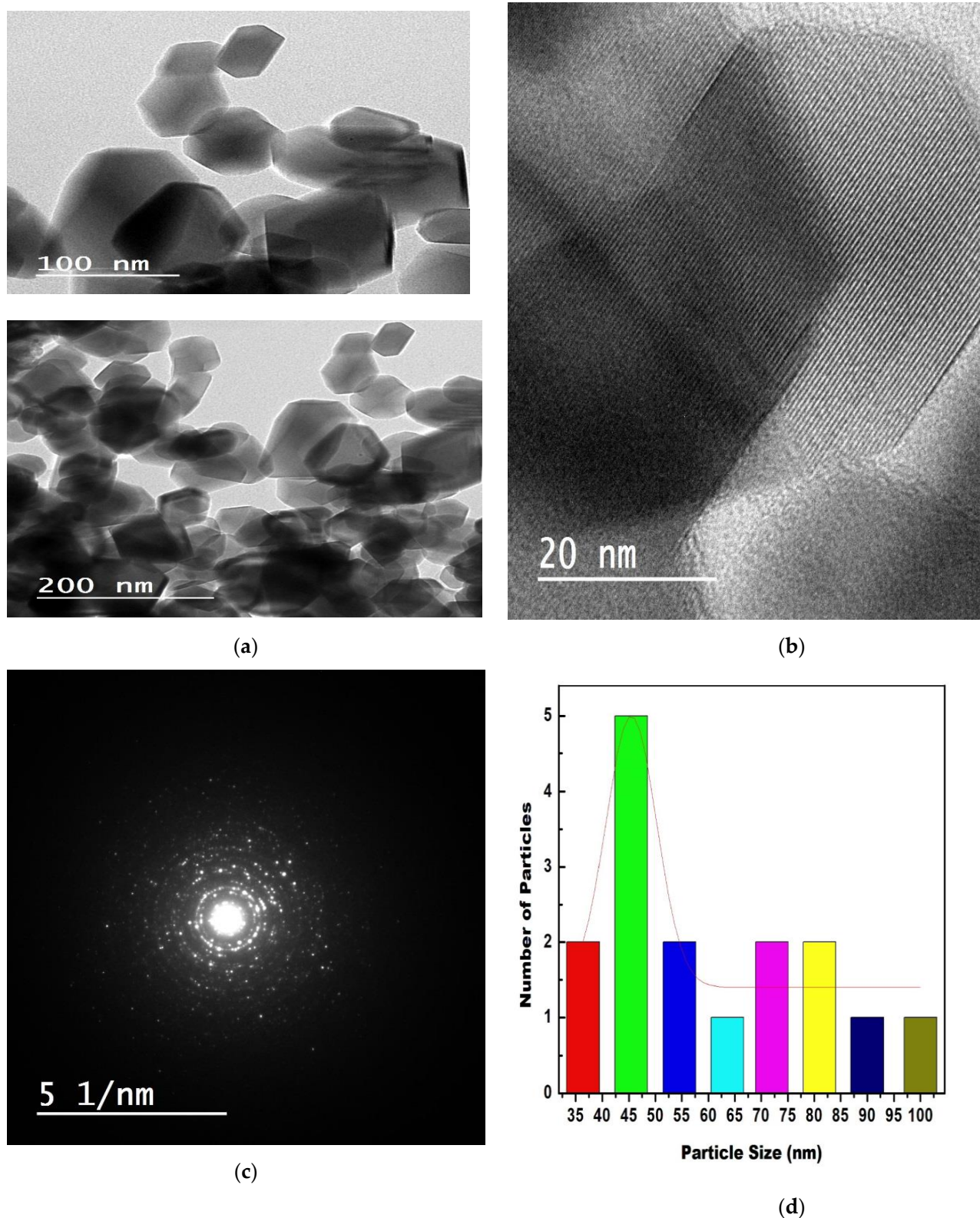


Figure 4. (a) TEM, (b) HRTEM, (c) SAED, and (d) particle size distribution of the S2 sample.

3.4. Textural Characteristics of Virgin and Lithia-Doped Nickel Ferrites

We were able to distinguish between the various textural characteristics of samples S1 and S3 using N_2 -adsorption/desorption isotherms at 77 K. S_{BET} , V_P , V_m , and $\hat{r}$ are shown in these properties for both the S1 and S3 samples. These type II isotherms with a type

H3 hysteresis loop comprise the entirety of Figure 5. Table 4 contains values for S_{BET} , V_p , V_m , and $\bar{r}$; the table shows a modest rise in S_{BET} , V_m , and the other values as a result of the 6 mol% Li_2O doping, as seen by the S3 sample textural characteristics. The total pore volume may have somewhat increased, which would explain this rise. Additionally, Figure 6 displays the pore-size distribution of the S2 and S3 samples. According to this figure, the majority of the holes in the S1 sample are between 2 and 7 nm in size, with a 4.5 nm average. As can be seen, most of the pores in the S2 sample are between 0.5 and 3 nm, with an average pore size of 1 nm.

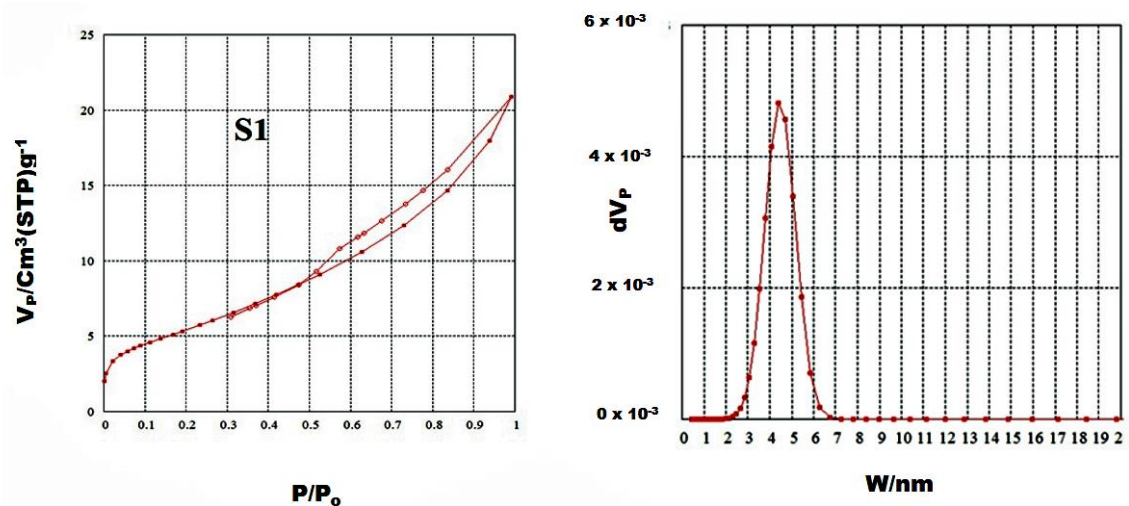


Figure 5. N_2 - adsorption/desorption isotherm and pore size distribution of the S1 sample.

Table 4. Surface properties of the S1 and S3 samples.

Samples	S_{BET} (m^2/g)	Error %	V_p (cm^3/g)	Error %	$\bar{r}$ (nm)	Error %	V_m (nm)	Error %
S1	19.90	0.101	0.0322	0.621	6.465	0.077	4.572	0.043
S3	20.83	0.144	0.0405	0.493	7.769	0.025	4.786	0.062

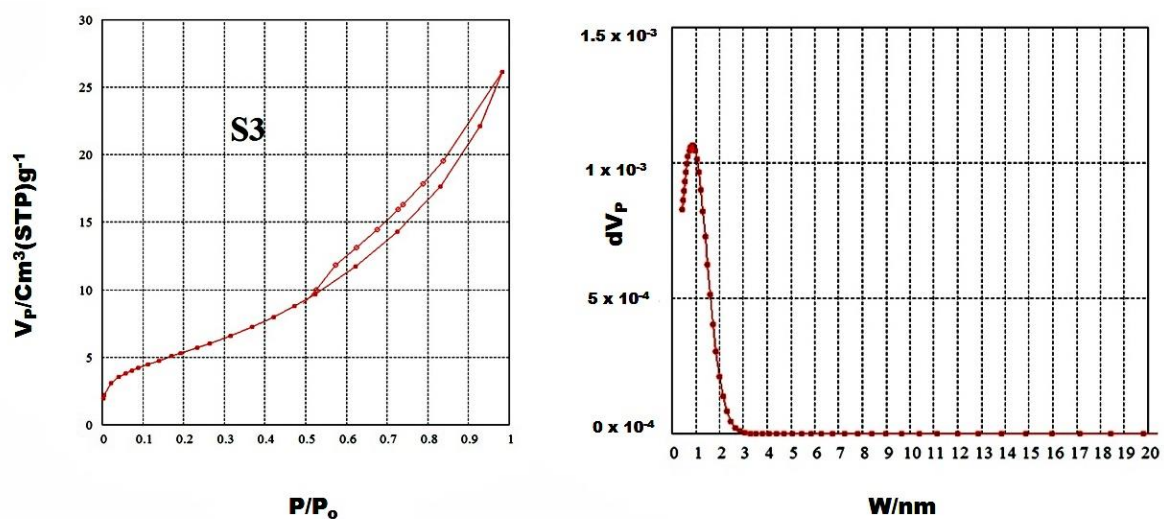


Figure 6. N_2 - adsorption/desorption isotherm and pore size distribution of the S3 sample.

3.5. Magnetic Properties

The size and shape of the hysteresis loop are known to be significantly influenced by the grain size, porosity, and exchange interaction caused by cation dispersion. The magnetic domain is known to be reoriented at the start of an applied field, which causes a rapid increase in magnetization, then becomes sluggish and eventually saturated as a result of the spin revolution. The hysteresis loop used to compute magnetic properties, such as the coercive field (H_c), remanent magnetization (M_r), saturation magnetization (M_s), squareness (M_r/M_s), anisotropy constant (K_a), initial permeability (μ_i), and magnetic moment (μ_m) per unit formula in Bohr magnetons for the tested materials (S1, S2, and S3). These variables are displayed in Table 5. These numbers, corresponding to the properties of magnetism, were extrapolated from the magnetic curves produced in Figure 7. At room temperature, a magnetic field ranging from -20 to +20 kG was applied while these graphs were produced using the VSM method. Based on these data, ferromagnetic property in lithium-doped and undoped Ni ferrite nanoparticles was established. This table demonstrates that as the lithia concentration increases, the values of M_r , M_s , μ_m , and μ_i decrease. On the other hand, the M_r/M_s value decreases in the presence of 3 mol% Li_2O and then increases with 6 mol% Li_2O . The opposite behavior was observed for the K_a value with the previous content of lithia.

Table 5. The magnetic properties of the S1, S2 and S3 samples.

	S1	Error %	S2	Error %	S3	Error %
M_s (emu/g)	31.544	0.012	27.447	0.014	25.510	0.019
M_r (emu/g)	6.613	0.060	5.406	0.073	5.618	0.053
M_r/M_s (emu/g)	0.2096	0.238	0.1969	0.406	0.2202	0.090
H_c (G)	133.99	0.074	175.27	0.039	163.78	0.036
μ_m	1.3249	0.011	1.1527	0.052	1.0714	0.046
μ_i	59.525	0.005	7.054	0.028	7.340	0.027
K_a (erg/cm ³)	4402.69	0.013	5011.08	0.001	4352.11	0.014

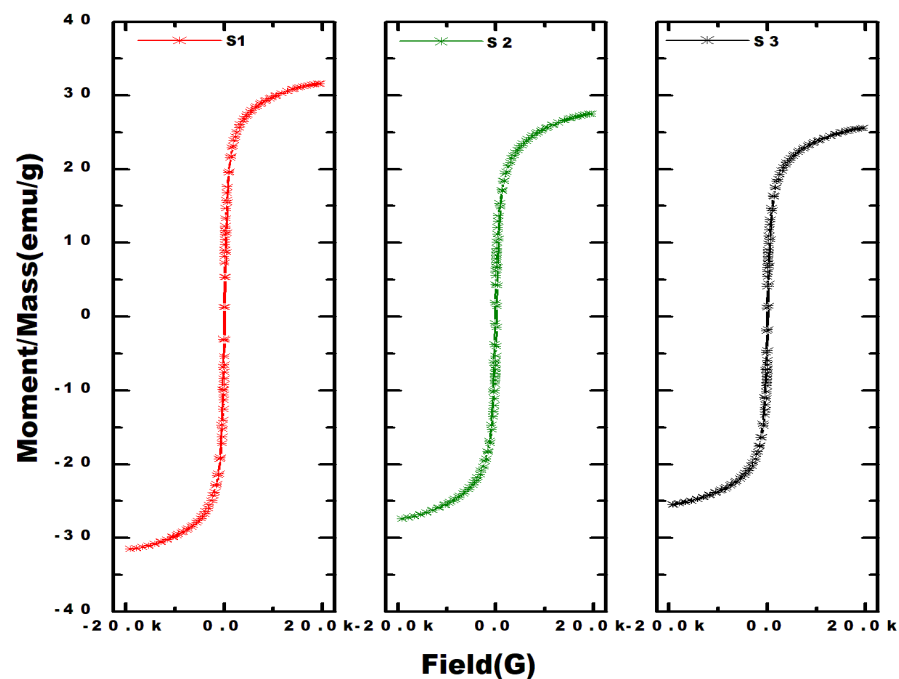


Figure 7. The magnetic hysteresis loops of S1, S2 and S3 at room temperature.

4. Discussion

4.1. Formation of NiFe_2O_4 -Based Mixed or Random Spinel Structures

In this study, fabrication of NiFe_2O_4 NPs using 6 mol egg-white assisted combustion resulted in the formation of random spinel structures. On the other hand, our previous work reported that using 3 mol or 10 mol egg white resulted in the production of inverse spinel nickel ferrite [23]. In other words, the preparation method, especially the egg-white content, affects the cation distribution in spinel nickel ferrite. Indeed, substitution of some Fe^{3+} ions by some Ni^{2+} ions at the A-site involved in NiFe_2O_4 crystallites resulted in the formation of random spinel structures. Because the atomic mass of iron is lower than that of nickel, this displacement led to an increase in the value of D_x from 5.4019 or 5.4769 to 5.4857 for NiFe_2O_4 crystallites prepared by 10, 3, and 6 mol egg white, respectively [23]. The peak height at the (4 4 0) reflection plane was equal to that at the (2 2 0) reflection plane indicating distribution of both Ni^{2+} and Fe^{3+} ions in the A- and B-sites, yielding partially inverse (random) spinel nickel ferrite. In our previous work, the peak heights at these planes were not equal due to the formation of inverse spinel nickel ferrite containing all Ni^{2+} ions at the A-site [24]. However, the FTIR spectra for the virgin sample shows two small bands (ν_1^* and ν_2^*) located at 867 cm^{-1} and 550 cm^{-1} that may be related to Ni–O vibrations

Nevertheless, we must note the following; firstly, the solid-state reaction between Ni and Fe oxides resulted in the formation of NiFe_2O_4 NPs at suitable preparation conditions. In other words, fabrication of NiFe_2O_4 NPs is due to the solid–solid interaction between Ni^{2+} and Fe^{3+} ions in the presence of oxygen. Secondly, one cannot ignore that both NiO and Fe_2O_3 include lattice defects, namely, Fe^{2+} and Ni^{3+} ions, respectively. Both Fe^{2+} and Ni^{3+} ions can be observed at the NiO and Fe_2O_3 interfaces, respectively. These cations cannot participate in the formation of NiFe_2O_4 NPs. The presence of both Fe^{2+} and Ni^{3+} ions must, therefore, be overcome to enhance nickel ferrite formation. The problem of the presence of the Fe^{2+} ions diminish and is almost negligible; this is because of the ease with which these ions pass via contact with, and diffusion through, the NiO surface, and their subsequent interaction with NiO in the presence of oxygen, to form nickel ferrites depending upon the preparation method [23]. The main problem, however, is the presence of nickel (Ni^{3+}) ions that do not participate and even inhibit the formation of nickel ferrites. Hence, this problem being clear, the aim of the current study was to overcome the presence of Ni^{3+} ions which was achieved by Li_2O doping. The treatment of the precursor containing nickel and ferric nitrates with small amounts of lithium nitrate in the presence of a certain amount of egg white resulted in the conversion of Ni^{3+} ions to Ni^{2+} ions which participated in the formation of NiFe_2O_4 NPs. This will be explained in detail based on our results.

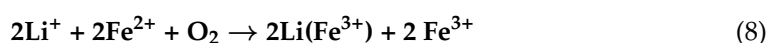
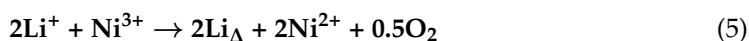
4.2. Cation Distribution of Lithium-Doped NiFe_2O_4 NPs

Most scientists consider the fact that cation distribution affects the distinctive properties of each material. Because of this, they sought to control cation distribution using many different mechanisms, including preparation methods, doping, precursors, and the stoichiometries of constituents. To complement this goal, we studied the effect of Li_2O doping on the type and formation of NiFe_2O_4 NP spinel structure. In fact, Li_2O doping of virgin NiFe_2O_4 crystallites resulted in the appearance of the same diffractograms of NiFe_2O_4 NPs in XRD patterns, with a change in the intensity and position of the diffraction peaks. Furthermore, it was found that the XRD peak can be widened by defects or internal stress and strain.

The Li_2O -based doping process was widely applied for a large number of solids, depending on the history of lithia and preparation conditions [25–27]. Lithium exhibits some affinity for dissolution in both Fe_2O_3 and NiO lattices due to the similarity of its ionic radius with the radii of both Fe^{3+} and Ni^{2+} ions [28–30]. This dissolution can proceed by incorporation or location of Li^+ ions in an interstitial position and/or replacement of a host cation. In fact, the maximum quantity of lithia that can be dissolved in NiO can attain a value of 11 mol% before a lithium–nickel compound is formed [31,32]. It is well known

that non-stoichiometric NiO contains Ni^{3+} ions in interstitial positions as lattice defects. In this study, Li_2O doping of Ni and Fe oxides resulted in the conversion of some Ni^{3+} ions into Ni^{2+} ions which took part in the formation of nickel ferrite, with subsequent lattice expansion. This expansion could be attributed to the difference in the ionic radii of both Ni^{3+} (0.062 nm) and Ni^{2+} (0.078 nm) ions. Accordingly, lithia doping of the precursors containing a mixture of Ni and Fe nitrates with a certain amount of egg white enhances the formation of lithium-doped nickel ferrites.

Furthermore, Li_2O doping increased the unit cell volume and crystallite size of NiFe_2O_4 NPs because it increased the interatomic space, which is dependent on an increase in the lattice according to Vegard's law [33]. The X-ray density decreased when lithia was pre-sent and the unit cell capacity increased. The variation in X-ray density occurs according to the fluctuation in cation distribution within A- and B-sites [34]. The hopping length (L_A and L_B) between the magnetic ions within A- and B-sites increases in the presence of dopant due to substitution of some Fe^{3+} ions by some Ni^{2+} ions at the A-site, with subsequent migration of the substituted Fe^{3+} ions, that have an ionic radius lower than that of Ni^{2+} ions, to the B-site. Similar behavior was observed with the values of ionic radii A-O, B-O, r_A , and r_B . Moreover, the increase in the r_A value is greater than that of the r_B value due to lithia doping depending on the incorporation of Ni^{2+} ions within the A-site, besides its presence in the B-site [34]. Bearing in mind that the Li^+ and Ni^{2+} ions have a high tendency for octahedral (B)-site occupancy, while Fe^{3+} ions are unevenly divided between tetrahedral (A) and octahedral (B) sites, several authors reported the octahedral-site preference of Ni^{2+} , Fe^{3+} , and Li^+ ions in lithium-doped NiFe_2O_4 as $\text{Ni}^{2+} > \text{Li}^+ > \text{Fe}^{3+}$ [35]. However, the obtained results imply that Li ions play important roles at A- and B-sites, yielding redistribution of cations at these sites. The replacement of certain host Ni^{2+} and Fe^{3+} ions and their placement in interstitial sites producing solid solution can both contribute to the dissolution of Li^+ ions in the NiO and Fe_2O_3 lattices participating in the production of nickel ferrite. Kroger's notations [5] can be used in the following ways to streamline the dissolving procedure.



$\text{Li}(\text{Ni}^{2+})$ and $\text{Li}(\text{Fe}^{3+})$ are the monovalent lithium ions located in the positions of host NiO and Fe_2O_3 , respectively; Li_Δ represents lithium ions located in the interstitial positions of nickel and ferric oxide lattices; C.V. represents created cationic vacancies. The cationic vacancies that were created by Reactions (6) and (7) in the lattices of the reacting oxides may increase the mobility of the reacting oxide cations (Ni^{2+} and Fe^{3+}), improving the cation distribution and leading to the production of nickel ferrite. On the other hand, dissolution of lithium ions in nickel and ferric oxide lattices according to Reactions (5) and (8) is expected to increase the number of Ni^{2+} and Fe^{3+} ions via converting Ni^{3+} and Fe^{2+} ions that may be present as lattice defects. Finally, the incorporation of Li^+ ions at A- and B-sites augments the cation distribution based on the previous equations as follows. First, at the B-site, cationic vacancy stimulates the migration of the substituted Ni^{2+} ion by 2Li^+ ions to the A-site, as shown in Equation (6). In addition, the transformation of the Fe^{2+} ion, which is expected to be present, to an Fe^{3+} ion enhances the ferrite formation. Second, at the A-site, cationic vacancy promotes the migration of the substituted Fe^{3+} ion by Li^+ ion to the A-site, as shown in Equation (7).

In fact, most ferrite-based semiconductors contain antisite defects (ADs), which are greatly affected by the precursor history, doping, and preparation method. The change in ADs density resulted in significant modifications to the electronic, morphological, surface, and magnetic properties of ferrites. In NiFe_2O_4 , although the total Fe and Ni atomic

concentrations are well controlled, it is much more difficult to control the distribution of Fe and Ni atoms in the two interleaving lattices (A- and B-sites) during synthesis. In this study, all samples contained a certain number of ADs, where some of the Fe atoms exchanged positions with Ni atoms. Typically, these ADs are randomly distributed, and their number is characterized by the degree of Fe/Ni ordering in the sample, which depends on the content of Li dopant. Indeed, when NiFe_2O_4 contains ADs, some Fe occupy Ni sites (Fe_{Ni}) and the same number of Ni atoms occupy Fe sites (Ni_{Fe}), which produces new $\text{Fe}_{\text{Ni}}\text{-O-Fe}$ and $\text{Ni}_{\text{Fe}}\text{-O-Ni}$ bonds as a consequence of this disorder. We observed their effects on the distribution of iron and nickel ions, which was demonstrated through the displacement in the strength and location of the main bands, as shown in Figure 2. Additionally, the formation of oxygen vacancies (O^*) in NiFe_2O_4 , corresponding to the removal of neutral O atoms from the lattice, alters the number of delocalized electrons and also affects the magnitude of the magnetic moments of surrounding atoms.

The population of lithium ions at A- and B-sites involved in spinel nickel ferrite was confirmed by the appearance of two shoulder bands (ν_1^{**}) at 760 cm^{-1} and 767 cm^{-1} due to lithia doping with 3 and 6 mol%, respectively. Moreover, it was seen from the S1 XRD results and FTIR spectra that both Ni and Fe ions were unevenly divided between the A- and B-sites yielding random spinel structure. In addition, Li_2O doping of virgin NiFe_2O_4 crystallites resulted in the appearance of the same functional groups, with a change in the intensity and frequency of the bands. On the other hand, the solubility of lithium ions in different cations (Ni^{2+} and Fe^{3+}) at A- and B-sites resulted in redistribution of these cations, with a subsequent increase in the dislocation density, stress, and microstrain of the NiFe_2O_4 lattice, as observed in Table 2. These changes resulted in slight increases in the morphological and surface properties of NiFe_2O_4 NPs. Indeed, the change in the surface properties of nickel ferrite was monitored due to doping with lithium, depending on the change in total pore volume and the transformation of pore type from mesopores to micro/mesopores. In other words, the majority of pores in the S1 sample are meso-type pores, while the majority of pores in the S3 sample are micro-type pores. One cannot ignore the presence of some mesopores in the S3 sample. These modifications depend on the cation redistribution caused by lithia doping, which was confirmed by FTIR results, as shown in Figure 2.

4.3. Effect of Lithia Doping on Magnetization of NiFe_2O_4 NPs

Variation in the magnetic properties of nano particle ferrite materials depends entirely on the change in cation distribution which can be tuned by preparation method, doping, microstructures, and textural and lattice parameters. Moreover, the magnetization of different spinel ferrites results from variation in the magnetic moments of the ions between and at two sub lattices (A- and B-sites). In other words, the Neel model, which emphasizes A-B exchange interactions over A-A and B-B super-exchange ion interactions, can account for the fluctuation in the M_s value. Virgin and lithium-doped Ni ferrite nanoparticles exhibit different ferromagnetic behaviors, as shown by the S-shaped hysteresis loop [36]. Examination of the magnetic properties of the S1, S2, and S3 samples, tabulated in Table 4, revealed the following. Firstly, lithia doping of virgin nickel ferrite results in a slight decrease in both the value of M_r and M_s due to a decrease in particle size. However, the magnetic dilution of spinel nickel ferrite by lithia doping could be attributed to dissolution of nonmagnetic lithium ions in the crystal lattice. Additionally, because the net magnetic moment of the spinel lattice is equal to the difference between the magnetic moments of the A- and B-sites, the net magnetic moment of virgin nickel ferrite decreases as the amount of lithia increases [37]. Consequently, this decrease could be attributed to the replacement of Fe^{3+} ions having magnetic moments of $5\ \mu_B$ with the Ni^{2+} ions having lower magnetic moments of $2\ \mu_B$ at the tetrahedral site, with the subsequent migration of Fe^{3+} ions from the tetrahedral site to the octahedral site depending on the dissolution of lithium ions in the crystal lattice of nickel ferrite. Secondly, the treatment with Li_2O created an increase in the coercivity of NiFe_2O_4 NPs. The range of H_c values depends on numerous factors, such

as morphology, magneto crystalline anisotropy, size distributions, and exchange coupling. In this study, an increase in coercivity is the result of the change in the magnetic anisotropic constant, which may be due to the higher magneto anisotropy of Ni^{2+} ions [38]. Due to Jahn–Teller distortions brought on by Ni^{2+} ions at the A-site of the spinel ferrite system under the influence of 3 mol% Li_2O doping, this behavior was more pronounced in the case of the S2 sample. Thirdly, the investigated ferrites display uniaxial anisotropy rather than cubic anisotropy because the squareness ratio (M_r/M_s) is less than 0.5. In addition, the small squareness values refer to the surface spin-disorder effects resulting from the canted spin on the surface of nanoparticles. Hence, the lower value of this ratio strongly promotes the studied ferrites for their use in high-frequency applications [38]. Finally, the measured total saturation magnetization of NiFe_2O_4 is interpreted as being due to the presence of antisite and cation-excess defects.

5. Conclusions

The authors concentrated their efforts on preparing pure and similar nickel ferrites using an easy, simple, and inexpensive method. In their quest to prepare these ferrites, their aim was to study the effect of doping with lithium oxide on the structural, surface, morphological, and finally, magnetic properties. Accordingly, and from the results obtained, the following conclusions were made:

1. There is a slight decrease in the crystallite size of nickel ferrite, although there is an increase in the lattice constant. This increase could be attributed to the replacement of some ferric cation at the spinel structure-based tetrahedral site with some nickel cations, with the subsequent migration of ferric cations to the octahedral site. In addition, inconsistencies and differences may exist between the size of both the crystallite and the particles because size was determined by Scherrer's formula, the typical or "apparent" crystallite size, which is not always the same as the particle size.
2. XRD results confirm that the as-prepared ferrites belong to the cubic spinel structure NiFe_2O_4 of random type. However, lithia doping of this ferrite led to the shape transformation of particles, becoming cubic shape. Selected area electron diffraction (SAED) images depict a regular pattern of bright spots, indicating polycrystalline samples, powders, or nanoparticles.
3. Lithia doping of virgin nickel ferrite resulted in change in microstructure, with subsequent changes in stress, strain, and dislocation. Slight increases in the surface area and total pore volume were observed with increasing lithia content.
4. Magnetization and coercivity of NiFe_2O_4 increase by increasing the content of lithia-based dopant. The magnetic parameters were strongly affected by particle size and shape, as well as by their distribution, crystallinity and magnetic domain sizes, and micro-strains induced by Li_2O doping. In addition, this doping led to changes in the antisite and cation excess defects, which enhanced the magnetization nickel ferrite.

Author Contributions: Validation was carried out by N.M.D., G.M.A.-S., F.F.A.-F., R.S.A. and O.H.A.-E.; formal analysis, N.M.D., G.M.A.-S. and O.H.A.-E.; systematic evaluation, N.M.D., G.M.A.-S. and O.H.A.-E.; writing—original preparation of the draft, N.M.D. and O.H.A.-E.; resources, N.M.D., G.M.A.-S. and O.H.A.-E.; curation of results, N.M.D., G.M.A.-S. and O.H.A.-E.; visualization; N.M.D. and O.H.A.-E.; supervision; G.M.A.-S., F.F.A.-F. and R.S.A.; project management; and the acquisition of funding. The final, published version of the paper has been read and approved by all authors. All authors have read and agreed to the published version of the manuscript.

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