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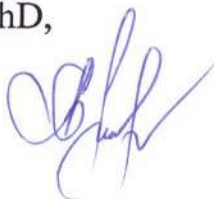
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Мақала

$\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ жаңа күрделі ферритін синтездеу және оның физика-химиялық әрі электрофизикалық зерттеулері

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Аңдатпа:

Бұл мақалада $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ жаңа күрделі феррит материалын синтездеудің физика-химиялық негіздерін сипаттайтын синтез әдісі ретінде золь-гель әдісі қолданылды. Синтезделген ферриттің құрылымы мен құрамы рентгендік фазалық талдау әдісімен анықталды. Талдау нәтижелері бойынша қосылыстың бір фазалы, спинель құрылымды және сингониясы кубтық типке жататыны анықталды.

Қосылыстың микроқұрылымы мен элементтік сандық құрамы сканерлеуші электрондық микроскоп (SEM) көмегімен зерттелді. SEM арқылы $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ типті кристаллиттің әртүрлі бөліктерінен микросуреттер алынып, кристалдардың элементтік құрамы талданды және күрделі ферриттің беткі қабатының жалпы сипаты көрсетілді.

Нәтижесінде қосылыстың бір фазадан тұратыны, оның құрылымының айқындылығы топографиясы мен химиялық құрамы арқылы дәлелденді. Сонымен қатар, синтезделген жаңа күрделі ферриттердің $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ формуласына сәйкес келетіні анықталды. Түзілген бөлшектердің өлшемдері ірі болып, шамамен 50,0 мкм, 20,0 мкм және 10,0 мкм аралығында екені байқалды.

Электрофизикалық өлшеулер 293–483 К температура аралығында және 1,5 пен 10 кГц жиіліктерінде LCR-800 құрылғысында жүргізілді. Жиіліктің 10 кГц-ке дейін артуы зерттелген температура диапазонында (293–483 К) ε мәнінің төмендеуіне әкелетіні анықталды.

Түйін сөздер: золь-гель әдісі; ферриттер; рентгендік талдау; пикнометриялық тығыздық; элементар ұяшық параметрлері; сканерлеуші электрондық микроскоп.

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Article

Synthesis of New Complex Ferrite $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$: Chemical–Physical and Electrophysical Research

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Abstract: In this article, the sol–gel method was used as a synthesis method, which shows the physico-chemical nature of the synthesis of a new complex material, ferrite $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$. The structure and composition of the synthesized ferrite were determined by X-ray phase analysis. According to analysis indicators, it was found that our compound is a single-phase, spinel-structured, and syngony-cubic type of compound. The microstructure of the compound and the quantitative composition of the elements contained within it were analyzed under a scanning electron microscope (SEM). Under a scanning electron microscope, microsystems were taken from different parts of $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ -type crystallite; the elemental composition of crystals was analyzed; and the general type of surface layer of complex ferrite was shown. As a result, given the fact that the compound consists of a single phase, the clarity of its construction was determined by the topography and chemical composition of the compound. As a result, it was found that the newly synthesized complex ferrites correspond to the formula $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$. The particles of the formed compounds have a large size (between 50.0 μm or 20.0 μm and 10.0 μm). Electrophysical measurements were carried out on an LCR-800 unit at intervals of 293–483 K and at frequencies of 1.5 and 10 kHz. An increase in frequency to 10 kHz led to a decrease in the value ε in the range of the studied temperature (293–483 K).

Keywords: sol–gel method; ferrites; X-ray; pycnometric density; elementary cell parameters; scanning electron microscope



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1. Introduction

Research in nanotechnology is a rapidly developing field at the forefront of physics and continues to be of particular interest to the scientific community [1]. Multifunctional materials, in particular, are studied for their effective properties combined in one smart compound [2,3]. A wide range of intelligent materials has been presented, and among them, significant attention has been paid to ferrites, which are considered promoter hosts due to their very prominent features [4,5].

Important results in this category of ceramics generally show excellent physicochemical properties, such as environmental mental health [6], chemical stability [7], high electrical resistance [8], and low price availability [9]. It is worth noting that in order for spinel ferrites to be useful for potential technological applications, a promising road was laid. Some prominent review articles indicate that the use of spinel ferrites for biomedical applications may be unavoidable due to their high biocompatibility [10] and low toxicity [6]. They have also been shown to have special properties for magnetic hyperthermia and magnetic resonance imaging [11,12]. Similarly, many discoveries have confirmed the development of this type of material as a suitable approach for ferrofluidic technology [13], wastewater treatment, catalysts [14], selective sun absorbers [15], and medical instruments [16].

With impressive properties such as conductivity, magnetic behavior, and high-temperature magnetic transition, mixed ferrites are soft ferrimagnetic compounds that show that they already exist in the field of technology. These materials are important due to their careless magnetization in room conditions [17,18].

The widespread use of spinel ferrites is due to their low price, easy preparation, and abundant use in technological and industrial applications [19]. The technological and industrial applications of these magnetic materials are due to their excellent electrical and magnetic properties, which leads to their use in magnetic memory devices, transformers, high-frequency applications, and electronic devices, such as mobile phones, camcorders, laptops, and computers [20].

Many researchers have studied the structural, electrical, and magnetic properties of various Li-based ferrites, such as Li-Mn [21], Li-Ni [22], Li-Co [23], Li-Mg [24], Li-Cr. [25], Li-Zn [26], and Li-Ti [27], which are manufactured by a ceramic method. However, ferrite prepared by the ceramic method involves the synthesis of high calcination temperatures to complete the solid-state reaction between the constituent oxides or carbonates, and the particles are obtained in significantly larger and uneven sizes. This leads to the formation of voids, and then low-density ferrites, when uneven particles are compacted. To overcome these difficulties, the sol–gel method is the preferred method for synthesizing nanoferrite on a volumetric scale by obtaining homogeneous particles, and the most convenient method for synthesizing nanoparticles. This is because of the simplicity of this method; the low-cost precursors; the fact that the annealing process is carried out in a short time; good control of the amount of crystallite; and other properties of materials [28].

Nanoscale manganese ferrites exhibit interesting structural and magnetic properties. They have a wide range of applications, from basic research to industrial applications. In this work, the composition of ferrite nanoparticles is studied.

2. Materials and Methods

The sol–gel method was used as an effective way to synthesize a new mixed ferrite of a complex mixed composition. In order to determine the composition of a new complex mixed ferrite obtained by the sol–gel method, an X-ray phase study was carried out, and in order to conduct quantitative and qualitative analysis, an examination under a scanning electron microscope and a study of electrophysical properties (dielectric conductivity and electrical resistance) were carried out.

As a primary raw material, distilled water of a manganese (III) oxide (“chemical pure”) brand, iron (III) oxide (“chemical pure”) brand, and lithium carbonate (“chemical pure”) brand were used. Trihydric alcohol–glycerin and citric acid were used as gel-forming reagents. The raw materials obtained by stoichiometric calculation were weighed on an analytical balance with a maximum accuracy of 0.0001 g, mixed, ground in an agate sieve, placed in an Alund crucible, and fired in a muffle furnace at a temperature of 1100 °C.

The resulting dried gel was fired in a muffle oven for 10 h at a temperature of 600 °C until a black powder was obtained. Finally, the resulting powder was fired in stages for 7 h at a temperature of 700–1100 °C in the air [29].

The pycnometric density of ferrites was determined by a previously described method [30]. Toluene and distilled water were used as indifferent liquids. The density of composite materials was measured five times, and the average values were calculated.

3. Results and Discussion

3.1. X-Ray Analysis

X-ray analysis was carried out at the Kazakh National Women’s Pedagogical University on a diffractometer Miniflex/600 (Rigaku. Almaty, Kazakhstan). Analysis using a sika beam ($U = 30$ KV, $J = 10$ MA, rotation speed 1000 pulses per second, time constant $t = 5$ s, 2θ , with an angle interval between 5 and 90) was carried out on a Miniflex 600 RIGAKU, and filtered by a filter.

During the X-ray observation of the synthesized mixed complex ferrite, it was observed that the amorphous state of the samples decreased; the crystallization process was complete; and the kinetics of the sol–gel reaction were low. In addition, it is proved from the diffractogram shown in Figure 1 below that the samples changed from an amorphous state to a fully polycrystalline state, and the independent phase was completely formed.

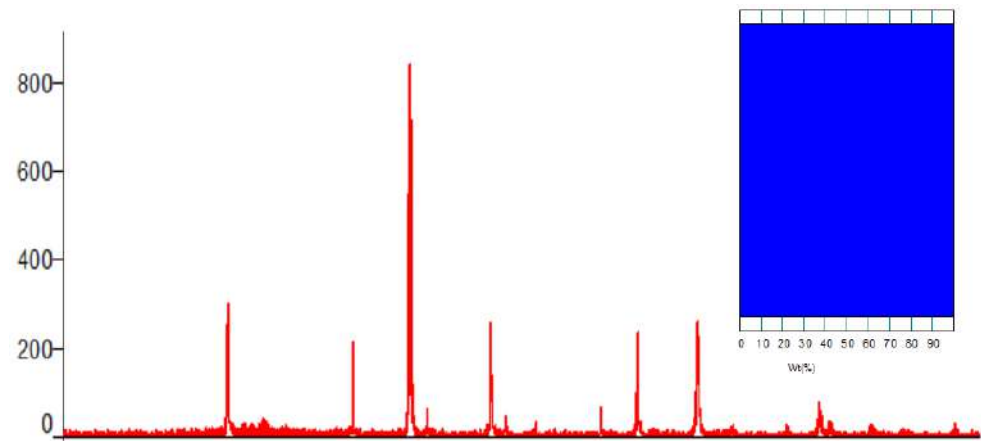


Figure 1. X-ray diffractogram of the complex ferrite $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$. Insert: phase ratio diagram.

Below are the results of the Rietveld method of indexing the diffractogram of the complex ferrite studied by X-ray analysis.

According to Scherrer's formula, the average size of crystallites can be written as follows:

$$d = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where

d —average size of crystals;

K —dimensionless particle shape coefficient (Scherrer constant);

λ —wavelength of X-ray radiation;

β —half-height reflex width (2θ in radians and units);

θ —diffraction angle (Bragg angle).

A new complex mixed ferrite synthesized by the X-ray phase analysis method has the unit number $Z = 8$. $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ is crystallized in a cubic wall-centered cell, and the space group is $Fd\bar{3}m$. Cell parameters: $a = 8.3677$, $b = 8.3677$, and $c = 8.3677$. The average size of crystallites according to Scherrer's formula through the X-ray wavelength of ferrite is $40.6 \mu\text{m}$, and the correctness of the results of the X-ray studies was confirmed by the coincidence of the values of X-ray and pycnometric densities.

3.2. Scanning Electron Microscope Observations

The spectrum of the distribution of the element was studied by a scanning electron microscope (Sam) (Application Note team, Brooker, Berlin, Germany) in order to conduct quantitative and qualitative analysis, and study the percentage content of elements.

A scanning electron microscope (SEM) is designed to obtain a magnified image of an object by scanning it with an electron beam directed at it and recording the signal generated by the interaction of electrons with a detector. The small diameter of the probe, even at low accelerating voltages and high currents, allows for elemental analysis of samples with dimensions of the analyzed area of several tens of nanometers. The beam current detector is located on the microscope column below the aperture of the objective lens, so that the beam current can be monitored at any time during the analysis.

To study the morphology of the surface layer of samples of new complex mixed ferrite synthesized by the sol–gel method, an image of electric diffraction was studied using

an electron microscope scanning luminous microstructures. Electron monographs of the compound taken in an imaging electron microscope are given in Figure 2a–c.

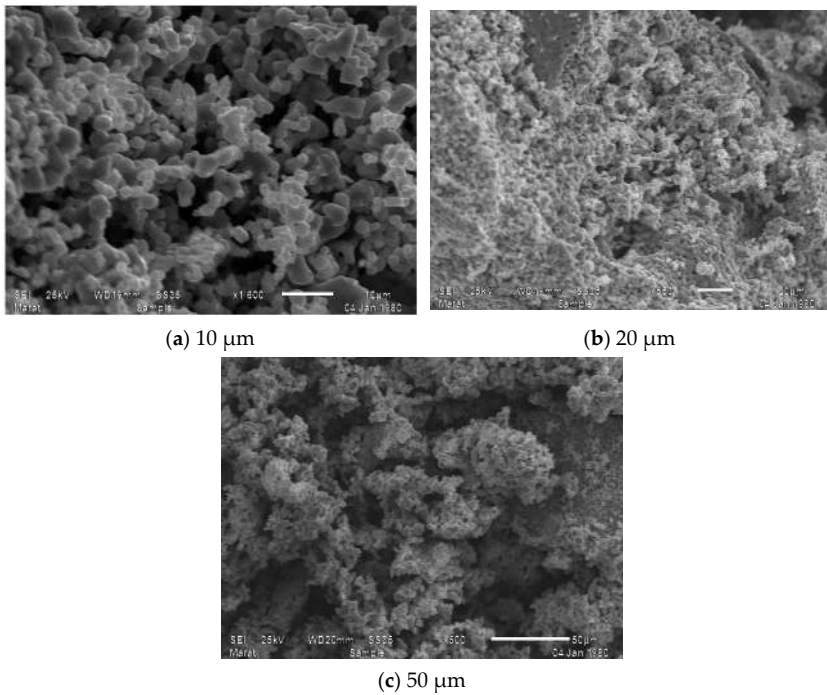


Figure 2. Image of the new mixed complex ferrite $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ measured with three different micrometer accuracies: (a) 10 μm ; (b) 20 μm ; (c) 50 μm .

The above images show the results of micrographs taken at magnifications of 10.0 μm , 20.0 μm , and 50.0 μm , and also show the general appearance of the complex ferrite surface layer. As a result, given that the compound consists of one phase, the clarity of its structure was determined by the topography and chemical composition of the compound. Figure 3 shows the spectrum patterns of the newly synthesized mixed complex ferrite $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ and the elemental analysis results of the $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ compound.

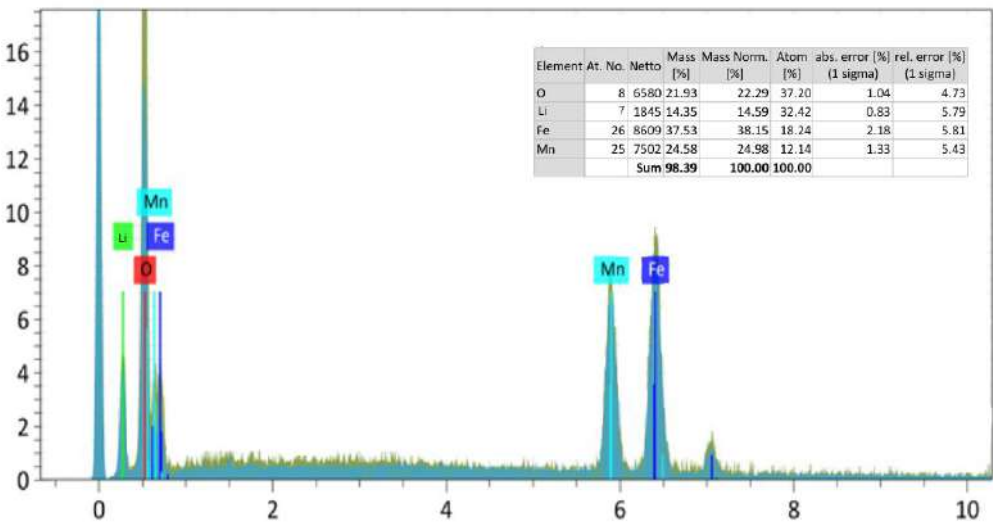


Figure 3. Spectrum samples of the $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ compound. The results of the element analysis are built-in.

Based on the distribution map of elements, on the basis of solving the nature of crystallization, the chemical composition with microstructure and distribution zones of

chromium, iron, lithium, and oxygen atoms were studied. As a result of the numerical elemental composition study, it can be concluded that iron, chromium, lithium metals, oxygen, and carbon atoms are distributed in the 10.0 μm regions (Figure 2a). With an imaging electron microscope, it is possible to obtain nanoscale measurements of solids in powder form.

The spectrum of the distribution of the element was studied using a scanning electron microscope in order to conduct quantitative and qualitative analysis, and study the percentage content of elements. The spectrum samples of the synthesized new mixed complex ferrite and the results of the elemental analysis are shown in Figure 3.

3.3. Electrophysical Research

This was carried out according to methods of measuring electrophysical properties [31,32].

The study of electrophysical properties (dielectric constant and electrical resistance) was carried out by measuring the electrical capacitance of samples in the serial device LCR-800 (Taiwan) in thermostatic mode at an operating frequency of 1 kHz in continuous dry air, with holding time at each set temperature.

Samples parallel to the plane in the form of discs with a diameter of 10 mm and a thickness of 2–6 mm were pre-prepared with a binder mixture (1.5%). Pressing was carried out at a pressure of 20 kg/cm². The resulting discs were fired in the laboratory furnace “SNOL” at a temperature of 400 °C for 6 h. Then, they were carefully leveled on both sides.

The dielectric constant was determined from the electrical capacitance of the sample at certain values of the sample thickness and electrode surface. A Sawyer–Tower circuit was used to obtain the relationship between the electric induction d and the electric field strength E . Visual control of D (hysteresis loop e) was carried out on a C1-83 oscilloscope with a voltage divider consisting of a resistance of 6 M Ω and 700 K Ω and a reference capacitor of 0.15 μF . The frequency of the generator was 300 Hz. In all temperature studies, samples were placed in a furnace; the temperature was measured with a chromium–aluminum thermocouple connected to a voltmeter B2-34 with an error of 0.1 MV. The temperature change rate was 5 K/min. The value of the dielectric constant at each temperature was determined by the following formula:

$$\varepsilon = \frac{C}{C_0} \quad (2)$$

where $C_0 = \frac{\varepsilon_0 \cdot S}{d}$ is the capacitance of the capacitor without the test substance (air).

Calculation of the width of the forbidden zone (ΔE) of the substance under investigation was determined by the following formula:

$$\Delta E = \frac{2kT_1T_2}{0.43(T_2 - T_1)} \lg \frac{R_1}{R_2}, \quad (3)$$

where k is the Boltzmann constant equal to $8.6173303 \times 10^{-5} \text{ eV} \cdot \text{K}^{-1}$; R_1 is the resistance at T_1 ; and R_2 is the resistance at T_2 .

To ensure the reliability of the data obtained, the dielectric constant of a standard substance, barium titanate BaTiO₃, was measured at frequencies equal to 1 kHz, 5 kHz, and 10 kHz. Table 1 below shows the results of measurements of the electrophysical characteristics of BaTiO₃.

As can be seen from the data in Table 2, the values of the dielectric constant 293 K at 1 kHz and 5 kHz correspond satisfactorily to its recommended value of 1400 ± 250 . In addition, observed changes in the electrical conductivity of BaTiO₃ at all frequencies (1 kHz, 5 kHz, and 10 kHz) at 383 K correspond to its transition from the perovskite cubic phase Pm3m to the tetragonal (polar) ferroelectric phase with the space group P4mm [33–35].

Table 1. Symmetry type and unit cell parameters of $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$.

Sample	$\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$
Space group	$Fd\bar{3}m$, cubic side centered
Z	8
Parameter cell (Å)	8.3677(9)
a=	8.3677(9)
b=	8.3677(9)
c=	585.90(11)
V(Å ³)	90
α	90
β	90
γ	40.6 μm
Average size of crystallites according to Scherrer's formula	
X-ray density (g/cm ³)	4.678
Pycnometric density (g/cm ³) *	4.675

* Calculated manually by pycnometer.

Table 2. Dependence of electrical resistance (R), electrical capacity (C), and dielectric constant (ϵ) on BaTiO_3 temperature.

T, K	C, nF	R, Ω	ϵ	lg ϵ	lgR
Measurement frequency at 1 kHz					
293	0.27278	13,400	1296	3.11	4.13
303	0.27426	13,270	1303	3.11	4.12
313	0.27715	12,910	1316	3.12	4.11
323	0.28125	12,560	1336	3.13	4.10
333	0.28772	11,890	1367	3.14	4.08
343	0.29313	11,210	1392	3.14	4.05
353	0.29916	10,290	1421	3.15	4.01
363	0.30751	9383	1461	3.16	3.97
373	0.31202	8831	1482	3.17	3.95
383	0.31702	9061	1506	3.18	3.96
393	0.32255	8814	1532	3.19	3.95
403	0.32967	7881	1566	3.19	3.90
413	0.3423	7098	1626	3.21	3.85
423	0.35119	6902	1668	3.22	3.84
433	0.36668	6153	1742	3.24	3.79
443	0.38018	6317	1806	3.26	3.80
453	0.39802	6010	1891	3.28	3.78
463	0.4169	5584	1980	3.30	3.75
473	0.43147	5149	2050	3.31	3.71
483	0.45456	4656	2159	3.33	3.67
Measurement frequency at 5 kHz					
293	0.25678	29,630	1220	3.09	4.47
303	0.2683	21,650	1274	3.11	4.34
313	0.2775	13,080	1318	3.12	4.12
323	0.28638	5236	1360	3.13	3.72
333	0.29667	4301	1389	3.14	3.63
343	0.30226	4733	1409	3.15	3.68
353	0.30226	3296	1436	3.16	3.52
363	0.30787	2966	1462	3.17	3.47
373	0.31283	2805	1486	3.17	3.45
383	0.31843	2529	1513	3.18	3.40
393	0.32148	2669	1527	3.18	3.43
403	0.32578	3172	1547	3.19	3.50
413	0.32976	4434	1566	3.19	3.65
423	0.33303	6377	1582	3.20	3.80
433	0.33948	9644	1613	3.21	3.98
443	0.35613	11,520	1692	3.23	4.06
453	0.3713	10,430	1764	3.25	4.02
463	0.3925	8021	1864	3.27	3.90
473	0.41682	5978	1980	3.30	3.78
483	0.44245	3799	2102	3.32	3.58

Table 2. Cont.

T, K	C, nF	R, Ω	ϵ	$\lg \epsilon$	$\lg R$
Measurement frequency at 10 kHz					
293	0.11814	152,300	561	2.75	5.18
303	0.18494	70,790	878	2.94	4.85
313	0.22927	32,200	1089	3.04	4.51
323	0.25954	11,870	1233	3.09	4.07
333	0.27501	4842	1306	3.12	3.69
343	0.28531	3312	1355	3.13	3.52
353	0.29302	2689	1392	3.14	3.43
363	0.29988	2257	1424	3.15	3.35
373	0.30652	1946	1456	3.16	3.29
383	0.31215	1689	1483	3.17	3.23
393	0.31667	1737	1504	3.18	3.24
403	0.32294	3130	1534	3.19	3.50
413	0.32779	5945	1557	3.19	3.77
423	0.33406	8231	1587	3.20	3.92
433	0.34256	8805	1627	3.21	3.94
443	0.35658	8052	1694	3.23	3.91
453	0.378	5967	1796	3.25	3.78
463	0.39475	4604	1875	3.27	3.66
473	0.41687	3343	1980	3.30	3.52
483	0.44203	2353	2100	3.32	3.37

It should be noted that despite the decrease in the values of the dielectric constant of BaTiO₃ at a frequency of 10 kHz, and at T equal to 293 K, 303 K, and 313 K, and all values of ϵ BaTiO₃ at all three frequencies (1 kHz, 5 kHz, and 10 kHz) in the range of 313–483 K, there are values up to about 2150, indicating that this frequency change does not particularly affect the temperature dependence of the dielectric constant. BaTiO₃ is in the range of 313–483 K.

Electrophysical measurements of Li_{0.5}MnFe_{1.5}O₄ in the range of 293–483 K and frequencies equal to 1, 5, and 10 kHz were carried out at the LCR installation (Table 3, Figure 4).

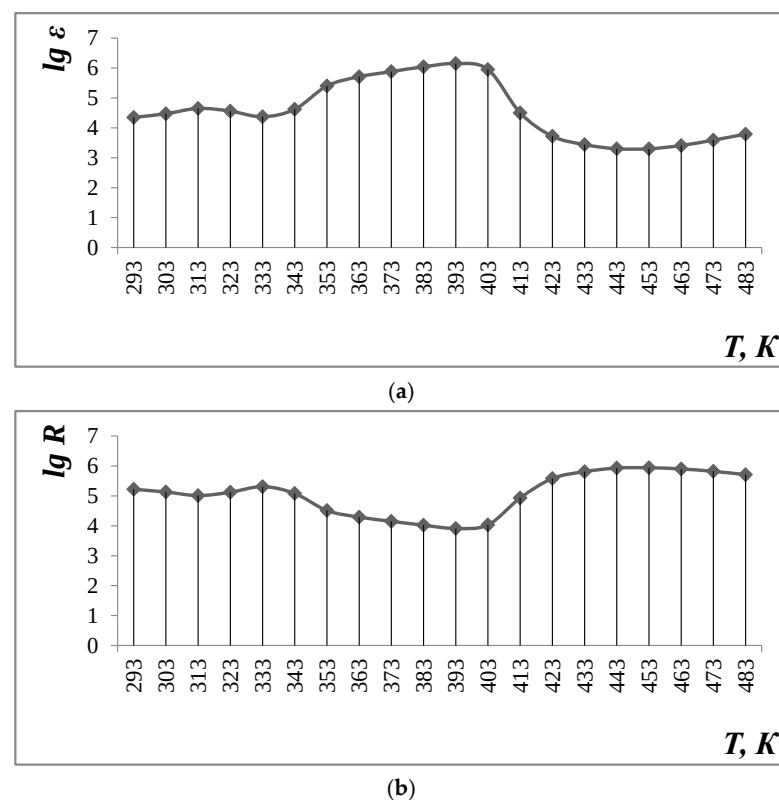


Figure 4. Dependence of dielectric constant (a) and electrical resistance (b) on temperature and frequency equal to 1 kHz.

Table 3. Dependence of electrical capacity (C), electrical resistance (R), and dielectric constant (ϵ) of material 2 on temperature and frequency.

T, K	C, nF	R, Ω	ϵ	lg ϵ	lgR
Measurement frequency at 1 kHz					
293	7.7977	165,700	22,448	4.35	5.22
303	10.302	135,800	29,658	4.47	5.13
313	15.68	101,700	45,140	4.65	5.01
323	12.489	131,900	35,954	4.56	5.12
333	8.1684	203,600	23,515	4.37	5.31
343	14.516	121,200	41,789	4.62	5.08
353	86.933	32,510	250,266	5.40	4.51
363	176.65	19,300	508,547	5.71	4.29
373	265.14	14,110	763,295	5.88	4.15
383	378.56	10,520	1,089,813	6.04	4.02
393	495.27	8184	1,425,802	6.15	3.91
403	312.87	10,720	900,702	5.95	4.03
413	11.039	85,500	31,779	4.50	4.93
423	1.8105	379,400	5212	3.72	5.58
433	0.95809	649,900	2758	3.44	5.81
443	0.68585	843,600	1974	3.30	5.93
453	0.70009	874,900	2015	3.30	5.94
463	0.89737	796,100	2583	3.41	5.90
473	1.3483	666,100	3882	3.59	5.82
483	2.1329	511,600	6140	3.79	5.71
Measurement frequency at 5 kHz					
293	1.3926	140,100	4009	3.60	5.15
303	1.8886	115,400	5437	3.74	5.06
313	2.728	89,690	7853	3.90	4.95
323	1.517	128,100	4367	3.64	5.11
333	0.9064	184,900	2609	3.42	5.27
343	2.5719	93,070	7404	3.87	4.97
353	15.47	29,090	44,536	4.65	4.46
363	31.031	17,900	89,333	4.95	4.25
373	45.909	13,060	132,165	5.12	4.12
383	64.608	9799	185,996	5.27	3.99
393	82.944	7702	238,782	5.38	3.89
403	40.501	11,420	116,596	5.07	4.06
413	1.7403	79,660	5010	3.70	4.90
423	0.28075	293,500	808	2.91	5.47
433	0.13233	455,500	381	2.58	5.66
443	0.09429	533,500	271	2.43	5.73
453	0.09084	555,900	262	2.42	5.74
463	0.10483	536,400	302	2.48	5.73
473	0.13985	477,500	403	2.60	5.68
483	0.19866	401,900	572	2.76	5.60
Measurement frequency at 10 kHz					
293	0.62512	126,300	1800	3.26	5.10
303	0.85581	103,700	2464	3.39	5.02
313	1.188	83,510	3420	3.53	4.92
323	0.50394	127,500	1451	3.16	5.11
333	0.3238	169,200	932	2.97	5.23
343	1.2082	76,460	3478	3.54	4.88
353	7.2162	26,640	20,774	4.32	4.43
363	14.584	16,680	41,985	4.62	4.22
373	21.676	12,440	62,402	4.80	4.09
383	30.192	9304	86,918	4.94	3.97
393	39.355	7426	113,297	5.05	3.87
403	14.483	12,940	41,694	4.62	4.11
413	0.64942	83,960	1870	3.27	4.92
423	0.12128	243,700	349	2.54	5.39
433	0.06145	329,700	177	2.25	5.52
443	0.04865	351,900	140	2.15	5.55
453	0.04694	362,500	135	2.13	5.56
463	0.05137	360,400	148	2.17	5.56
473	0.06369	339,200	183	2.26	5.53
483	0.08415	306,100	242	2.38	5.49

The data in Table 3 and Figure 4 show that the value of the dielectric constant (ϵ) reaches a maximum value at 383 K, which is equal to $1.43 \cdot 10^6$ (1 kHz). Then, it decreases

to 6140 at 483 K (frequency 1 kHz). Increasing the frequency to 5 and 10 kHz leads to a decrease in ϵ in the entire temperature range under study (293–483 K).

The study of the temperature dependence of electrical resistance shows the complex nature of conductivity: at $\Delta T = 293$ –313 K—semiconductor, at $\Delta T = 313$ –333 K—metallic, at 333–393 K—semiconductor, at $\Delta T = 393$ –453 K—metallic, and at $\Delta T = 453$ –483 K—again, semiconductor.

The band spacing of 2 materials AT $\Delta T = 293$ –313 K (Table 4) and 333–393 K (Table 5) is equal to 1.91 and 1.19 eV (narrowband semiconductor), respectively, and AT $\Delta T = 453$ –483 K (Table 6) is equal to 3.04 eV (broadband semiconductor).

Table 4. Calculation of the width of the forbidden zone (ΔE) in the range 293–313 K.

T, K	lg R
293	5.22
313	5.01
$\Delta E = \frac{2 \times 0.000086173 \times 293 \times 313}{0.43(313 - 293)} \lg \frac{5.22}{5.01} = 1.91 \text{ eV}$	

Table 5. Calculation of the width of the forbidden zone (ΔE) in the range 333–393 K.

T, K	lg R
333	5.31
393	3.91
$\Delta E = \frac{2 \times 0.000086173 \times 333 \times 393}{0.43(393 - 333)} \lg \frac{5.31}{3.91} = 1.19 \text{ eV}$	

Table 6. Calculation of the width of the forbidden zone (ΔE) in the range 453–483 K.

T, K	lg R
453	5.94
483	5.71
$\Delta E = \frac{2 \times 0.000086173 \times 453 \times 483}{0.43(483 - 453)} \lg \frac{5.94}{5.71} = 3.04 \text{ eV}$	

4. Conclusions

Summing up the results of the study, a new mixed complex ferrite with the composition $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ was synthesized for the first time by the sol–gel method. In order to determine the composition of the resulting new complex mixed ferrite, an X-ray phase study was carried out, and in order to conduct quantitative and qualitative analysis, an examination under a scanning electron microscope and a study of electrophysical properties (dielectric conductivity and electrical resistance) were carried out.

For the first time, syngony types and parameters of elementary cells of a complex mixed ferrite synthesized by the method of X-ray phase analysis were determined. $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ (cubic, $a = 8.3677(9)$, $B = 8.3677(9)$, $C = 8.3677(9)$ Å, $Z = 8$, $\text{denX-ray} = 4.678 \text{ g/cm}^3$, $\text{denPycno} = 4.675 \text{ g/cm}^3$); through the X-ray wavelength of ferrite, the average size of crystallites according to Scherrer’s formula is 40.6 μm . The results of radiographic research showed that the synthesized compound is polycrystalline. The accuracy of crystallochemical data is evidenced by a satisfactory correspondence of X-ray and pycnometric densities.

Through a scanning electron microscope, microsystems were taken from different parts of $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ -type crystallite; the elemental composition of crystals was analyzed; and the general type of surface layer of complex ferrite was shown. As a result, given the fact that the compound consists of a single phase, the clarity of its construction was determined

by the topography and chemical composition of the compound. As a result, it was found that the newly synthesized complex ferrites correspond to the formula $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$. The particles of the formed compounds have a large size (between 50.0 μm or 20.0 μm and 10.0 μm). The results of the elemental analysis were presented in the form of a table.

Electrophysical measurements of $\text{Li}_{0.5}\text{MnFe}_{1.5}\text{O}_4$ on the LCR-800 unit were carried out at intervals of 293–483 K and frequencies of 1.5 and 10 kHz.

Given the fact that 293 K is equal to a relatively high value of 2.69×10^5 at 1 kHz, the maximum value of 383 K reached 1.43×10^6 . Next, it was reduced to 6140 at 483 K (frequency of 1 kHz). An increase in frequency to 5 and 10 kHz led to a decrease in the entire test temperature interval (293–483 K).

The study of the dependence of electrical resistance on temperature shows the complex nature of conductivity: $t = 293\text{--}313\text{ K}$ —semiconductor, $t = 313\text{--}333\text{ K}$ —metal, $333\text{--}393\text{ K}$ —semiconductor, $t = 393\text{--}453\text{ K}$ —metal, and $T = 453\text{--}483\text{ K}$ —semiconductor again.

In the future, I will study the temperature dependence and magnetic sensitivity of the magnetization of synthesized complex ferrite in various magnetic fields.

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Мақала

Жаңа спинель типті Li–Cr наноферриттері: құрылымы, морфологиясы және электрлік/диэлектрлік қасиеттері

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Аңдатпа:

Бұл мақалада золь-гель әдісі арқылы синтезделген $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ жаңа күрделі феррит материалының синтезі және физика-химиялық сипаттамалары қарастырылған. Рентгендік дифракция (XRD) талдауы синтезделген қосылыстың бір фазалы, спинель типті құрылымға ие және кубтық симметриялы екенін растады.

Раман спектроскопиясы тербелістік режимдерді зерттеу үшін қолданылып, Fe–O және Cr–O байланыстарына сәйкес келетін шыңдар қосылыстың спинель тәрізді құрылымын қосымша дәлелдеді. Қосылыстың микроқұрылымы мен элементтік құрамы сканерлеуші электрондық микроскопия (SEM) әдісімен зерттелді. $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ кристалдарының әртүрлі аймақтары талданып, біртекті фазаның бар екені анықталды және беттік морфология мен химиялық құрам туралы толық мәлімет алынды.

Синтезделген феррит бөлшектерінің өлшемдері салыстырмалы түрде ірі болып, шамамен 5, 30, 100 және 200 мкм аралығында екені анықталды. Диэлектрлік қасиеттері материалдың сыртқы электр өрісіне жауабын бағалау үшін зерттеліп, оның электрлік зарядтың поляризациясына қабілетті екені көрсетілді. Сыйымдылық пен электр өткізгіштік температура артқан сайын өсетіні анықталды.

Электрофизикалық өлшеулер 293–483 K температура аралығында және 1,5 кГц пен 10 кГц жиіліктерінде LCR-800 құрылғысында жүргізілді. Жиіліктің 10 кГц-ке дейін артуы бүкіл температура диапазоны бойынша диэлектрлік өтімділік (ϵ) мәнінің төмендеуіне әкелді.

Түйін сөздер: золь-гель әдісі; ферриттер; симметрия; рентгендік дифракция; пикнометриялық тығыздық; элементар ұяшық параметрлері; сканерлеуші электрондық микроскоп.

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Article

Novel Spinel Li–Cr Nano-Ferrites: Structure, Morphology, and Electrical/Dielectric Properties

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Abstract

This article reports on the synthesis and physicochemical characterization of a novel complex ferrite material, $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$, prepared via the sol-gel method. X-ray diffraction (XRD) analysis confirmed that the synthesized compound is a single-phase material with a spinel-type structure and cubic symmetry. Raman spectroscopy was employed to investigate the vibrational modes, and the observed peaks corresponding to Fe–O and Cr–O bonds further validated the spinel-like structure of the compound. The microstructure and elemental composition were examined using scanning electron microscopy (SEM). Multiple regions of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ crystals were analyzed, revealing a homogeneous phase and providing detailed insight into the morphology and chemical composition of the surface. The synthesized ferrite particles exhibited relatively large dimensions, with sizes measured at approximately 5, 30, 100, and 200 μm . The dielectric behavior was studied to assess the material's response to an external electric field, demonstrating its capacity for electric charge polarization. Both capacitance and electrical conductivity were found to increase with rising temperature. Electrophysical measurements were conducted using the LCR-800 system over a temperature range of 293–483 K and at frequencies of 1.5 kHz and 10 kHz. An increase in frequency to 10 kHz resulted in a decrease in the dielectric constant (ϵ) across the entire temperature range.

Keywords: sol-gel method; ferrites; symmetry; X-ray diffraction; pycnometric density; unit cell parameters; scanning electron microscope



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1. Introduction

Despite the rapid advancement in the field of materials science in recent years, there are still many unresolved issues. These include materials with perovskite, spinel, ferrite-garnet, and garnet-type structures [1]. Various methods are employed for obtaining multiferroic materials, including sol-gel, high-temperature solid-state synthesis, hot soap, spray pyrolysis, co-precipitation, and combustion techniques [2–12]. Among them, nano-sized ferrite

particles are of particular interest due to their unique properties and wide range of applications. Ferrite materials have been widely used in numerous technological applications, including magnetically controlled drug delivery for cancer therapy, color imaging, gas sensing materials, and catalytic materials [13–20].

Spinel ferrite magnetic ceramics are gaining increasing attention in the electronics industry due to their exceptional properties, including high magnetic permeability, low dielectric losses, moderate saturation magnetization, strong magnetic induction, mechanical hardness, low eddy current losses, and ease of processing [21–27]. The demand for ferrites continues to grow rapidly, driven by their wide range of applications in fields such as biomedicine, targeted drug delivery, electromagnetic wave absorption, catalysis, computer memory systems, magnetic recording, and switching devices [28,29].

Among these, lithium (Li) ferrite stands out as a highly promising material for microwave technologies and is extensively used in electronic and communication systems [30]. Lithium ferrites are regarded as some of the most versatile ferrite materials due to their wide application spectrum. Owing to their excellent rectangular hysteresis loops, they are commonly utilized in data storage systems, switching components, and phase shifters [31–33]. With a relatively high Curie temperature of around 640 °C, lithium ferrites can operate effectively across a broad range of technically important temperatures. At room temperature, lithium ferrites offer better conductivity and higher saturation magnetization than yttrium iron garnet, while also being more cost-efficient. Moreover, they are widely used in lithium-ion batteries [34,35]. Lithium ferrite ($\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$) is also recognized as a valuable material for use in construction and engineering sectors [36].

Among the various synthesis methods, the sol–gel technique is considered particularly effective and straightforward, as it allows for material formation at relatively low agglomeration temperatures. This method offers several benefits, including the use of low-cost precursors, operational simplicity, energy efficiency, and the ability to produce nanoparticles with fine, controlled structures.

The primary aim of this study is to explore the potential of lithium ferrites ($\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$) in a variety of applications, including energy storage electrodes (such as those used in lithium-ion batteries), magnetic core inductors, military stealth technologies, and multilayer components in electronic devices. Accordingly, the investigation of synthesis parameters, X-ray structural analysis, and studies of the thermodynamic, physicochemical, and magnetic properties of these materials remain areas of significant scientific interest. This work presents a comprehensive investigation into the synthesis and characterization of ferrite nanoparticles using various preparation techniques.

2. Results

2.1. X-Ray Diffraction (XRD) Analysis

The XRD pattern of the synthesized $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ complex ferrite was recorded at room temperature. As illustrated in Figure 1, the diffraction results confirm the complete transformation of the sample from an amorphous to a fully polycrystalline state, with the formation of a well-defined single phase. All observed peaks correspond to specific crystallographic planes, indicating the presence of a spinel-type cubic structure with space group $F\bar{4}3m$.

The X-ray analysis also showed that the lattice parameter of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite is influenced by the calcination conditions. The absence of secondary or impurity phases in the diffraction pattern attests to the high purity and structural quality of the synthesized material. The X-ray pattern card number is No. 04-010-5163.

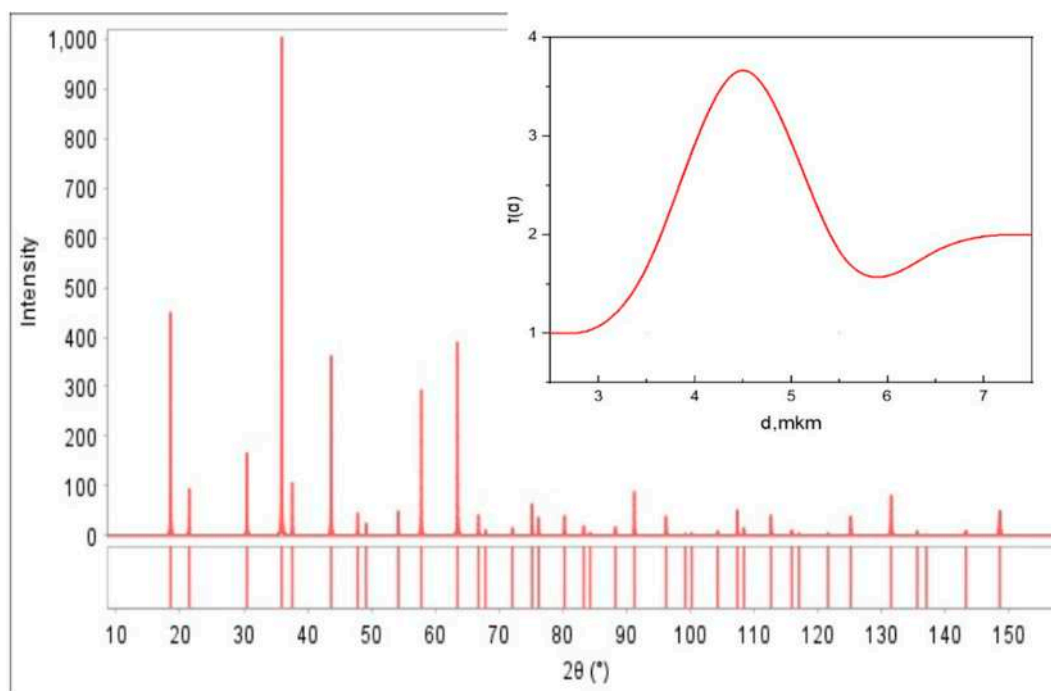


Figure 1. X-ray diffraction pattern of the complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$.

The figure above depicts the unit cell of the crystal structure. A unit cell represents the smallest repeating structural element in a crystalline material. In this case, the cell exhibits a cubic geometry, reflecting the material's high degree of symmetry. Lithium (Li) and Chromium (Cr) atoms are shown in the same color, indicating that they are statistically distributed over identical crystallographic sites.

Lithium atoms are positioned at the tetrahedral sites, each surrounded by four oxygen atoms, while Chromium and Iron (Fe) atoms occupy octahedral sites, where they are coordinated by six oxygen atoms. The lines connecting the atoms represent the chemical bonds that maintain the integrity of the structure, which are predominantly ionic in nature.

The $\text{Fd}\bar{3}\text{m}$ space group is a defining feature of spinel structures. Figure 2 shows the crystal structure of the complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$, which belongs to the $\text{Fd}\bar{3}\text{m}$ space group. In the case of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ compound, this crystallographic symmetry significantly influences its physical and chemical properties by determining the precise atomic arrangement and electronic structure. Such a configuration also has a pronounced effect on the magnetic properties of the material.

Below, the results of the Rietveld refinement based on the X-ray diffraction data for the synthesized complex ferrite are presented.

The reliability of the structural analysis is supported by the close agreement between the X-ray density and the values obtained from pycnometric measurements.

Elevated temperatures can contribute to the removal of structural defects or residual impurities, thereby enhancing crystal quality and promoting the growth of larger crystallites. The average crystallite size was estimated using the Debye–Scherrer equation, which is commonly used to determine crystallite dimensions from X-ray diffraction peak broadening. The Scherrer formula used for this calculation is as follows:

$$d = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where

d —average size of crystals;

K—dimensionless particle shape coefficient (Scherrer constant);
 λ —wavelength of X-ray radiation;
 β —half-height reflex width (2θ in radians and units);
 θ —diffraction angle (Bragg angle).

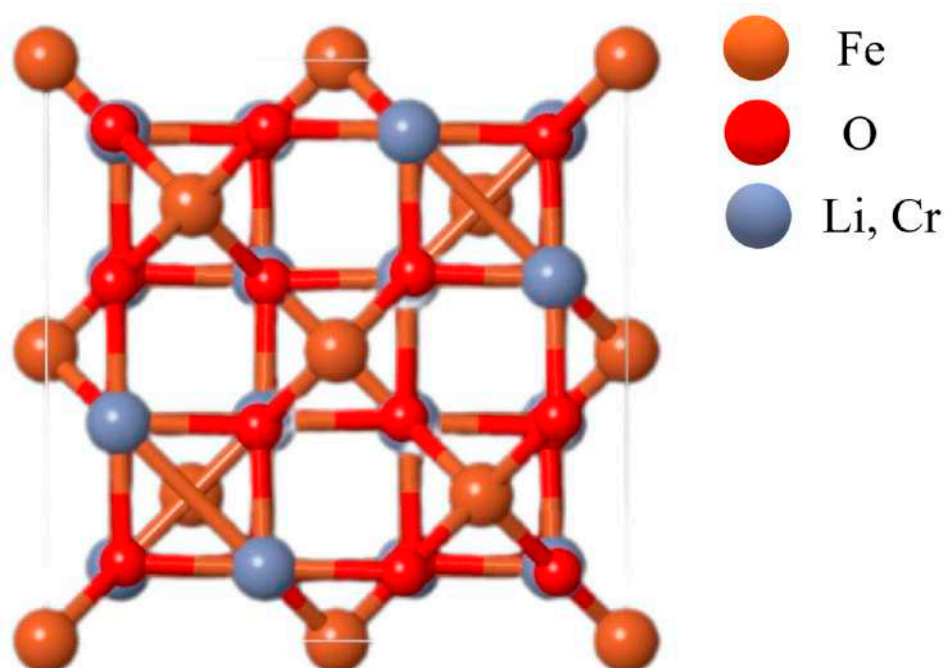


Figure 2. Crystal structure of the complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ with space group $\text{Fd}\bar{3}\text{m}$.

The microstructural characteristics, including crystallite size and microstrain, of the synthesized complex ferrite were determined using the Williamson–Hall (W–H) method. The Williamson–Hall equation is a widely used analytical approach for examining crystal structures and their mechanical properties. It enables the evaluation of microstructural features—specifically, the average crystallite size and lattice strain—based on data derived from X-ray diffraction analysis. The W–H equation is expressed as follows:

$$\beta \cos \theta = \frac{\kappa \lambda}{D} + 4\epsilon \sin \theta \quad (2)$$

where:

β —Full width of the diffraction peak (also called “width”) in radians,
 θ —Diffraction angle,
 κ —A system factor required for the calculation (usually taken as 0.9),
 λ —Wavelength of the X-ray radiation,
 D —Average crystallite size (sometimes referred to as “crystal size”),
 ϵ —Effect of internal strain, i.e., the expansion and compression due to stresses within the crystal.

To determine these parameters, a plot was constructed based on the relationship between $\beta \cos \theta$ and $4\sin \theta$, as illustrated in Figure 3. By applying linear regression to this plot, the slope of the line provides the microstrain, while the y-intercept corresponds to the crystallite size, as summarized in Table 1. The analysis was conducted using the Uniform Deformation Model (UDM), which assumes uniform strain in all crystallographic directions, indicating that the material exhibits isotropic behavior.

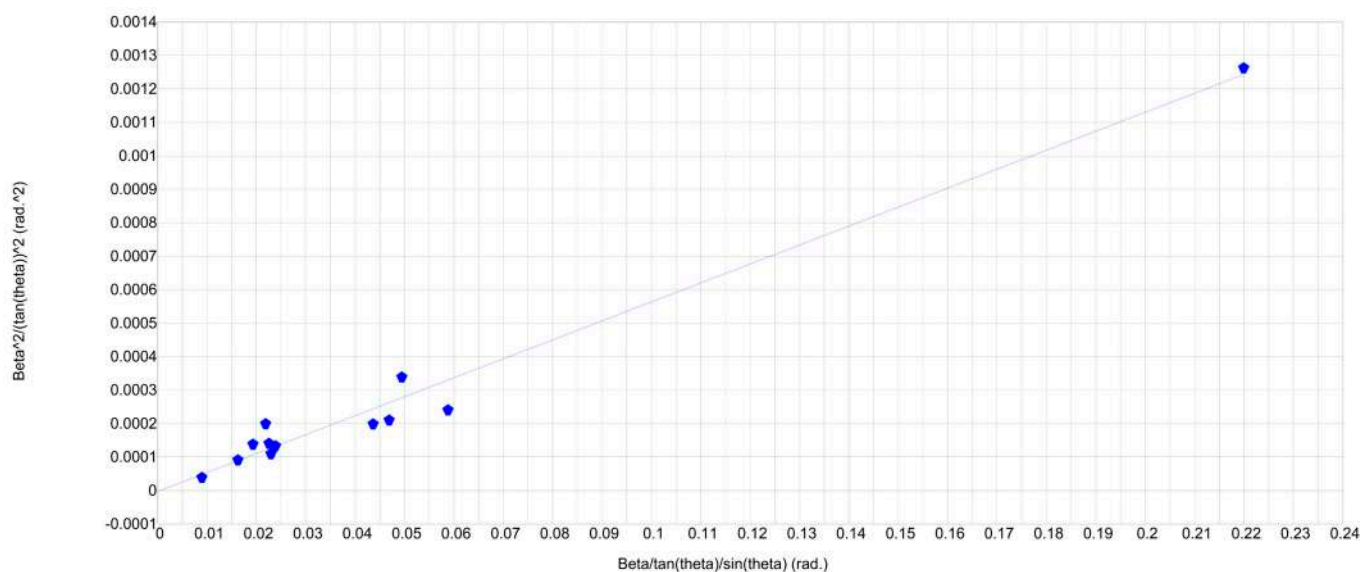


Figure 3. Williamson-Hall (W-H) plot of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ complex ferrite.

Table 1. This is a table. Structural parameters of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ obtained by Rietveld refinement of X-ray diffraction data.

Sample	$\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$
Space Group	$\text{Fd}\bar{3}\text{m}$, the cubic side is centered
Z	4
Cell Parameter (Å)	
a =	8279 Å
b =	8279 Å
c =	8279 Å
V(Å ³)	57.646 Å ³
Agreement factors	
R-factor	0.036
I/Ic	3.67
I/Ic- CW ND	0.76
α	90
β	90
γ	90
X-ray density (g/cm ³)	4.694 g/cm ³
Pycn.density (g/cm ³)	4.695 g/cm ³

The crystallite sizes calculated using the Williamson–Hall method were slightly larger than those obtained from the Scherrer equation. This difference is primarily attributed to lattice strain induced by varying heat treatment temperatures [37–41]. As shown in Table 2, increasing the calcination temperature results in a decrease in lattice strain, which facilitates recrystallization and grain growth, thereby reducing structural defects and internal stresses.

Table 2. Average crystallite size and average strain for all ferrite samples fired at different temperatures, according to the Scherrer and W-H methods.

Sample	Scherrer Method	Williamson-Hall Method	
	D (nm)	D (nm)	$\varepsilon \times 10^{-3}$
$\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$	5.2	9.12	1.71

2.2. Raman Spectroscopy

Raman spectroscopy was employed to analyze the phase structure and composition of the modified samples. This technique identifies the vibrational modes of molecules through molecular spectroscopy.

The measurements were performed using a SPECTRUM TERS system. A solid-state laser with a wavelength of 473 nm and a maximum power output of 35 kW was used for excitation, focusing on a laser spot of approximately 2 μm in diameter on the sample surface. The Raman signal was collected in backscattering geometry using an SSD detector, while the sample was cooled to $-65\text{ }^{\circ}\text{C}$. The spectral resolution of the 600/600 grating was 4 cm^{-1} , with a signal accumulation time of 30 s.

The Y-axis represents normalized intensity, enabling the comparison of relative Raman signal strengths across different spectra. The X-axis indicates the Raman shift in cm^{-1} , which corresponds to the vibrational energy levels of the molecules within the sample.

Figure 4 shows three Raman spectra. Peaks near the 200 cm^{-1} region (at 195, 200, and 206 cm^{-1}) are attributed to lattice vibrations involving Li^+ , Cr^{3+} , or Fe^{3+} cations. In the $500\text{--}600\text{ cm}^{-1}$ range (509 , 511 , 516 , 597 cm^{-1}), the peaks correspond to Fe–O bond vibrations within the spinel lattice. Peaks observed between 600 and 700 cm^{-1} (at 608 , 618 , 650 , 690 , and 705 cm^{-1}) relate to Fe–O vibrational modes in ferrite groups, especially those involving CrO_4^{2-} units. A prominent peak around 720 cm^{-1} signals a high concentration of ferrite groups.

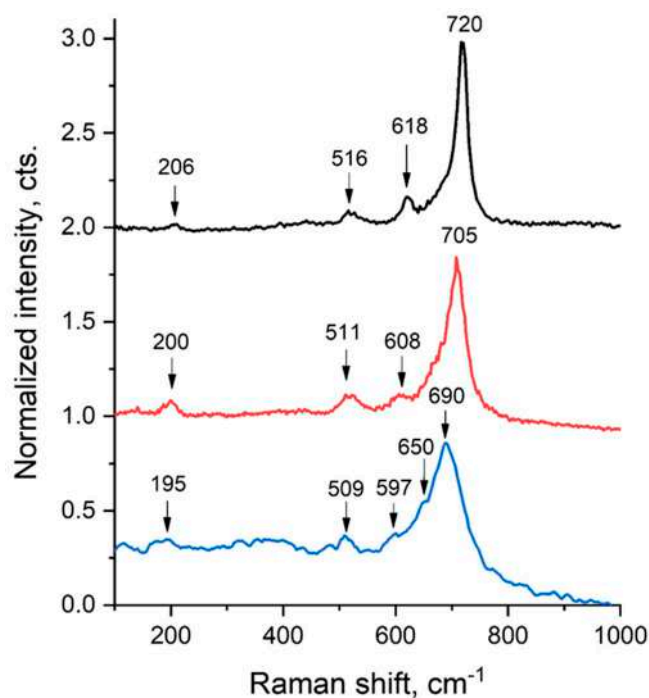


Figure 4. Raman Spectrum of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ Sample.

The detection of characteristic Fe–O and Cr–O vibrational peaks confirms that the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ sample has a spinel-type crystal structure. Variations in peak intensity and position among the three spectra suggest sample heterogeneity or differences in crystallite orientation in different regions. In addition, the colors (black, red, and blue lines) generally represent $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite samples obtained at different calcination temperatures. The blue line corresponds to the sample synthesized at a lower temperature, the red line represents the sample prepared at a medium temperature, and the black line indicates the sample obtained at a higher temperature.

As the temperature increases, the shift and intensity of the peaks change—this indicates the improvement of the crystalline structure, the strengthening of ionic bonds, or possible phase transitions.

Overall, the Raman spectrum of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ sample indicates a complex phase composition, consisting of both spinel structures and ferrite groups.

Raman (or combination scattering) spectroscopy provides a graphical representation of light scattering resulting from its interaction with molecules in a material. While Rayleigh scattering occurs without an energy change, Raman scattering involves a shift in energy corresponding to molecular vibrational modes. This scattering delivers important insights into the chemical composition and structural properties of the material.

As shown in Figure 5, the X-axis (Raman shift, cm^{-1}) represents the change in scattered light energy measured in reciprocal centimeters (cm^{-1}). Each vertex along this axis corresponds to a specific molecular oscillation mode in the sample.

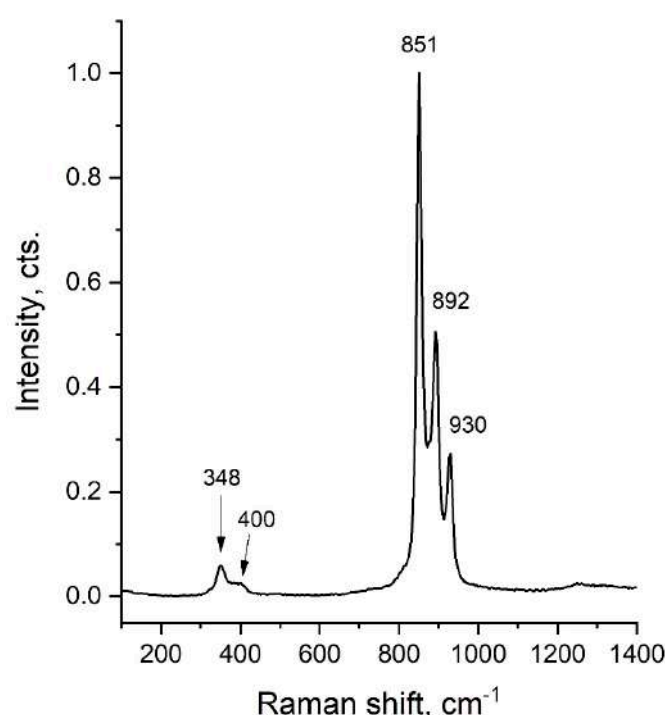


Figure 5. Spectrum of the sample ($\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$) similar to the typical spinel structure signal.

The Y-axis (Intensity, counts) indicates the intensity of the scattered light, which reflects the number of molecules participating in a particular vibrational mode. Higher peak intensities correspond to a larger number of molecules vibrating in that mode.

The spectrum displays several peaks with varying intensities, each representing distinct vibrational modes of the sample's molecules. For instance, the peak at 851 cm^{-1} is the most intense, signifying a dominant vibrational mode. Peaks at 892 cm^{-1} and 930 cm^{-1} , situated near the 851 cm^{-1} peak, correspond to other vibrational modes or different components within the sample. Peaks at 348 cm^{-1} and 400 cm^{-1} have lower intensities, suggesting they arise from less common vibrational modes or impurities present in the sample.

2.3. Scanning Electron Microscope

A scanning electron microscope (SEM) (APPLICATION Note Team, Bruker, Berlin, Germany) was utilized to analyze the distribution spectrum, perform both qualitative and quantitative assessments, and determine the elemental composition by percentage.

To examine the surface morphology of the newly synthesized complex mixed ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ samples prepared via the sol-gel method, an electron microscope capable of scanning electric diffraction images and illuminating microstructures was employed. Various parameters were calculated, all of which aligned well with the X-ray analysis results. The presence of dark regions behind the nanostructures suggests the absence of nano-materials in those areas. Furthermore, the micrographs reveal well-formed single-phase spinel ferrites, which are ideal for applications such as microwave absorption. Electron microscopy images of the compound's surface are shown in Figure 6a–d.

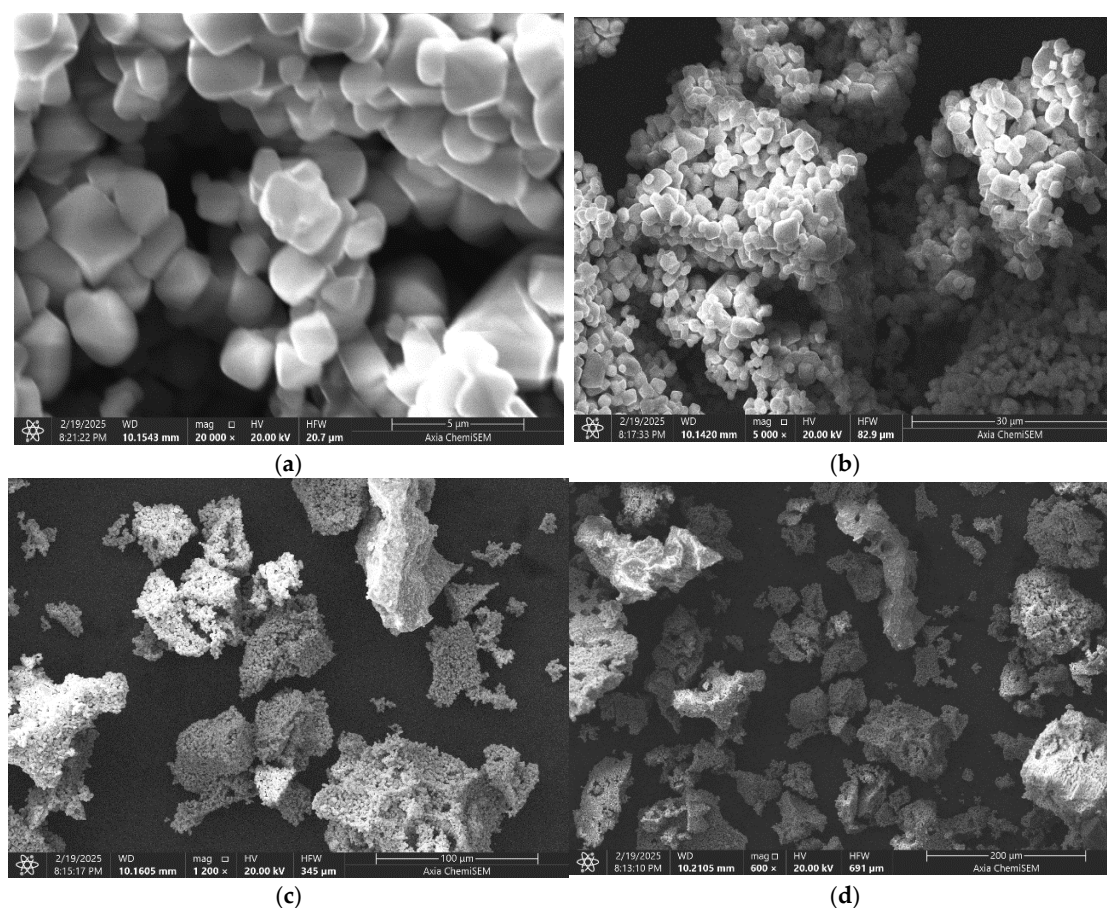


Figure 6. Micrometer-precision images of the new mixed complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ captured at four different magnification scales.

The micrographs taken at magnifications of 5, 30, 100, and 200 μm are presented above, showing the overall morphology of the surface layer of the complex ferrite. The analysis confirmed that the compound is single-phase, with its structural clarity defined by its topography and chemical composition. Figure 7 displays the spectral data and elemental analysis results for the synthesized new mixed complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$.

The microstructure, chemical composition, and elemental distribution of chromium, iron, lithium, and oxygen within the sample were investigated based on the elemental mapping and crystallization characteristics. Quantitative elemental analysis reveals that iron, chromium, lithium, oxygen, and carbon atoms are distributed across regions measuring approximately 100 μm . Scanning electron microscopy also allowed observation of nanometer-sized particles within the powdered solid material.

The elemental distribution, along with quantitative and qualitative analyses, was performed using scanning electron microscopy. Figure 8 presents the spectral data of the synthesized new complex ferrite and the corresponding elemental analysis results.

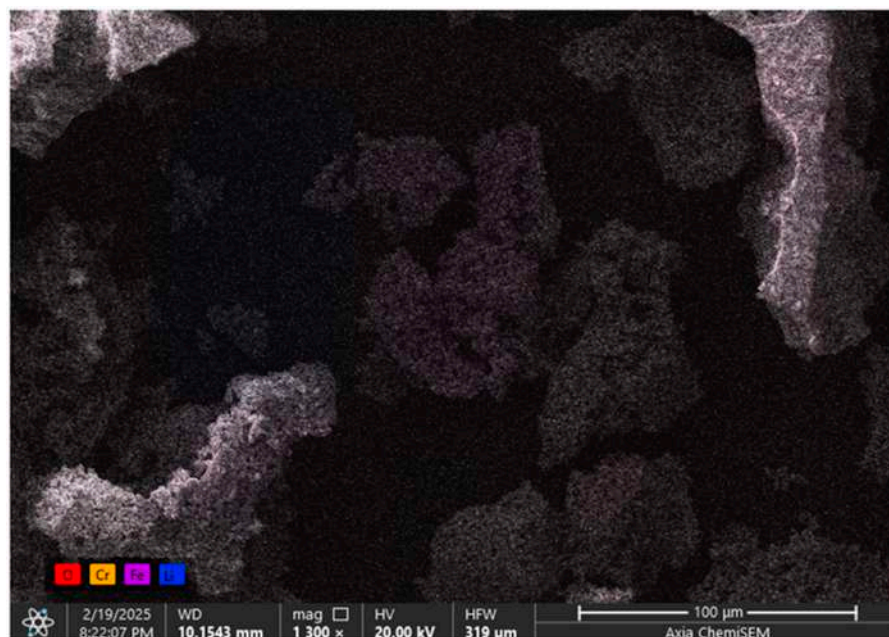


Figure 7. Distribution map of elements in the new mixed complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ (distribution order and color coding of Cr, Fe, Li, O elements in the map).

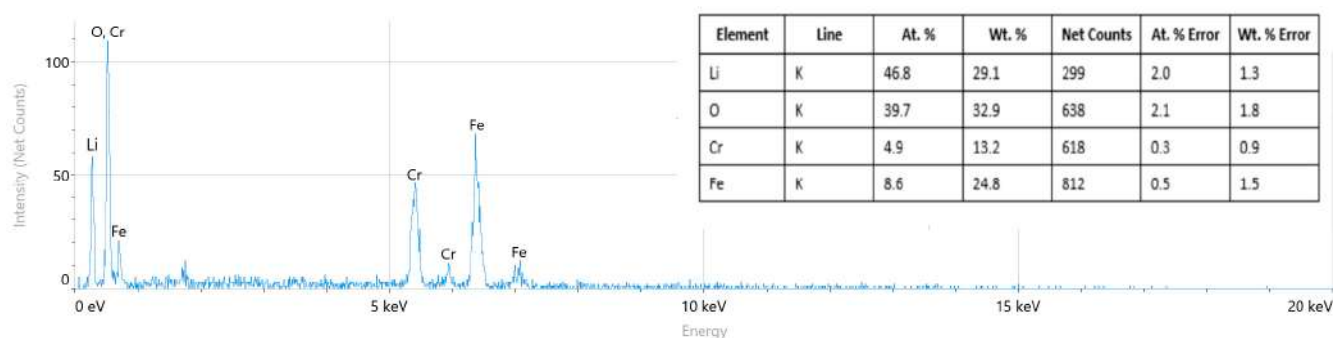


Figure 8. Spectral samples of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ compound. Elemental analysis results are integrated.

The elemental composition and distribution of the synthesized complex ferrite $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ were investigated using energy-dispersive X-ray (EDX) spectroscopy. The obtained EDX spectrum confirmed the presence of the main elements—lithium (Li), chromium (Cr), iron (Fe), and oxygen (O)—consistent with the theoretical stoichiometric composition of the compound.

Quantitative analysis revealed slight deviations in the relative atomic percentages of the elements compared to the theoretical values. These differences can be attributed to the surface sensitivity of the EDX technique and the inherent difficulty in accurately detecting light elements, particularly lithium.

A comparison of theoretical and experimental data showed that the elements are uniformly distributed according to the stoichiometric ratio of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ phase, confirming the homogeneous structure of the synthesized material. In addition, the slightly higher amounts of oxygen and lithium observed in the spectrum may be associated with surface oxidation or lithium accumulation at grain boundaries.

Overall, the EDX analysis confirms that the obtained ferrite forms a single-phase spinel structure with a uniform distribution of elements.

2.4. Broad Band Spectrometer Analysis

The variation of the capacitance (C_p) of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite with frequency at different temperatures is shown in Figure 9. At low frequencies, the capacitance exhibits relatively high values, while it gradually decreases as the frequency increases. This behavior can be attributed to space charge and interfacial polarization effects that dominate at low frequencies, whereas at higher frequencies, the dipoles fail to follow the alternating field, leading to a reduction in polarization. Furthermore, the increase in temperature enhances the mobility of charge carriers and the degree of polarization up to a certain limit, after which further heating results in a decrease in capacitance due to thermal vibrations and structural relaxation effects.

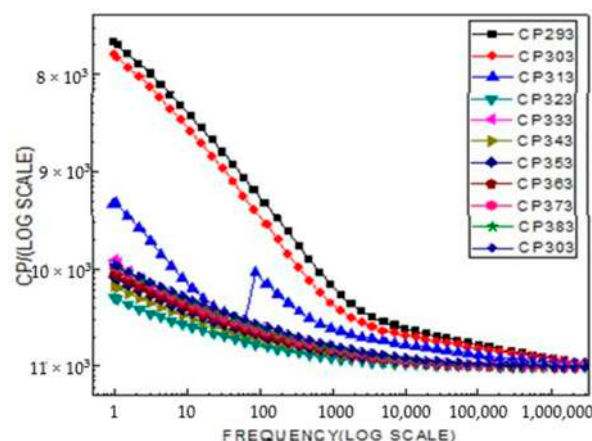


Figure 9. A part from Phil parameter, electrical conduction also increases with increase temperature.

According to the obtained results, the capacitance properties of the complex $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite are strongly temperature-dependent. With increasing temperature, the capacitance (C_p) of the material gradually rises and remains nearly constant up to approximately 303 °C. In this range, the increase in the thermal mobility of charge carriers and the strengthening of dipolar polarization play a key role. However, beyond 303 °C, a sharp decline in capacitance is observed. This phenomenon is associated with a reduction in defect concentration within the crystal lattice, the weakening of intergranular barrier layers, and changes in the relaxation dynamics of charge carriers under high-temperature conditions.

Overall, the obtained results indicate that the dielectric properties of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite are highly sensitive to its structural stability and temperature effects. This suggests that the material holds potential for application in electronic and energy-storage devices operating in high-temperature environments.

The real part of the modulus increases similarly with frequency. The presence of two plateau regions indicates the existence of two distinct relaxation processes in the material, which are reflected by two peaks in the modulus (M) curve. The peak observed in the low-frequency region corresponds to the long-range hopping motion of charge carriers, where charges move from one ionic site to neighboring sites at the grain boundaries. In contrast, the peak appearing in the high-frequency region (observed at 313 K) is associated with short-range hopping motion, where ions are spatially confined and exhibit localized movement within the grains. As the temperature increases, the shift of this peak toward higher frequencies, as shown in Figure 10, suggests that the short-range hopping transport process becomes more dominant at elevated temperatures.

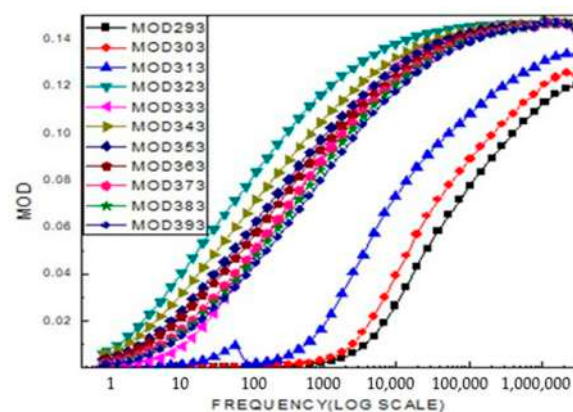


Figure 10. Shows a typical frequency dependent modulus real: $M(\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8)$.

Figure 11 shows that the conductivity of the samples is nearly independent of frequency in the low-frequency region. Their high insulating nature, characterized by low conductivity values ranging from 10^2 to 10^6 S/cm, and the stability of conductivity over a wide range of low frequencies make these samples suitable materials for the fabrication of high-quality resistors.

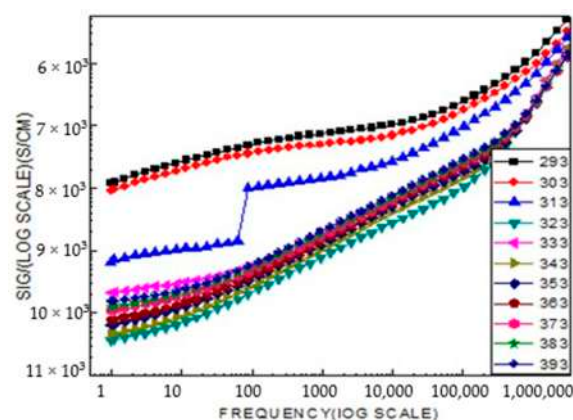


Figure 11. Shows the frequency (f) dependence of the real part σ of a.c. conductivity at selected temperatures in the range 293 K–393 K for the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ sample.

In a wide range of ceramic materials, conduction can occur through ionic conduction, mixed ionic–electronic conduction, or purely electronic conduction, while grain boundaries act as barriers to the movement of charge carriers. This barrier effect is generally more pronounced at lower temperatures (Figure 12). In this temperature range, perovskite-type titanates such as $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ are widely used in capacitor applications as materials with high dielectric permittivity. At temperatures well above the Curie point, $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ -based ceramics exhibit a pronounced effect of highly resistive grain boundaries (GB). This phenomenon is attributed to the space charge effect of a Schottky-type depletion layer. In such applications, titanates must be acceptor-doped to prevent semiconducting behavior in the ferrite materials.

Figure 13 shows the dielectric loss of the material, which is highly sensitive to both frequency and temperature. The shift of the dielectric relaxation peaks toward higher frequencies with increasing temperature clearly indicates that this process is thermally activated.

At low frequencies, the highly resistive grain boundaries exhibit reactive behavior, leading to greater absorption of electrical energy as current flows through the material. The variation in specific resistance with temperature for $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ across frequencies ranging from 1 Hz to 1 MHz is shown above. This behavior is attributed to the fact that

increasing temperature enhances the mobility of charge carriers, while the applied electric field acts as a driving force, aligning the electric dipoles in its direction. The data indicate that specific resistance decreases with increasing temperature and frequency.

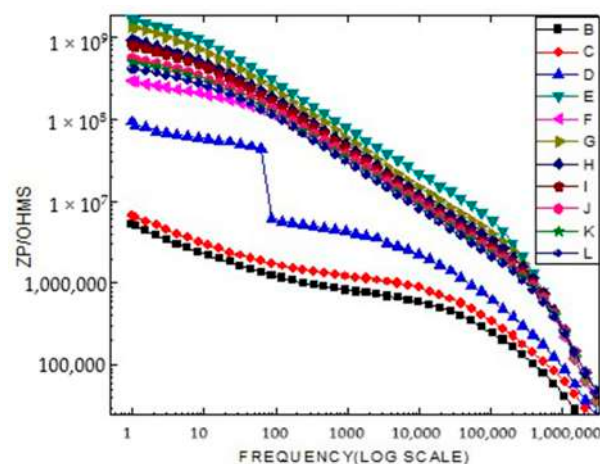


Figure 12. Grain boundaries play an important role in the electrical properties of a variety of ferrite materials and components.

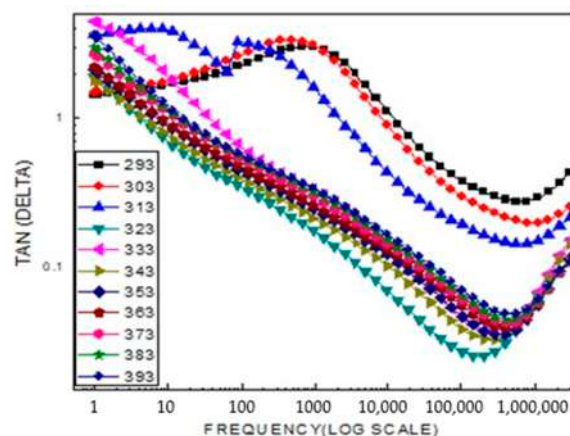


Figure 13. Dielectric loss ($\tan \delta$) show that dielectric loss rapidly decreases with increasing frequency and fall to 1 above 100 Hz.

2.5. Electrophysical Research

The procedure followed the established methods for measuring electrophysical properties [42,43]. The dielectric constant and electrical resistance were investigated by measuring the electrical capacitance of the samples using an LCR-800 serial device (Taiwan) under thermostatic conditions at a working frequency of 1 kHz in a continuous dry air environment, with a holding time at each specified temperature.

The samples were prepared as disc-shaped pellets, parallel to the plane, with a diameter of 10 mm and a thickness ranging from 2 to 6 mm. A binder mixture (1.5%) was added prior to pressing, which was performed at a pressure of 20 kg/cm². The resulting discs were sintered in a SNOL laboratory furnace at 400 °C for 6 h, followed by careful surface leveling on both sides.

The dielectric constant was calculated based on the sample's capacitance, taking into account its thickness and the surface area of the electrodes. A Sawyer–Tower circuit was employed to determine the relationship between electric displacement D and electric field strength E . The hysteresis loop D was visually monitored using a C1-83 oscilloscope,

connected to a voltage divider consisting of resistances of 6 M Ω and 700 k Ω , along with a reference capacitor of 0.15 μ F. The generator frequency was set to 300 Hz.

During all temperature-dependent measurements, the samples were placed inside a furnace. Temperature was monitored using a chromel–alumel (chromium–aluminum) thermocouple connected to a B2-34 voltmeter, with a measurement error of ± 0.1 mV. The heating rate was maintained at 5 K/min. The dielectric constant at each temperature was calculated using the following formula:

$$\varepsilon = \frac{C}{C_0} \quad (3)$$

where C_0 is the capacitance of the capacitor without the test material (in air).

The band gap energy (ΔE) of the material under investigation was calculated using the following formula:

$$\Delta E = \frac{2kT_1T_2}{0.43(T_2 - T_1)} \log \frac{R_1}{R_2}$$

where k is the Boltzmann constant equal to 8.6173303×10^{-5} eV $\cdot$ K $^{-1}$; R_1 is the resistance at T_1 ; and R_2 is the resistance at T_2 .

To verify the accuracy of the obtained data, the dielectric constant of a standard reference material, barium titanate (BaTiO₃), was measured at frequencies of 1 kHz, 5 kHz, and 10 kHz. Table 3 presents the measured electrophysical properties of BaTiO₃.

As shown in Table 3, the dielectric constant values of BaTiO₃ at 293 K for frequencies of 1 kHz and 5 kHz are in good agreement with the recommended value of 1400 ± 250 . Furthermore, the observed changes in the electrical conductivity of BaTiO₃ at all measured frequencies (1 kHz, 5 kHz, and 10 kHz) at 383 K correspond to its phase transition from the cubic perovskite structure (space group Pm3m) to the tetragonal (polar) ferroelectric phase (space group P4mm) [44–46].

Table 3. Dependence of electrical resistance (R), electrical capacity (C), and dielectric constant (ε) on BaTiO₃ temperature.

T, K	C, nF	R, O Ω	ε	lg ε	lgR
Measurement frequency at 1 kHz					
293	0.27278	13,400	1296	3.11	4.13
303	0.27426	13,270	1303	3.11	4.12
313	0.27715	12,910	1316	3.12	4.11
323	0.28125	12,560	1336	3.13	4.10
333	0.28772	11,890	1367	3.14	4.08
343	0.29313	11,210	1392	3.14	4.05
353	0.29916	10,290	1421	3.15	4.01
363	0.30751	9383	1461	3.16	3.97
373	0.31202	8831	1482	3.17	3.95
383	0.31702	9061	1506	3.18	3.96
393	0.32255	8814	1532	3.19	3.95
403	0.32967	7881	1566	3.19	3.90
413	0.3423	7098	1626	3.21	3.85
423	0.35119	6902	1668	3.22	3.84
433	0.36668	6153	1742	3.24	3.79
443	0.38018	6317	1806	3.26	3.80
453	0.39802	6010	1891	3.28	3.78
463	0.4169	5584	1980	3.30	3.75
473	0.43147	5149	2050	3.31	3.71
483	0.45456	4656	2159	3.33	3.67

Table 3. *Cont.*

T, K	C, nF	R, Ω	ε	lgε	lgR
Measurement frequency at 5 kHz					
293	0.25678	29,630	1220	3.09	4.47
303	0.2683	21,650	1274	3.11	4.34
313	0.2775	13,080	1318	3.12	4.12
323	0.28638	5236	1360	3.13	3.72
333	0.29667	4301	1389	3.14	3.63
343	0.30226	4733	1409	3.15	3.68
353	0.30787	3296	1436	3.16	3.52
363	0.31283	2966	1462	3.17	3.47
373	0.31843	2805	1486	3.17	3.45
383	0.32148	2529	1513	3.18	3.40
393	0.32578	2669	1527	3.18	3.43
403	0.32976	3172	1547	3.19	3.50
413	0.33303	4434	1566	3.19	3.65
423	0.33948	6377	1582	3.20	3.80
433	0.35613	9644	1613	3.21	3.98
443	0.3713	11,520	1692	3.23	4.06
453	0.3925	10,430	1764	3.25	4.02
463	0.41682	8021	1864	3.27	3.90
473	0.44245	5978	1980	3.30	3.78
483		3799	2102	3.32	3.58
Measurement frequency at 10 kHz					
293	0.11814	152,300	561	2.75	5.18
303	0.18494	70,790	878	2.94	4.85
313	0.22927	32,200	1089	3.04	4.51
323	0.25954	11,870	1233	3.09	4.07
333	0.27501	4842	1306	3.12	3.69
343	0.28531	3312	1355	3.13	3.52
353	0.29302	2689	1392	3.14	3.43
363	0.29988	2257	1424	3.15	3.35
373	0.30652	1946	1456	3.16	3.29
383	0.31215	1689	1483	3.17	3.23
393	0.31667	1737	1504	3.18	3.24
403	0.32294	3130	1534	3.19	3.50
413	0.32779	5945	1557	3.19	3.77
423	0.33406	8231	1587	3.20	3.92
433	0.34256	8805	1627	3.21	3.94
443	0.35658	8052	1694	3.23	3.91
453	0.378	5967	1796	3.25	3.78
463	0.39475	4604	1875	3.27	3.66
473	0.41687	3343	1980	3.30	3.52
483	0.44203	2353	2100	3.32	3.37

It is worth noting that although a decrease in the dielectric constant of BaTiO₃ was recorded at 10 kHz and temperatures of 293 K, 303 K, and 313 K, the dielectric constant (ε) values across the full temperature range of 313–483 K remain as high as approximately 2150 at all three frequencies (1 kHz, 5 kHz, and 10 kHz). This indicates that frequency variation within this range has little impact on the temperature dependence of the dielectric constant for BaTiO₃.

Electrophysical measurements for LiCr_{3.4}Fe_{1.6}O₈ were performed using the LCR setup over the temperature range of 293–483 K at frequencies of 1, 5, and 10 kHz (see Table 4 and Figure 14).

The data presented in Tables 4 and 5 and Figure 14 indicate that the dielectric permittivity (ϵ) of material 1 at 293 K is relatively high, measured at 2.69×10^5 , and it reaches a peak value of 2.47×10^6 at 373 K. As the temperature further increases to the range of 423–483 K, the ϵ values decrease significantly, dropping to 122 at 483 K. Additionally, increasing the frequency to 10 kHz causes a reduction in ϵ throughout the entire temperature range (293–483 K).

Table 4. Dependence of electrical capacity (C), electrical resistance (R), and dielectric constant (ϵ) of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ on temperature and frequency.

T, K	C, nF	R, Ω	ϵ	$\text{Lg}\epsilon$	lgR
Measurement frequency at 1 kHz					
293	7.7977	165,700	22,448	4.35	5.22
303	10.302	135,800	29,658	4.47	5.13
313	15.68	101,700	45,140	4.65	5.01
323	12.489	131,900	35,954	4.56	5.12
333	8.1684	203,600	23,515	4.37	5.31
343	14.516	121,200	41,789	4.62	5.08
353	86.933	32,510	250,266	5.40	4.51
363	176.65	19,300	508,547	5.71	4.29
373	265.14	14,110	763,295	5.88	4.15
383	378.56	10,520	1,089,813	6.04	4.02
393	495.27	8184	1,425,802	6.15	3.91
403	312.87	10,720	900,702	5.95	4.03
413	11.039	85,500	31,779	4.50	4.93
423	1.8105	379,400	5212	3.72	5.58
433	0.95809	649,900	2758	3.44	5.81
443	0.68585	843,600	1974	3.30	5.93
453	0.70009	874,900	2015	3.30	5.94
463	0.89737	796,100	2583	3.41	5.90
473	1.3483	666,100	3882	3.59	5.82
483	2.1329	511,600	6140	3.79	5.71
Measurement frequency at 5 kHz					
293	1.3926	140,100	4009	3.60	5.15
303	1.8886	115,400	5437	3.74	5.06
313	2.728	89,690	7853	3.90	4.95
323	1.517	128,100	4367	3.64	5.11
333	0.9064	184,900	2609	3.42	5.27
343	2.5719	93,070	7404	3.87	4.97
353	15.47	29,090	44,536	4.65	4.46
363	31.031	17,900	89,333	4.95	4.25
373	45.909	13,060	132,165	5.12	4.12
383	64.608	9799	185,996	5.27	3.99
393	82.944	7702	238,782	5.38	3.89
403	40.501	11,420	116,596	5.07	4.06
413	1.7403	79,660	5010	3.70	4.90
423	0.28075	293,500	808	2.91	5.47
433	0.13233	455,500	381	2.58	5.66
443	0.09429	533,500	271	2.43	5.73
453	0.09084	555,900	262	2.42	5.74
463	0.10483	536,400	302	2.48	5.73
473	0.13985	477,500	403	2.60	5.68
483	0.19866	401,900	572	2.76	5.60

Table 4. Cont.

T, K	C, nF	R, O _M	ε	Lgε	lgR
Measurement frequency at 10 kHz					
293	0.62512	126,300	1800	3.26	5.10
303	0.85581	103,700	2464	3.39	5.02
313	1.188	83,510	3420	3.53	4.92
323	0.50394	127,500	1451	3.16	5.11
333	0.3238	169,200	932	2.97	5.23
343	1.2082	76,460	3478	3.54	4.88
353	7.2162	26,640	20,774	4.32	4.43
363	14.584	16,680	41,985	4.62	4.22
373	21.676	12,440	62,402	4.80	4.09
383	30.192	9304	86,918	4.94	3.97
393	39.355	7426	113,297	5.05	3.87
403	14.483	12,940	41,694	4.62	4.11
413	0.64942	83,960	1870	3.27	4.92
423	0.12128	243,700	349	2.54	5.39
433	0.06145	329,700	177	2.25	5.52
443	0.04865	351,900	140	2.15	5.55
453	0.04694	362,500	135	2.13	5.56
463	0.05137	360,400	148	2.17	5.56
473	0.06369	339,200	183	2.26	5.53
483	0.08415	306,100	242	2.38	5.49

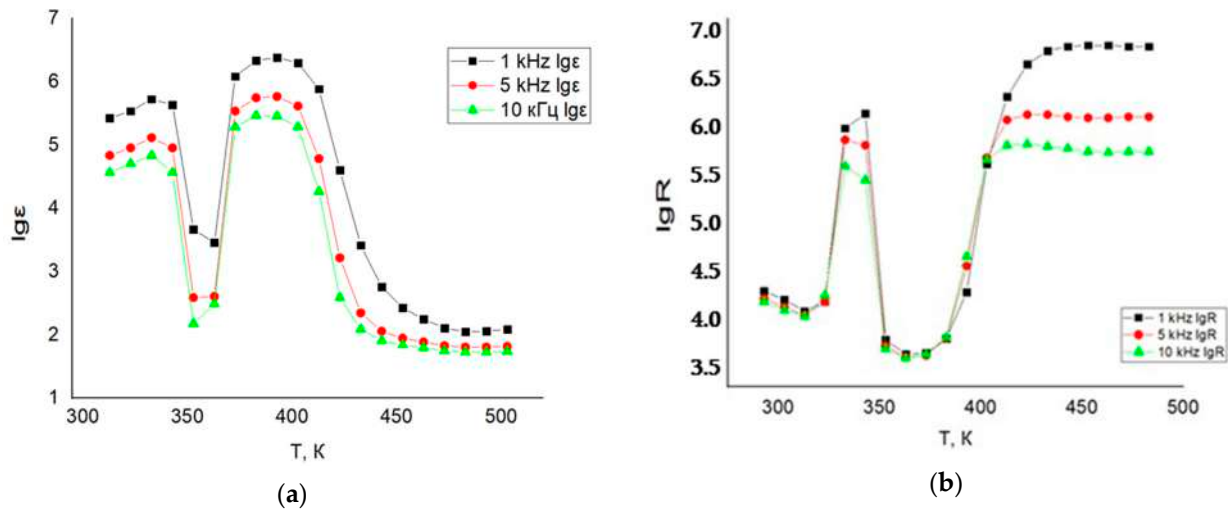


Figure 14. Temperature and frequency (1 kHz, 5 kHz, and 10 kHz) dependence of the dielectric permittivity (a) and electrical resistance (b) of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$.

Table 5. (a). Calculation of the band gap width (ΔE) in the range of 293–313 K. (b). Calculation of the band gap width (ΔE) in the range of 343–363 K.

(a)	
T, K	lg R
293	4.41
313	4.22
(b)	
T, K	lg R
343	6.10
363	3.81

The temperature dependence of electrical resistance reveals complex conductivity behavior: from 293 K to 313 K, the material behaves like a semiconductor; between 313 K and 343 K, it exhibits metallic conduction; from 343 K to 363 K, it returns to semiconductor-like behavior; and from 363 K to 483 K, it again shows metallic conductivity.

$$\Delta E = \frac{2 \times 0.00086173 \times 293 \times 313}{0.43(313 - 293)} \log \frac{4.41}{4.22} = 1.92 \text{ B}$$

$$\Delta E = \frac{2 \times 0.00086173 \times 343 \times 363}{0.43(363 - 343)} \log \frac{6.10}{3.81} = 4.0 \text{ B}$$

3. Discussion

A comprehensive investigation of the structural and physical properties of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ sample confirmed the formation of a well-defined cubic spinel structure. The average crystallite size was determined to be 5.2 nm using the Scherrer equation and 9.12 nm from the Williamson–Hall analysis, while the calculated microstrain ($\epsilon = 1.71 \times 10^{-3}$) indicated lattice distortion associated with the incorporation of Cr^{3+} ions. Raman spectra exhibited characteristic vibrational modes: Li^+ , Cr^{3+} , and Fe^{3+} ions near 200 cm^{-1} , Fe-O stretching vibrations within $500\text{--}600 \text{ cm}^{-1}$, and Cr-O vibrations in the $600\text{--}700 \text{ cm}^{-1}$ range. A sharp band at approximately 720 cm^{-1} further confirmed the high degree of structural ordering in the ferrite phase. SEM micrographs revealed uniformly distributed nanostructured grains, while elemental mapping (Cr, Fe, Li, O) demonstrated a homogeneous distribution of the constituent elements, confirming the effectiveness of the synthesis process. Dielectric measurements showed that the dielectric constant (ϵ') increased with rising temperature, reaching a maximum value of $\sim 2.5 \times 10^6$ in the range of 353–373 K. Conversely, ϵ' decreased with increasing frequency, which can be attributed to Maxwell–Wagner interfacial polarization and hopping conduction of charge carriers. Furthermore, the dielectric loss ($\tan \delta$) exhibited a decreasing trend with frequency, indicating that the material displays stable and typical dielectric behavior.

4. Materials and Methods

The sol–gel method was utilized as an efficient technique for synthesizing a novel complex mixed ferrite with a heterogeneous composition. To identify the composition of the synthesized ferrite, X-ray (Osaka, Japan) phase analysis was performed. Scanning electron microscopy (SEM) (Waltham, MA, USA) was used for both qualitative and quantitative characterization of the material. In addition, the electrophysical properties, such as dielectric conductivity and electrical resistivity, were thoroughly investigated.

The crystal structure of lithium chromium ferrite powder samples was examined using X-ray diffraction (XRD) analysis. Phase identification was carried out with a Rigaku Miniflex/600 (Tokyo, Japan) diffractometer, operating with $\text{CuK}\alpha$ radiation ($U = 30 \text{ kV}$, $I = 10 \text{ mA}$), a scanning speed of 1000 pulses per second, and a time constant of 5 s. Data were collected over a 2θ range of 5° to 90° and analyzed using the PDF-5+ database.

The synthesis process began with high-purity reagent-grade chromium (III) oxide, iron (III) oxide, and lithium carbonate as the primary raw materials, along with distilled water. Glycerol (a trihydric alcohol) and citric acid served as the gel-forming agents. The stoichiometric amounts of the raw materials were precisely weighed to an accuracy of 0.0001 g using an analytical balance, followed by mixing and grinding in an agate mortar. The prepared mixture was then transferred to an alumina crucible and dried at 100°C in an electric furnace to promote gel formation.

After drying, the gel was subjected to calcination at 600°C in a muffle furnace for 6 h, resulting in the formation of a fine black powder. This powder was then sintered in air,

with the temperature gradually increased from 700 °C to 1000 °C over a total duration of 30 h, using 6-h increments at each temperature step [47].

The pycnometric density of the ferrite samples was determined using the liquid displacement method [48], employing toluene and deionized water as inert displacement fluids. Each sample's density was measured five times, and the average values were calculated to ensure accuracy.

5. Conclusions

In conclusion, the novel mixed complex ferrite with the composition $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ was successfully synthesized for the first time using the sol-gel method. To characterize the new complex ferrite, X-ray phase analysis, scanning electron microscopy (for both qualitative and quantitative elemental analysis), and electrophysical property measurements (including dielectric constant and electrical resistance) were performed.

For the first time, X-ray phase analysis was used to determine the symmetry types and unit cell parameters of the synthesized complex ferrite. The compound $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ was identified as cubic ($a = b = c = 8.3001 \text{ \AA}$, $Z = 2$) with calculated and pycnometric densities of 4.745 g/cm^3 and 4.748 g/cm^3 , respectively. The X-ray results confirmed that the synthesized material is polycrystalline, and the crystallographic data accuracy is supported by the close agreement between X-ray and pycnometric densities.

Raman spectroscopy was employed to study the vibrational modes of the molecules. The presence of characteristic Fe–O and Cr–O vibrational peaks confirmed that the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ sample possesses a spinel-type structure. Variations in intensity and peak positions among the spectra suggest sample heterogeneity or differing crystal orientations in various regions. The Raman spectrum indicated a complex phase composition, including spinel structures and ferrite groups.

Scanning electron microscopy allowed the collection of micro-samples from different regions of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ crystals for elemental composition analysis. The overall morphology of the complex ferrite's surface was observed, confirming that the compound is single-phase. The structure was further elucidated by the topography and chemical composition analysis. The elemental analysis results, presented in tabular form, confirmed the formula $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$. The particle sizes ranged from $5 \text{ }\mu\text{m}$ to $200 \text{ }\mu\text{m}$.

Electrophysical studies showed that the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite's capacitance and conductivity increase with temperature. The material exhibits two distinct relaxation mechanisms depending on frequency: long-range hopping at low frequencies and short-range hopping at high frequencies. At low frequencies, the material exhibits low conductivity, making it suitable for resistor applications. Grain boundaries contribute high resistance at elevated temperatures, while dielectric losses decrease with increasing frequency. The real part of the resistance decreases with both temperature and frequency, indicating the material's potential use in electronic devices.

Electrophysical measurements of $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ were conducted over the temperature range of 293–483 K at frequencies of 1, 5, and 10 kHz. At 293 K and 1 kHz, the dielectric constant (ϵ) was 2.69×10^5 , reaching a maximum of 2.47×10^6 at 373 K. Increasing the frequency to 10 kHz resulted in a decrease in ϵ across the entire temperature range studied.

The temperature dependence of electrical resistance revealed complex conductivity behavior: semiconductor-like conductivity from 293 to 313 K, metallic behavior between 313 and 343 K, semiconductor-like again from 343 to 363 K, and metallic conduction from 363 to 483 K. The band gap was measured to be 1.92 eV within the 293–313 K range, typical of narrow-gap semiconductors, while at 343–363 K, it increased to 4.0 eV, characteristic of wide-gap semiconductors.

Overall, the obtained results suggest that $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ ferrite is a promising material for potential applications in electronics and energy storage devices.

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Шолу мақала

Шпинельді феррит нанобөлшектерінің «құрылым–қасиет–қолдану» өзара байланыстары: синтезден функционалдық жүйелерге дейін

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Аңдатпа:

Бұл шолу мақаласында шпинель құрылымды феррит нанобөлшектерінің (MFe_2O_4) синтез әдістері, құрылымдық ерекшеліктері және функционалдық қасиеттері жүйелі түрде талданады. Әртүрлі синтез тәсілдерінің, соның ішінде тұндыру (co-precipitation), жану (combustion), золь-гель, термиялық ыдырау, гидротермиялық, сольвотермиялық, микротолқынды, сонохимиялық, электрохимиялық және қатты фазалық реакция әдістерінің физика-химиялық негіздері, артықшылықтары мен шектеулері салыстырмалы түрде қарастырылады.

Синтез параметрлерінің кристалдық құрылымға, морфологияға, катиондардың тетраэдрлік және октаэдрлік позициялар арасында таралуына, сондай-ақ магниттік, диэлектрлік және оптикалық қасиеттерге әсері жан-жақты талданады. Сонымен қатар, шпинельді ферриттерді зерттеуде қолданылатын рентгендік дифракция (XRD), энергия-дисперсиялық спектроскопиямен біріктірілген сканерлеуші электрондық микроскопия (SEM/EDS), Фурье түрлендірулі инфрақызыл спектроскопия (FTIR), FT-Раман спектроскопиясы, диэлектрлік және магниттік өлшеулер сияқты сипаттау әдістерінің мүмкіндіктері кешенді түрде қорытындыланады.

Соңында, шпинельді феррит нанобөлшектерінің электроникада, микротолқынды құрылғыларда, суды тазарту технологияларында, катализде, сенсорларда және биомедициналық салаларда қолданылу әлеуеті жоғары екені көрсетіледі.

Түйін сөздер: шпинельді ферриттер; феррит нанобөлшектері; синтез әдістері; катиондардың таралуы; магниттік қасиеттер; диэлектрлік қасиеттер; құрылымдық сипаттау; катализ; суды тазарту; биомедициналық қолданулар.

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Review

Structure Property–Application Relationships of Spinel Ferrite Nanoparticles: From Synthesis to Functional Systems

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Abstract

This review article provides a systematic analysis of synthesis methods, structural characteristics, and functional properties of spinel-structured ferrite nanoparticles (MFe_2O_4). The physicochemical principles, advantages, and limitations of various synthesis techniques—including co-precipitation, combustion, sol–gel, thermal decomposition, hydrothermal, solvothermal, microwave-assisted, sonochemical, electrochemical, and solid-state reaction methods—are comparatively discussed. The influence of synthesis parameters on crystal structure, morphology, and cation distribution between tetrahedral and octahedral sites, as well as on magnetic, dielectric, and optical properties, is critically analyzed. Furthermore, the capabilities of characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS), Fourier-transform infrared spectroscopy (FTIR), FT-Raman spectroscopy, dielectric measurements, and magnetic measurements for investigating spinel ferrites are comprehensively summarized. Finally, the high potential of spinel ferrite nanoparticles for applications in electronics, microwave devices, water treatment, catalysis, sensors, and biomedical fields is highlighted.

Keywords: spinel ferrites; ferrite nanoparticles; synthesis methods; cation distribution; magnetic properties; dielectric properties; structural characterization; catalysis; water treatment; biomedical applications



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1. Introduction

Research in nanotechnology has rapidly emerged as a leading field in modern physics and has attracted significant interest from the scientific community [1]. Multifunctional materials, in particular, have been extensively investigated due to the integration of multiple efficient properties within a single intelligent compound [2–4].

Based on their crystalline structures, nanomaterials can be classified into four major groups: spinel ferrites, garnet ferrites, hexaferrites, and orthoferrites [5]. Nanoferrites are magnetic materials composed of metal oxides and are widely employed across various technological fields [6]. Among them, spinel ferrites have gained considerable attention as prominent nanomaterials owing to their unique physicochemical properties and broad range of applications, including biomedical fields [7], water treatment, and industrial electronic devices [8,9].

Ferrites are characterized by high electrical resistivity and magnetic permeability, along with low dielectric losses and eddy current losses; therefore, they are extensively used in numerous electronic devices, computers, and microwave components [10]. All ferrites exhibit ferrimagnetic or ferromagnetic behavior and can be further categorized into four additional classes based on their crystal structures: garnets, orthoferrites, hexagonal ferrites, and spinel ferrites, as illustrated in Figure 1.

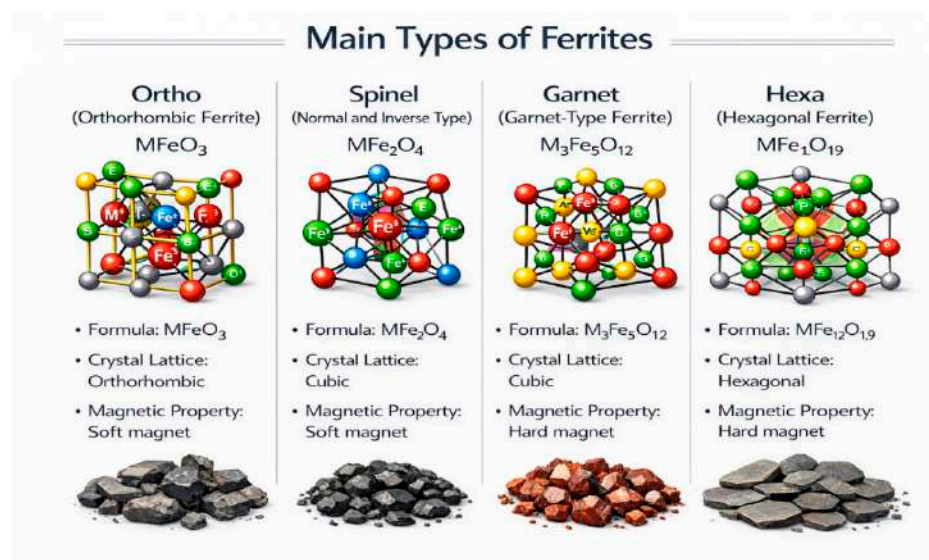


Figure 1. The four classes of ferrites: garnet-type ferrites, orthoferrites, hexagonal ferrites, and spinel ferrites.

Ferrites are ferrimagnetic materials characterized by the spatial arrangement of oxygen anions and metal cations in well-defined geometric configurations within a crystalline lattice. Owing to their excellent magnetic, structural, and catalytic properties, ferrites are widely utilized in various applications. The intrinsic properties of ferrites—such as their structural features, electrical behavior, and magnetic response—are strongly influenced by the synthesis method, chemical composition, and cation distribution. Ferrites can be classified into four main types: spinel ferrites, hexaferrites, garnets, and orthoferrites [11]. Among these, spinel ferrites are considered one of the most promising materials due to their superior structural, magnetic, and catalytic properties.

Spinel ferrites, with the general chemical formula MFe_2O_4 (where M represents divalent ions such as Fe^{2+} , Zn^{2+} , Ni^{2+} , and Co^{2+}), have been recognized as highly efficient metal-based catalysts. Their effectiveness arises from several intrinsic characteristics that make them particularly suitable for catalytic applications [12], including high catalytic activity, facile synthesis, and excellent recyclability. The catalytic performance is primarily governed by the cation distribution, which allows precise tuning of their catalytic properties. The use of spinel ferrites dates back more than three millennia, with magnetite (Fe_3O_4) being one of the earliest known magnetic materials [13].

Spinel ferrite nanoparticles (NPs) are of great scientific interest because they enable direct investigation of the relationship between crystal chemistry and magnetic properties. By varying the composition and concentration of divalent cations, several key properties of ferrite nanoparticles can be tailored, making them suitable for applications in data storage [14], environmental remediation [15], energy conversion and storage [16], biomedicine [17], and medical diagnostics [18]. The wide range of potential applications of spinel ferrite nanoparticles has driven the development of numerous synthetic approaches for their production. The magnetic properties of spinel ferrite nanoparticles are among their most intriguing features, as they often differ significantly from those of their bulk counterparts [19,20]. In

general, the magnetic behavior of nanoparticles is governed by size effects and surface phenomena. Size effects give rise to two important phenomena: superparamagnetism and the formation of single magnetic domains. Single-domain structures occur in nanoparticles with diameters below a critical size, where the formation of domain walls becomes energetically unfavorable, resulting in high magnetic anisotropy and a large magnetic moment per unit volume [21]. Magnetic anisotropy represents the energy barrier that stabilizes magnetic moments along a preferred direction. As particle size decreases further, thermal energy can overcome this barrier, leading to random fluctuations of magnetic moments—a phenomenon known as superparamagnetism [22].

Over the past decade, metal oxides and ferrite-based nanomaterials have been intensively investigated for energy-related applications due to their availability, low cost, stability, environmental friendliness, and ease of synthesis [23–28]. This is particularly true for first-row transition metal spinel ferrites (MFe_2O_4), which exhibit attractive properties such as high specific capacity, narrow energy bandgap, and rich redox chemistry, in addition to intrinsic magnetic characteristics including high saturation magnetization, low coercivity, and high Curie temperature [29,30]. Several recent review articles have examined the use of spinel ferrites, ferrites, and transition metal oxides in photoelectrochemical cells—such as water-splitting systems and dye-sensitized solar cells—highlighting the factors that limit photoelectrode performance and discussing strategies to overcome these challenges in detail [29,31–37].

Figure 2 illustrates the cubic close-packed array of oxygen atoms that constitutes the crystal structure of spinel ferrites. The chemical formula of spinel ferrites is MeFe_2O_4 , where Me^{2+} and Fe^{3+} ions occupy two distinct crystallographic sites: tetrahedral (A-sites) and octahedral (B-sites). The coordination of oxygen atoms differs for each site. Specifically, each formula unit contains eight tetrahedral sites and sixteen octahedral sites. In a normal spinel configuration, Me^{2+} ions occupy the A-sites, while Fe^{3+} ions reside at the B-sites. In contrast, in an inverse spinel configuration, Fe^{3+} ions fully occupy the A-sites, whereas the B-sites are shared between Fe^{3+} and Me^{2+} cations. Many spinel ferrites exhibit partial inversion, in which both Me^{2+} and Fe^{3+} ions are distributed between the A- and B-sites. These materials typically display ferrimagnetic ordering, where cations within each sublattice are aligned parallel to one another, while antiparallel alignment exists between the A- and B-sublattices. Since the number of B-sites is twice that of A-sites, this magnetic arrangement results in a net magnetic moment, giving rise to ferrimagnetism. The magnetic properties of spinel ferrites can be effectively tuned by modifying the type of metal cations and their distribution between the A and B sites [38,39].

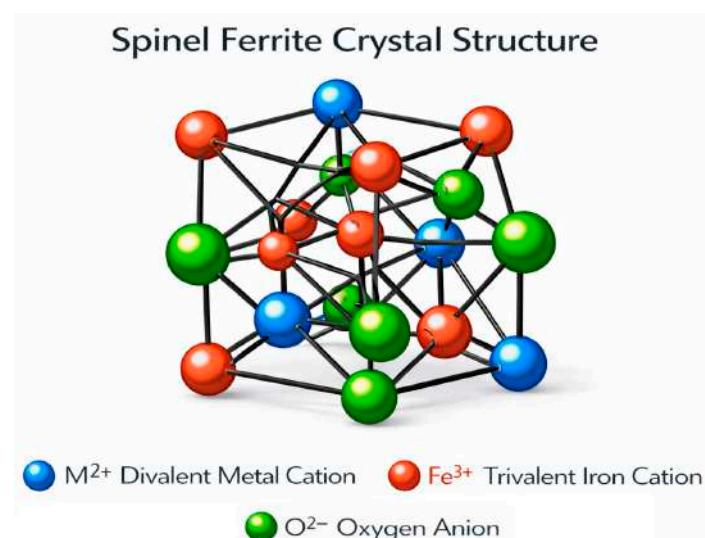


Figure 2. Schematic representation of the crystal structure of spinel ferrites.

The aim of this review article is to provide a systematic analysis of the main synthesis methods for spinel-structured ferrite nanoparticles, their scientific principles, and the influence of synthesis parameters on the morphological, structural, and functional properties of the materials. The scope of the article covers chemical, physicochemical, and high-efficiency synthesis approaches for ferrite nanoparticles, along with their advantages and limitations, as well as the magnetic, electrical, optical, and resistive properties of the resulting nanostructures.

2. Synthesis Methods of Spinel Ferrite Nanoparticles

The synthesis of spinel ferrite nanoparticles involves a variety of techniques, including co-precipitation, sol–gel, hydrothermal methods, ball milling, wet chemical routes, thermal decomposition, ceramic methods, and solid-state synthesis. Accordingly, numerous synthesis strategies have been developed for the preparation of cobalt ferrite nanoparticles; however, no universal method exists for their synthesis. Each synthesis technique possesses its own advantages and limitations.

Two general approaches are employed for nanoparticle synthesis and nanostructure fabrication. The first is the top-down approach, in which bulk materials are broken down into nanoscale structures or particles by removing material from larger objects. This approach typically involves mechanical processes such as milling or attrition. The second is the bottom-up approach, where chemical or physical forces acting at the nanoscale are used to assemble basic building units into larger structures. A wide range of bottom-up techniques—such as hydrothermal and combustion synthesis—have been developed for the preparation of metal oxide nanomaterials.

The main synthesis routes for spinel ferrites (MFe_2O_4) are illustrated (Figure 3), including the sol–gel, solvothermal, hydrothermal, co-precipitation, combustion, microwave-assisted, and biosynthesis methods. These approaches enable the controlled tuning of particle size, phase purity, and magnetic properties to meet specific application requirements.

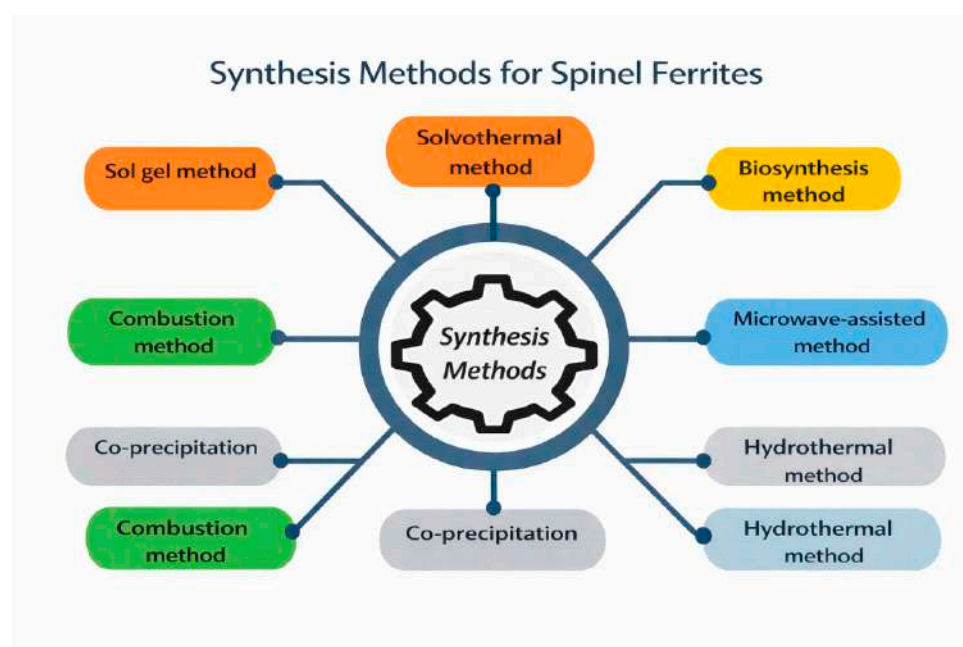


Figure 3. Schematic overview of chemical synthesis methods employed for nanoferrite preparation.

2.1. Co-Precipitation Method

The co-precipitation method is a widely used technique for nanoparticle synthesis, in which precursors are dissolved in an aqueous solution followed by the addition of a base under continuous stirring in a reducing environment at room temperature, leading to

the formation of a precipitate and subsequent nanoparticle formation. Process variables such as the type and ratio of salts used, pH, reaction temperature, and ionic strength allow control over the colloidal properties of the final particles [40]. Co-precipitation is a simple wet-chemical method that is extensively employed for the synthesis of spinel ferrites due to its low cost, simplicity, ability to produce fine particles, and good stoichiometric control. In this approach, metal salts are dissolved in common solvents in stoichiometric proportions, and a precipitating agent is added to achieve the desired pH, resulting in a single-phase homogeneous inorganic solid [41]. Aqueous solutions containing mixtures of divalent and trivalent transition metal ions are typically prepared in a 1:2 molar ratio [42,43]. Subsequently, sodium hydroxide is added under continuous stirring until the pH reaches 12, followed by the addition of oleic acid as a surfactant [44]. The resulting mixture is heated at 80 °C for 1 h and then cooled. The precipitate is washed and centrifuged to remove impurities, dried, and finally calcined at 600 °C for 10 h to obtain the desired nanoparticles [45]. Researchers have emphasized the use of high-purity chemicals and careful purification procedures to ensure particle purity. In one approach, Ristić and co-workers combined co-precipitation with autoclaving to produce nanoparticles, a process referred to as “combined synthesis” [46].

2.2. Combustion Method

The combustion method is one of the most efficient techniques for synthesizing a wide range of inorganic materials on an industrial scale [47]. Due to its high efficiency, straightforward implementation, low cost, and energy-saving nature, the combustion method has been widely adopted and intensively investigated within the scientific community. In this approach, spinel ferrite nanoparticles are produced through a highly exothermic oxidation–reduction (redox) reaction: metal nitrates (acting as oxidizers, including $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) are typically mixed with a fuel in stoichiometric proportions, and after gelation/thickening the mixture undergoes self-ignition, yielding a voluminous and porous (“fluffy”) powder. Although glycine is most commonly used as the fuel, citric acid, urea, and mixed fuel systems such as urea–glucose can also serve as effective alternatives [48]. Salunke et al. observed that this process is extremely rapid, with ignition and foam formation occurring within approximately 5 s [49]. By adjusting the glycine-to-nitrate ratio, parameters such as crystallite size and surface area can be controlled. The resulting product is typically voluminous and porous. In contrast, Prabhakaran and co-workers employed DL-alanine gel as a fuel in the combustion synthesis route [50]. Using this technique, DL-alanine was combined with metal and iron nitrate solutions to start a self-sustaining exothermic reaction. Metal ferrite was created by igniting the gel, and it was then calcined at various temperatures. Because it allows for the adjustment of factors that directly affect the final product, the combustion method provides a flexible approach for the synthesis of nanoparticles.

2.3. Sol–Gel Method

The sol–gel method is a chemical process in which solid particles are synthesized from colloidal suspensions through hydrolysis and polymerization reactions, involving the transformation of a liquid into a gel, followed by drying and heat treatment to obtain a dense material. This method provides high purity and homogeneity, operates at relatively low processing temperatures, and offers multifunctional material design with precise control over nanostructure [51]. In order to create spinel ferrite nanoparticles, metal alkoxide solutions are hydrolyzed and then condensation polymerized to create a gel. Heating is used to eliminate volatile components after synthesis. The creation of nanosized, homogenous, and highly active powders is made possible by the method’s many benefits, which include the use of cheap precursors, low energy consumption, ease of preparation,

and minimal equipment requirements. However, careful control of reaction parameters such as calcination temperature, stirring rate, and solution concentration is required [42]. A major drawback is the potential impurity of the final product, which necessitates post-synthesis thermal treatment to achieve high purity. Nevertheless, the sol–gel method allows effective control over particle composition, size, homogeneity, and distribution [52].

2.4. Thermal Decomposition Method

Thermal decomposition refers to the process of breaking chemical bonds in compounds through the application of heat. This method is particularly useful for synthesizing monodisperse nanoparticles [53]. One of its main advantages is the provision of sufficient thermal energy to heal defects through high-temperature decomposition. Thermal decomposition techniques have been employed to control nanoparticle size, polydispersity, and morphology. However, these reactions are typically conducted under oxygen-free conditions at high temperatures using organic solvents and vapor phases to address safety concerns. Nanoparticles produced by oxygen-free thermal decomposition often exhibit inferior magnetic characteristics due to a broader distribution of magnetic diameters relative to their physical diameters [54].

2.5. Hydrothermal Method

In the hydrothermal synthesis method, soluble salts containing divalent and trivalent transition metals are dissolved separately and then combined in a 1:2 molar ratio [42]. To create a homogenous mixture, organic solvents like ethanol or ethylene glycol are progressively added to the aqueous solutions while being constantly stirred. After that, the mixture is put in an autoclave and cooked under intense pressure. The hydrothermal method seems to be one of the most promising methods for producing ferrite nanoparticles on a large scale when compared to other synthesis processes. By selecting appropriate solvent mixtures and varying temperature, pressure, and reaction time, nanoferrites with well-defined sizes and morphologies can be obtained [55,56].

2.6. Ceramic Method

The ceramic method is a simple and effective approach for synthesizing ferrite nanoparticles at very high temperatures. It involves grinding pre-certified oxides in an agate mortar, followed by pre-calcination at elevated temperatures to obtain the desired samples. Spinel ferrite powders prepared using this method are typically calcined at 1000 °C for up to 72 h [42].

2.7. Solid-State Reaction Method

The solid-state reaction method is an efficient and straightforward technique for nanoparticle synthesis, in which atoms or ions react through diffusion at high temperatures (typically above 800 °C). This method is suitable for large-scale production and can yield high-purity spinel ferrite nanoparticles; however, the reaction time is generally longer compared to other synthesis routes. It has been widely used to produce various types of nanoparticles [57].

2.8. Microwave

Microwave-assisted synthesis is another method used for the preparation of ferrite nanoparticles. In this technique, energy is directly delivered to materials through electromagnetic radiation, which is converted into thermal energy via molecular interactions, resulting in rapid heating. Typically, reaction temperatures range from 100 °C to 200 °C, and reaction times are significantly shorter than those of conventional methods. An exhaust system is employed to remove vapors generated during heating. Although microwave-assisted synthesis enables large-scale preparation of ferrite nanoparticles, its productivity is generally lower than that of thermal or hydrothermal decomposition and co-precipitation

methods. Careful selection of reagents and precursors is essential to meet requirements related to cost-effectiveness and scalability [58].

2.9. Solvothermal Method

The solvothermal process has been widely employed due to its ability to precisely control particle size and morphology. Solvothermal synthesis refers to a chemical process carried out in non-aqueous solvents under elevated temperature and pressure conditions. Accordingly, $X\text{Fe}_2\text{O}_4$ ($X = \text{Mn}, \text{Co}, \text{Ni}$) spinel ferrites were prepared via a solvothermal route in an ethylene glycol medium: stoichiometric amounts of analytical-grade metal chlorides ($X\text{Cl}_2 \cdot n\text{H}_2\text{O}$) and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ were dissolved, and upon the addition of NaOH to induce hydroxide precipitation, the resulting mixture was treated in a Teflon-lined autoclave at 200 °C for 24 h. Finally, the products were purified by centrifugation, washed three times with ethanol and DI water, and dried under vacuum at 60 °C [59]. In this method, precursor solutions containing metal and iron salts are sealed in a high-pressure vessel and heated at high temperatures. The process yields nanoparticles with precise crystalline phases and controlled shape and size distributions. Parameters such as reaction time, temperature, surfactant type, solvent, and precursor composition can be adjusted. The simplicity of this method makes it particularly useful for preparing spinel ferrites with enhanced chemical and physical properties for biomedical and industrial applications [43].

2.10. Sonochemical Method

The sonochemical method is widely applied for the synthesis of spinel ferrite nanoparticles due to its ability to provide uniform mixing and easy control over reaction conditions. Ultrasonic waves, temperature, and intensity directly influence particle size, enabling the formation of crystalline phases at relatively low temperatures [60,61]. Ultrasonic-assisted sonochemical synthesis has emerged as a powerful, environmentally benign, and cost-effective alternative for nanoparticle fabrication. Compared with conventional approaches, this technique offers several key advantages, including accelerated reaction kinetics, enhanced product purity, and a narrower nanoparticle size distribution. The application of ultrasound can markedly reduce the final particle size and suppress agglomeration, thereby yielding more uniform products with improved crystallinity. Moreover, relative to high-temperature solid-state routes, sonochemical processing enables the formation of crystalline phases at substantially lower temperatures [62,63]. The sonochemical approach is of particular importance because it enables the production of high-purity materials with a narrow particle size distribution, offers an environmentally benign route, allows precise control over reaction conditions, and accelerates reaction kinetics, thereby facilitating the formation of nanoparticles with uniform morphologies. In addition, this method can enhance the performance of phase-transfer catalysts and improve their catalytic reactivity [64,65]. Notably, no calcination step is required in this process [66]. As a result, sonochemical synthesis offers advantages such as reduced particle size, improved uniformity, and high stability, although it also has certain drawbacks, including the need for specialized equipment and complex reaction conditions [67].

2.11. Electrochemical Method

The electrochemical synthesis of nanoparticles is similar to the co-precipitation method but utilizes ion sources generated through anodic dissolution of electrodes. This approach offers high-purity products and precise control over particle size. Sample purity and particle dimensions can be optimized by adjusting parameters such as electrode spacing and current density. Factors including pH, current density, electrolyte concentration, and electrode selection play crucial roles in achieving the desired quality of ferrite nanoparticles [68].

2.12. Mechanical Milling Method

In materials science, metallurgy, and nanotechnology, mechanical milling is a common solid-state powder processing method for creating alloyed materials and fine powders. In high-energy ball mills, powder particles are repeatedly fractured and cold-welded. This method is commonly employed to synthesize nanoferrites with a broad size distribution, typically ranging from 35 nm to 85 nm [69–72].

Table 1 summarizes and compares the most widely used synthesis routes for spinel ferrite nanoparticles in terms of processing temperature, typical particle size range, and the key advantages and limitations of each method. Overall, low-temperature wet-chemical approaches (e.g., co-precipitation, sol–gel, hydro/solvothermal, microwave-assisted) generally yield smaller particles with improved control, whereas high-temperature solid-state/ceramic routes tend to produce larger grains with limited tunability despite their scalability.

Table 1. Comparison of synthesis methods, processing temperatures, particle sizes, advantages, and disadvantages for spinel ferrite nanoparticles.

Synthesis Method	Temperature (°C)	Particle Size (nm)	Advantages	Disadvantages
Co-precipitation	Room temp–100	5–50	Low cost, simple process, good stoichiometric control, scalable	Agglomeration, limited size control
Combustion	300–800	10–60	Fast synthesis, energy efficient, low cost	Porous structure, poor morphology control
Sol–gel	200–600	5–40	High purity, homogeneous particles, good size control	Requires calcination, sensitive parameters
Thermal Decomposition	200–350	5–20	Monodisperse particles, excellent size control	High temperature, toxic solvents
Hydrothermal	120–250	10–80	High crystallinity, controlled morphology	Long reaction time, autoclave required
Ceramic Method	900–1200	>100	Simple, suitable for bulk production	Large particle size, poor control
Solid-State Reaction	800–1200	>100	High purity, industrially scalable	Long processing time, coarse particles
Microwave-assisted	100–200	10–50	Short reaction time, energy efficient	Limited scalability, lower yield
Solvothermal	150–300	10–50	Good size and shape control, high crystallinity	Expensive solvents, high pressure
Sonochemical	<100	5–30	Low temperature, uniform particles, eco-friendly	Specialized equipment required
Electrochemical	Room temp–80	10–40	High purity, controlled particle size	Sensitive to pH and current density

3. Characterization Techniques for Spinel Ferrite Nanoparticles

3.1. X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) is one of the most widely used techniques for the analysis of nanoparticles. Important information such as crystal structure, phase composition, lattice parameters, and crystallite size can be obtained from XRD measurements [73]. Using XRD analysis, the synthesized spinel ferrites are comparatively investigated in terms of crystal symmetry, unit cell parameters, pycnometric density, and X-ray density [74].

The intensities of diffraction peaks are calculated on a 100-point scale along the probability line. Data processing and calculations were performed using the PDXL/2

software [4]. For example, high calcination temperatures contribute to the elimination of structural defects or residual impurities, thereby improving crystal quality and promoting the growth of larger crystallites. The average crystallite size was estimated from the broadening of XRD peaks using the Debye–Scherrer equation, which is commonly applied for crystallite size determination [75]:

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

The microstructural characteristics of the synthesized complex ferrites, including crystallite size and microstrain, were further determined using the Williamson–Hall (W–H) method. The Williamson–Hall approach is a widely employed analytical technique for investigating crystalline structures and their mechanical properties. Based on XRD data, this method enables the evaluation of microstructural parameters, particularly the average crystallite size and lattice strain.

The Williamson–Hall equation is expressed as:

$$\beta \cos \theta = \frac{k\lambda}{D} + 4\epsilon \sin \theta \quad (2)$$

where

β —Full width of the diffraction peak (also called “width”) in radians;

θ —Diffraction angle;

k —A system factor required for the calculation (usually taken as 0.9);

λ —Wavelength of the X-ray radiation;

D —Average crystallite size (sometimes referred to as “crystal size”);

ϵ —Effect of internal strain, i.e., the expansion and compression due to stresses within the crystal.

The W–H method generally yielded slightly larger crystallite sizes compared to those obtained using the Scherrer equation. This difference is mainly attributed to lattice strain effects induced by variations in calcination temperature.

Figure 4 Williamson–Hall plots ($\beta \cos \theta$ vs. $4 \sin \theta$) and the corresponding linear fits are presented for different compositions ($x = 0.0$ – 0.3). The slope reflects the microstrain in the crystal lattice, while the intercept is used to estimate the crystallite size.

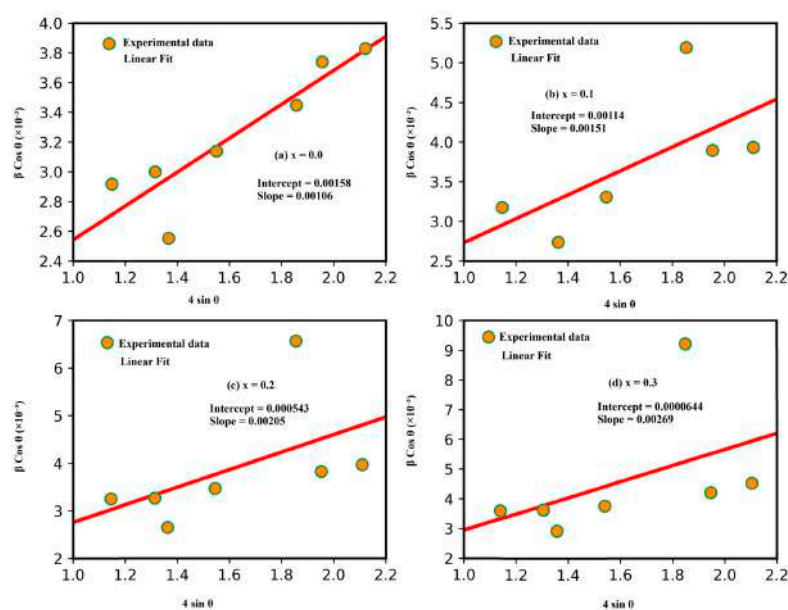


Figure 4. W–H plots of $\text{Cd}_x\text{Li}_{0.4-x}/2\text{Ni}_{0.2}\text{Fe}_{2.4-x}/2\text{O}_4$ samples at different Cd contents [76].

3.2. Density Determination of Spinel Ferrites

The experimental densities of the synthesized spinel ferrites were determined using the pycnometric method. Toluene was selected as the pycnometric liquid because it is chemically inert with respect to the investigated compounds and its density exhibits minimal variation with temperature. A pycnometer with a volume of 1 mL and an analytical balance with a precision of 0.0001 g were used. The experimental procedure was carried out as follows [74]:

- the empty pycnometer was weighed (M_0);
- the pycnometer filled with distilled water was weighed (M_1);
- the pycnometer filled with the pycnometric liquid (toluene) was weighed (M_2);
- the pycnometer containing the sample was weighed (M_3);
- the pycnometer containing the sample and filled with the pycnometric liquid was weighed (M_4).

The density of the sample was then calculated using the following equation:

$$\rho = \frac{M_3 - M_0}{\frac{M_1 - M_0}{\rho_1} - \frac{M_4 - M_3}{\rho_2}} \quad (3)$$

where ρ_1 is the density of water at 20 °C (0.9983 g·cm⁻³) and ρ_2 is the density of the pycnometric liquid.

The density of the pycnometric liquid was calculated using the equation:

$$\rho_2 = \frac{M_2 - M_0}{M_1 - M_0} \cdot \rho_1 \quad (4)$$

The X-ray density was calculated using the standard formula based on the XRD data:

$$\rho_{\text{rent}} = \frac{1.66 \cdot M_c \cdot Z}{V_{\text{tor}}} \quad (5)$$

where Z is the number of formula units per unit cell, $M_{(C)}$ (cell) is the volume of the unit cell, and $M_{(C)}$ is the molecular weight of the compound.

3.3. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is one of the most important techniques used to investigate the morphology and surface structural features of nanosized ferrites. This method provides high-resolution images that allow determination of particle size and shape [77–86].

SEM analysis enables visualization of nanostructural features, particularly agglomerated structures, which is crucial for understanding the synthesis process and its influence on the final material properties [87–89]. In addition, SEM offers valuable information on the porosity of ferrite nanostructures. Porosity plays a significant role in applications such as catalysis and magnetic devices, as it directly affects the functional performance of the material [87,90].

By combining SEM with energy-dispersive X-ray spectroscopy (EDS), the elemental composition of ferrites can be examined, confirming the presence of the desired elements and their distribution within the nanostructure [87,91]. The average particle sizes of the ferrite nanoparticles were estimated, and the obtained results are consistent with those derived from XRD analysis [87]. In Figure 5a, the LiMg_{0.5}Fe₂O₄ spinel ferrite particles appear as irregularly shaped grain agglomerates with heterogeneous grain sizes, indicating a rough and microporous surface morphology. The agglomeration is attributed to the large specific surface area of the particles and their interaction through weak van der

Waals forces. The histogram in Figure 5b shows an average grain size of approximately 2.27 μm , confirming that the solid-state synthesis produces micrometer-sized grains. The energy-dispersive spectrum (Figure 5c) and the quantitative data in Table 2 confirm the presence of the required Mg, Fe, and O elements and demonstrate good agreement between the experimental composition and the theoretical values [92].

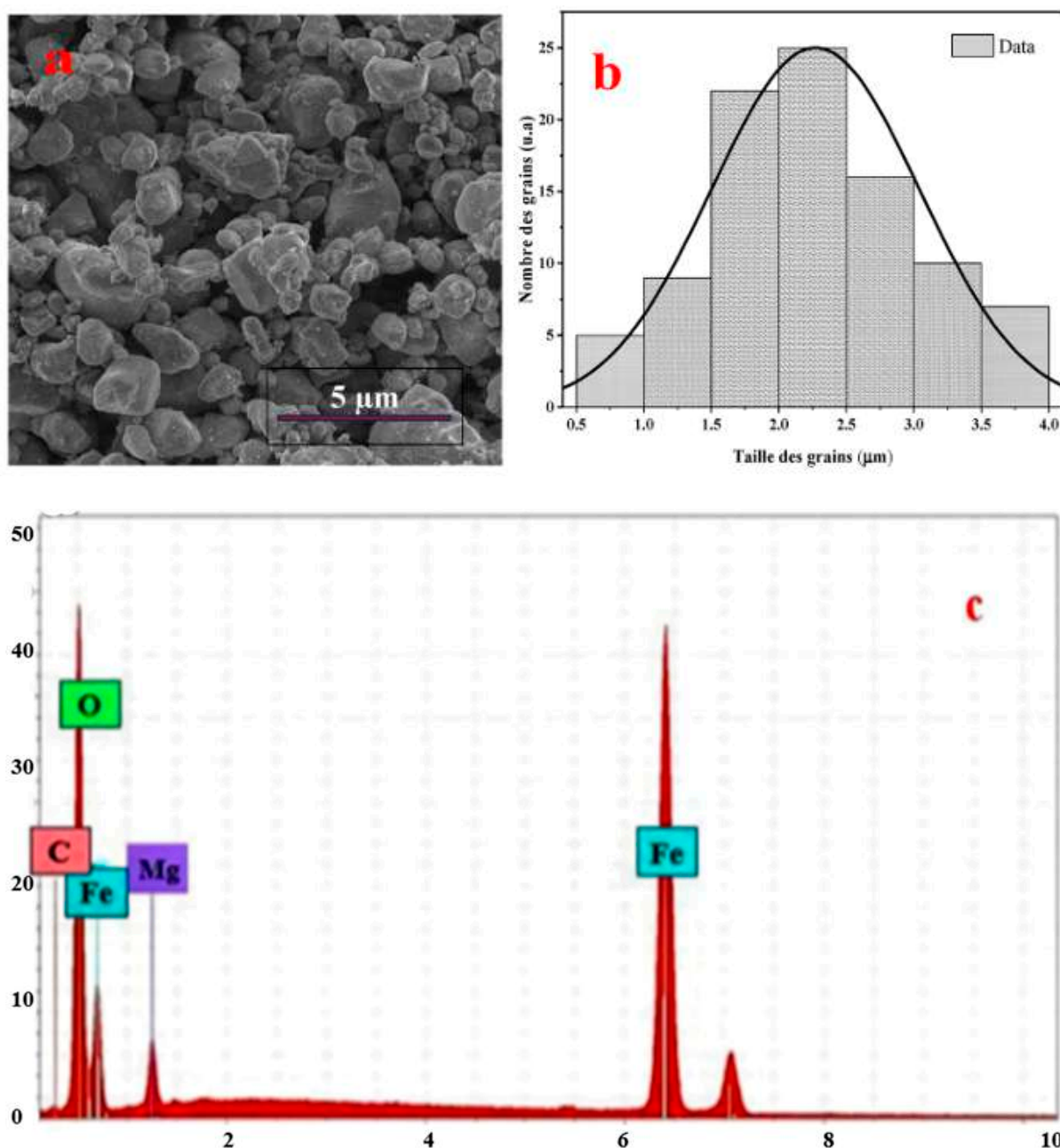


Figure 5. SEM image (a), particle size distribution (b), and EDX spectra (c) of $\text{LiMg}_{0.5}\text{Fe}_2\text{O}_4$.

Table 2. Atomic masses, percentages, and the corresponding theoretical values of $\text{LiMg}_{0.5}\text{Fe}_2\text{O}_4$.

Element	Experimental Mass (%)	Experimental Atomic (%)	Theoretical Mass (%)	Theoretical Atomic (%)
Carbon	1.35	5.26	—	—
Oxygen	33.15	57.22	32.96	53.33
Magnesium	4.59	6.30	6.25	6.66
Iron	57.91	31.22	57.51	26.66

The absence of the Li-related peak is attributed to the detection limit of the instrument. The carbon-related peak originates from the sample substrate. The absence of foreign elements confirms the successful synthesis of the $\text{LiMg}_{0.5}\text{Fe}_2\text{O}_4$ material. Furthermore, Li is isomorphically substituted for Mg; thus, lithium atoms occupy the Mg lattice sites.

3.4. FTIR Analysis

Fourier transform infrared (FTIR) spectroscopy is employed to identify functional groups and metal–oxygen chemical bonds in nanosized ferrites and plays a crucial role in confirming the formation of the spinel structure [93]. Typically, absorption bands observed in the FTIR spectra around $\sim 600\text{ cm}^{-1}$ and $\sim 425\text{ cm}^{-1}$ correspond to the stretching vibrations of metal–oxygen bonds at tetrahedral and octahedral sites, respectively [94]. These characteristic bands indicate the successful formation of the spinel ferrite phase.

In addition, shifts in the absorption bands reflect changes in the local environment of metal ions, which can influence the magnetic and dielectric properties of ferrites [93]. FTIR analysis enables the assessment of the purity of synthesized nanoferrites and facilitates investigation of the effects of dopant elements on structural properties [95,96]. For instance, as shown in Figure 6, the FTIR spectra of $\text{Li}_{1.8}\text{Ni}_{0.1}\text{Dy}_y\text{Fe}_{2-y}\text{O}_4$ ($y = 0.025$ and 0.075) compounds confirm the characteristic features of a spinel ferrite structure. The absorption bands in the $600\text{--}400\text{ cm}^{-1}$ region are attributed to Fe–O bond vibrations, while an increase in Dy content leads to slight band shifts, indicating structural modifications within the crystal lattice [97].

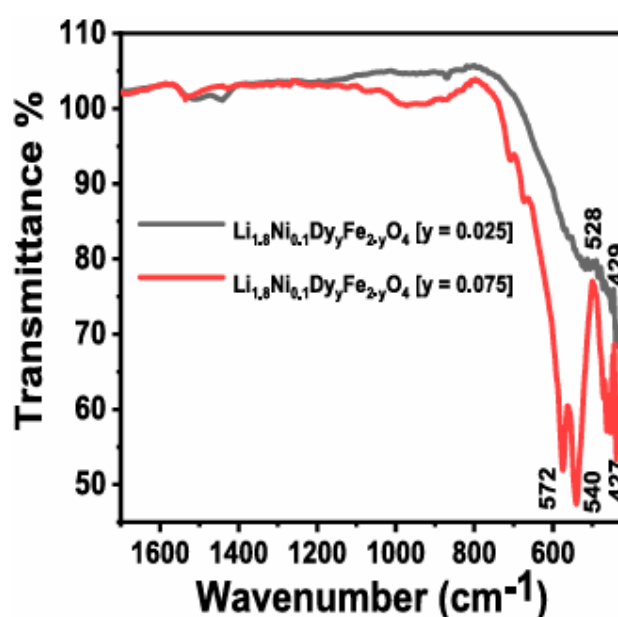


Figure 6. FTIR spectra of $\text{Li}_{1.8}\text{Ni}_{0.1}\text{Dy}_y\text{Fe}_{2-y}\text{O}_4$ ($y = 0.025, 0.075$) compounds.

3.5. FT-Raman

Raman spectroscopy is considered an effective analytical technique for investigating the vibrational modes within the crystal structure of ferrite materials. This method enables confirmation of the formation of the spinel phase during the synthesis process and plays a crucial role in evaluating the structural integrity and functional properties of the material. Since the FT-Raman technique allows the analysis of nanoscale ferrites without altering their physical or chemical state, it is particularly well suited for the characterization of delicate nanostructures [87].

In addition, this technique provides valuable information on internal stresses and structural defects in the synthesized ferrites. As these factors significantly influence the

magnetic and electronic properties of the materials, their identification is essential for optimizing synthesis conditions. FT-Raman spectroscopy also makes it possible to examine the effect of dopant elements on the vibrational characteristics of ferrites, thereby offering insight into how different substituting ions modify the material properties and overall performance.

As an example, Figure 7 presents three Raman spectra. The peaks observed near 200 cm^{-1} (195 , 200 , and 206 cm^{-1}) are attributed to lattice vibrations involving Li^+ , Cr^{3+} , or Fe^{3+} cations. The bands appearing in the $500\text{--}600\text{ cm}^{-1}$ region (509 , 511 , 516 , and 597 cm^{-1}) correspond to Fe–O stretching vibrations in the spinel lattice. The peaks identified in the $600\text{--}700\text{ cm}^{-1}$ range (608 , 618 , 650 , 690 , and 705 cm^{-1}) are associated with Fe–O vibrational modes in ferrite groups, particularly those related to CrO_4^{2-} units. A pronounced band around 720 cm^{-1} indicates a high concentration of ferrite groups [98]. Furthermore, FT-Raman spectroscopy can be applied to investigate the temperature dependence of vibrational modes, allowing the thermal stability and possible phase transitions of nanoscale ferrites to be determined. By analyzing the position and intensity of Raman bands, this technique serves as a reliable tool for quality control of synthesized nanoferrites, ensuring their suitability for various practical applications.

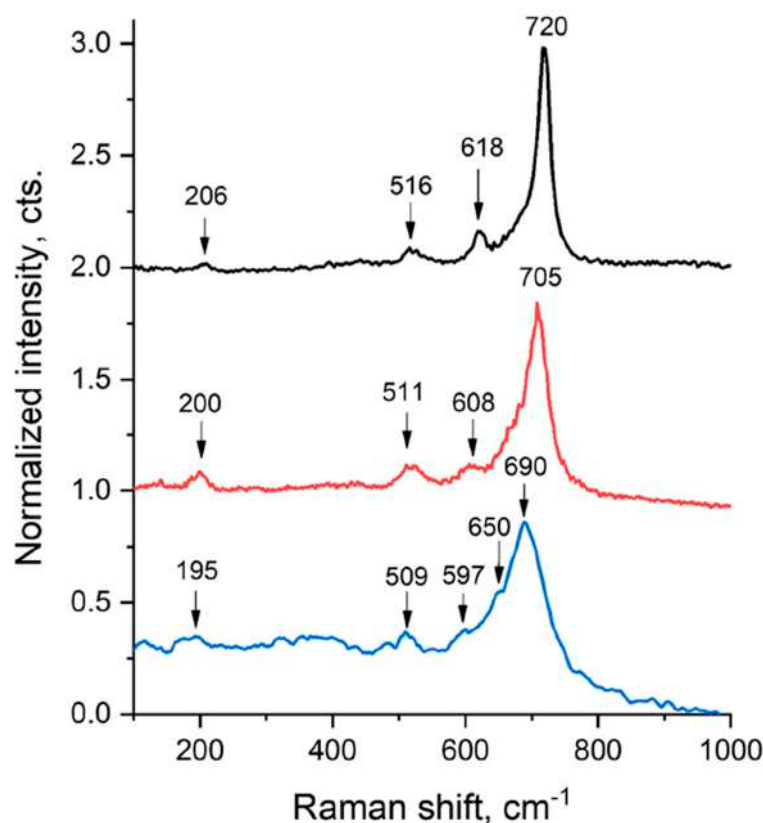


Figure 7. Raman Spectrum of the $\text{LiCr}_{3.4}\text{Fe}_{1.6}\text{O}_8$ Sample.

3.6. Dielectric Properties

The dielectric properties of nanosized ferrites represent one of the key factors determining their effectiveness in electronic device applications, as these parameters directly influence energy storage capability and signal transmission quality [87,99]. The dielectric constant (ϵ) and dielectric loss tangent ($\tan \delta$) are regarded as the principal indicators describing the insulating behavior of ferrite materials, and they are of particular importance for applications in electronic components such as capacitors and inductive elements [99,100].

Investigation of dielectric characteristics makes it possible to evaluate the influence of microstructural features of ferrites, including grain size, porosity level, and overall electrical behavior [100,101]. Moreover, analysis of the variation in dielectric parameters over a wide frequency range provides deeper insight into the electrical response of nanoferrites intended for high-frequency device applications [99]. For this purpose, the frequency- and temperature-dependent behavior of the dielectric constant (ϵ') was examined, and the obtained results are presented in graphical form. For instance, the results derived from Figure 8 indicate that both the dielectric constant (ϵ') and the dielectric loss factor ($\tan \delta$) decrease with increasing frequency, whereas an increase in temperature and Cd-ion concentration enhances charge carrier activity, leading to more pronounced polarization and relaxation mechanisms. These observations confirm that the investigated materials are promising candidates for high-temperature and high-frequency electronic applications [76].

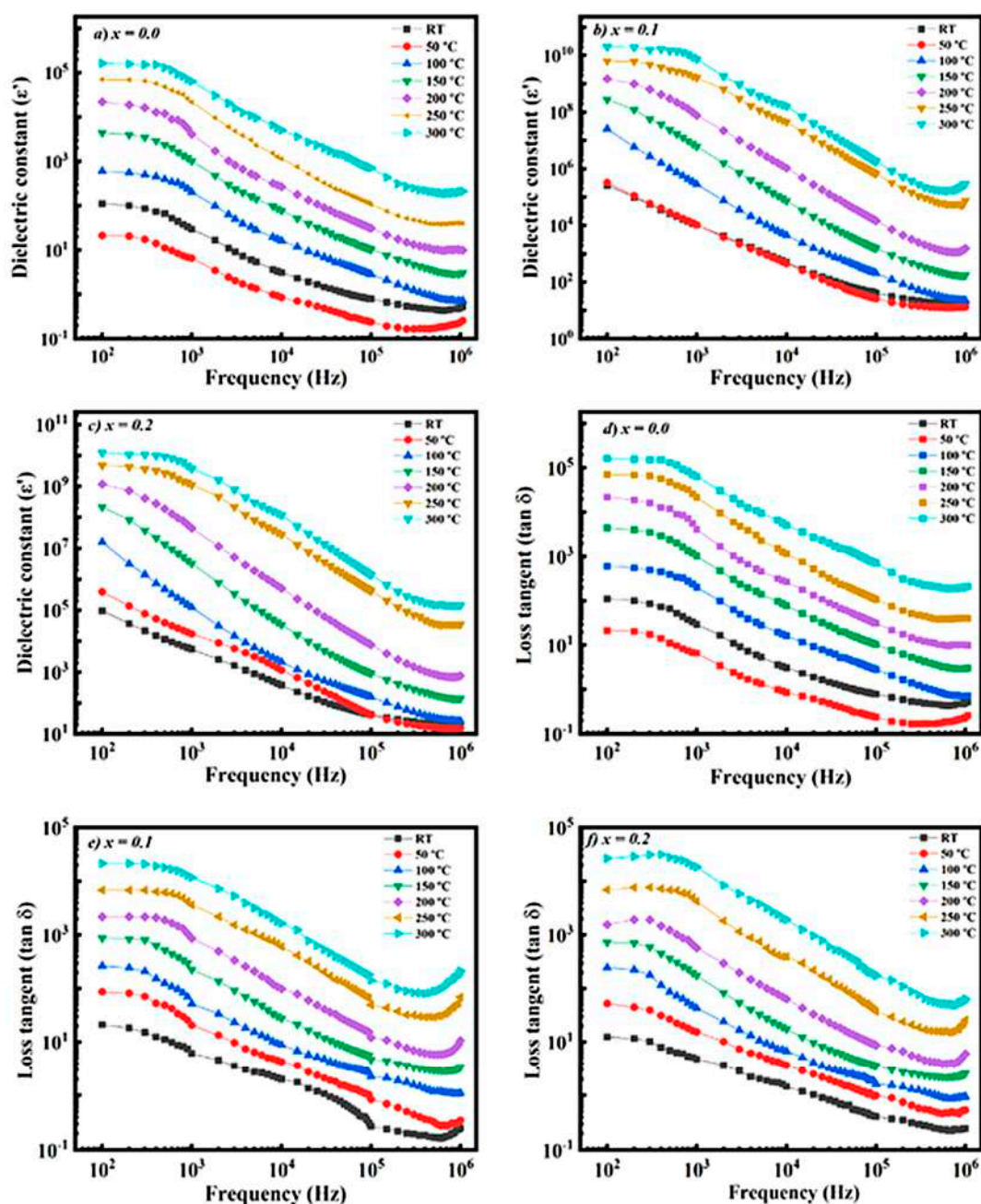


Figure 8. Frequency dependence of the dielectric constant (a–c) and dielectric loss factor ($\tan \delta$) (d–f) at selected temperatures.

As illustrated in Figure 9, the dielectric constant (ϵ') of all samples increases with increasing temperature, while it decreases with increasing frequency. This behavior indicates the dominance of space charge and interfacial polarization mechanisms at low frequencies. The dielectric loss tangent ($\tan \delta$) also exhibits a temperature-dependent increase, whereas it shows significantly lower values at higher frequencies. An increase in Cr concentration leads to a reduction in both ϵ' and $\tan \delta$ values, suggesting an improvement in the electrical stability of the material. These results demonstrate that the investigated materials are suitable candidates for high-frequency and temperature-resistant electronic applications [76].

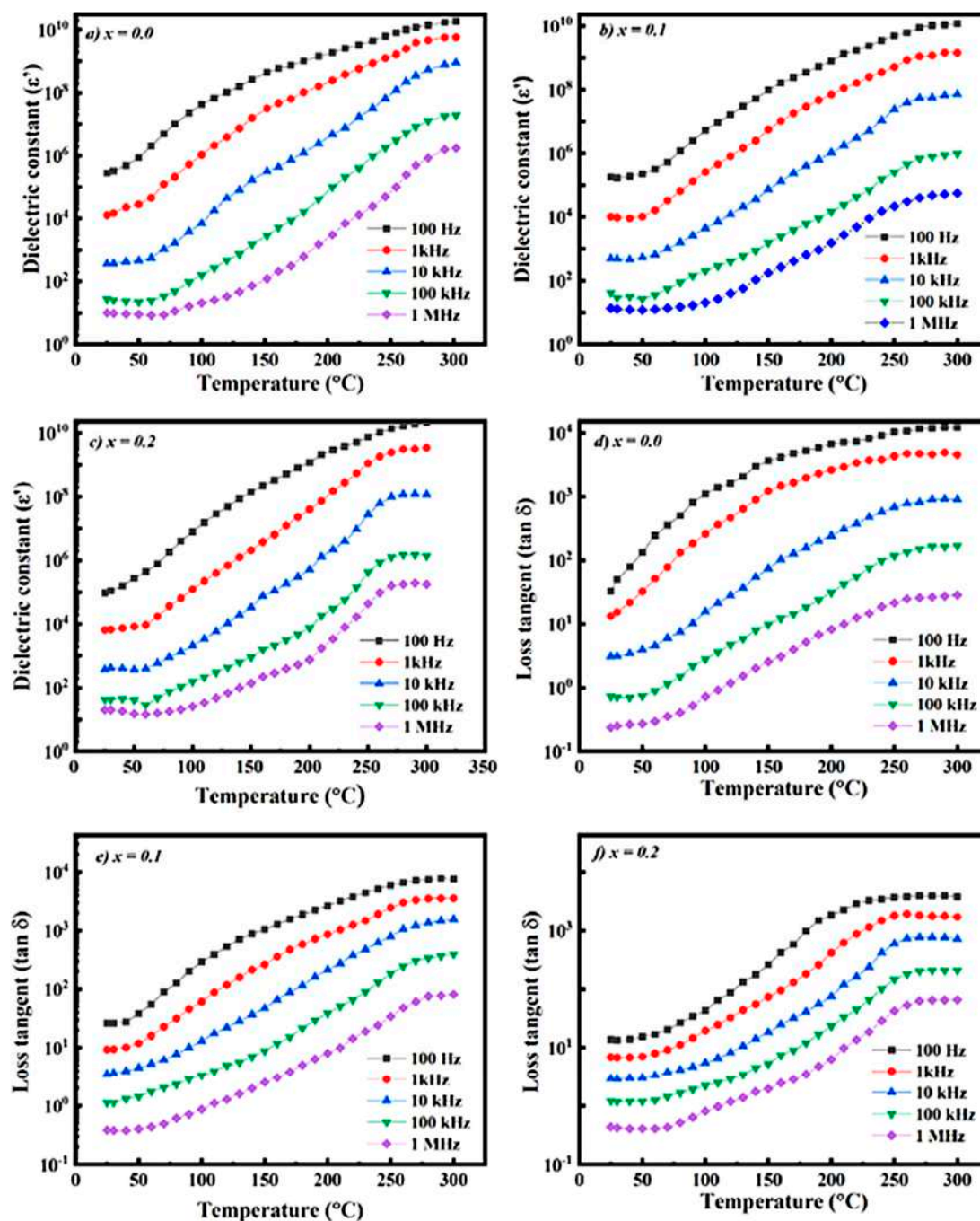


Figure 9. Temperature dependence of the dielectric constant (a–c) and dielectric loss tangent $\tan \delta$ (d–f) at selected frequencies.

3.7. Magnetic Properties

The magnetic properties of nanosized ferrites are commonly investigated using advanced measurement techniques such as vibrating sample magnetometry (VSM) and superconducting quantum interference device (SQUID) magnetometry. These methods enable accurate determination of key magnetic parameters, including saturation magnetization, coercivity, and remanent magnetization, which play a decisive role in describing the magnetic behavior of materials [101].

The hysteresis loops obtained from magnetic measurements provide essential information on the magnetic nature of nanoferrites. The shape and area of the hysteresis loop reflect the coercivity and remanent magnetization levels of the material, parameters that are of particular importance for applications in magnetic memory devices, sensors, and other functional systems [102–104].

The magnetic properties of nanoferrites vary with temperature, including their dependence on the Curie temperature. Therefore, temperature-dependent magnetization is frequently examined to evaluate phase transitions and the thermal stability of the material, which is especially critical for devices operating under high-temperature conditions [89]. In this context, elucidating the correlation between structural parameters—such as crystallite size and morphology—and magnetic properties is of great interest. To this end, X-ray diffraction (XRD) and scanning electron microscopy (SEM) are often employed in conjunction with magnetic measurements to analyze the relationship between structural features and magnetic behavior [101]. Such comprehensive studies enable a deeper understanding of the magnetic properties of nanoferrites and facilitate their efficient utilization in electronics, magnetic recording systems, and biomedical applications [103].

Spinel ferrites (SFs) can be categorized into six main groups—paramagnetic, diamagnetic, ferrimagnetic, ferromagnetic, antiferromagnetic, and superparamagnetic—depending on the type (magnetic or non-magnetic) and distribution of cations at the tetrahedral (A) and octahedral (B) sites. For instance, Figure 10 shows the three main magnetic properties of spinel ferrites: coercive field (H_c), remanent magnetization (M_r), and saturation magnetization (M_s).

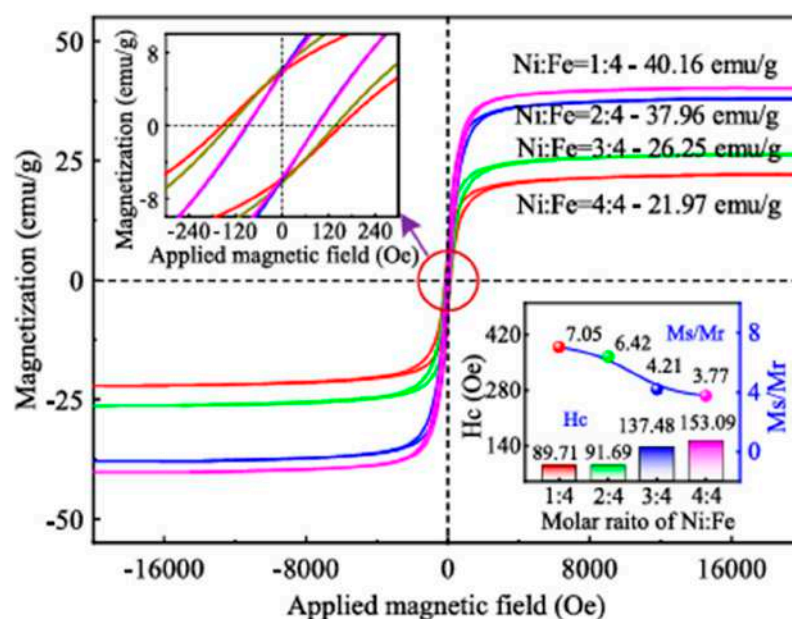


Figure 10. Magnetization hysteresis loops of synthetic $\text{Ni}_x\text{Fe}_{3-x}\text{O}_4$ samples roasted at an oxidation roasting temperature range of 1200 °C for a roasting time of 2.0 h with different molar ratios of Ni/Fe in an air atmosphere.

A high saturation magnetization (M_s) combined with negligible coercive field (H_c) is characteristic of superparamagnetic behavior, a property of particular importance for water treatment applications. Superparamagnetism is typically observed in small ferromagnetic or ferrimagnetic nanoparticles with diameters below approximately 3–50 nm, depending on the material [104]. For example, Figure 10 shows the magnetization hysteresis loops of synthetic $Ni_xFe_{3-x}O_4$ samples with different Ni/Fe molar ratios, roasted in an air atmosphere at 1200 °C for 2.0 h. As the Ni:Fe ratio increases from 1:4 to 2:4, the saturation magnetization (M_s) gradually decreases from 40.16 to 37.96 emu/g, whereas for ratios higher than 2:4, M_s markedly drops to 21.97 emu/g. This behavior is attributed to the increased concentration of Ni^{2+} ions, which inhibits grain growth, leading to a reduction in grain size; additionally, the fraction of non-collinear magnetic structures in the surface layer increases, resulting in a lower saturation magnetization. Moreover, the coercivity (H_c) increases from 89.71 to 153.09 Oe as the Ni:Fe ratio rises from 1:4 to 4:4, while the M_s/M_r ratio decreases from 7.05 to 3.77. The continuous increase in remanent magnetization suggests that the magnetic domain size (approximately equal to the grain size) gradually decreases and approaches the critical dimension for the transition to single-domain behavior [105].

3.8. Optical Characterization

Two main techniques are commonly employed to investigate the optical properties of spinel ferrites: ultraviolet–visible (UV–Vis) spectroscopy and photoluminescence (PL) spectroscopy. UV–Vis spectroscopy enables the determination of the light absorption characteristics of spinel ferrites in the wavelength range of 200–800 nm. Based on the obtained absorption data, the Tauc relation is applied to estimate the direct optical band gap energy of the material. According to the literature, the direct band gap energies of spinel ferrites determined using the Tauc method typically lie in the range of approximately 1.7–4.0 eV [39,106,107].

Photoluminescence spectroscopy is widely used to investigate structural defects in spinel ferrites, including lattice imperfections and oxygen vacancies. PL emission spectra recorded at a specific excitation wavelength are closely related to the defect states of the material. Several studies have reported characteristic PL emission bands of spinel ferrites in the range of about 450–530 nm, which are attributed to crystallographic defects and oxygen deficiency in the lattice [39,106,107].

4. Applications of Spinel Ferrites

Spinel ferrites (SFs) are among the materials that exhibit broad applicability across various scientific and technological fields due to the synergistic combination of their structural, electrical, and magnetic properties. The diverse application areas of these materials are summarized schematically in Figure 11. In particular, spinel ferrites have been traditionally used as magnetic materials. In addition, they have been extensively investigated as efficient adsorbents for the removal of heavy metal ions and organic dyes in water and wastewater treatment, as well as catalysts and photocatalysts for the degradation of various pollutants.

Furthermore, spinel ferrites show high potential in biomedical applications, including their use as contrast agents, magnetic hyperthermia materials, and systems for drug delivery and controlled release. They are also suitable for use as sensors and biosensors in biotechnology and medical fields, as functional materials in electronics and microwave devices, in electromagnetic interference (EMI) shielding systems, and as magnetic recording media.

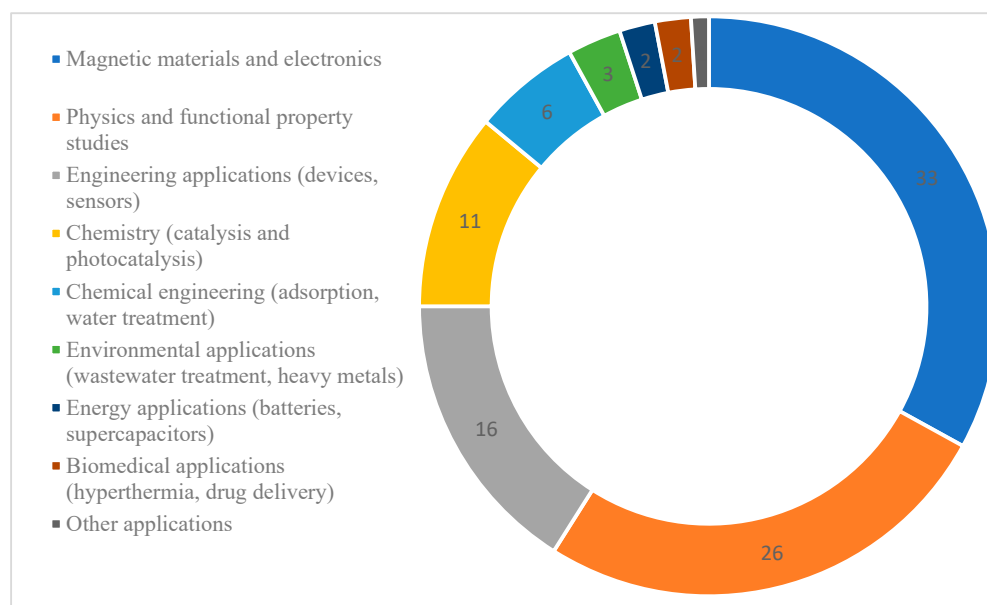


Figure 11. Quantitative comparison of application areas of spinel ferrites.

4.1. Microwave Devices

MnZn ferrites and zinc-substituted cobalt spinel ferrites are characterized by low dielectric losses, which makes them highly suitable for microwave device applications. Low losses reduce energy dissipation and enhance device efficiency. In addition, these materials exhibit stable performance over a wide frequency range and possess high mechanical strength and chemical stability, thereby extending the service lifetime of microwave devices [108,109].

4.2. Telecommunication Applications

Spinel ferrite nanoparticles, particularly Zn-doped CoFe_2O_4 , are widely used in telecommunication systems due to their low power losses. They are effective in the fabrication of inductive components and transformers, improving signal filtering and amplification processes. Furthermore, the integration of ferrites into antenna structures broadens the operating bandwidth and enhances the overall performance of telecommunication systems [108,109].

4.3. Dielectric Applications

Spinel ferrites with high dielectric constants and low dielectric losses are effectively employed in capacitors and high-frequency electronic components. The combination of magnetic and dielectric properties makes them promising magnetodielectric materials for sensors and actuators. Moreover, these materials retain their dielectric characteristics over a wide temperature range, ensuring reliable performance under various operating conditions [109,110].

4.4. Water Treatment

ZnFe_2O_4 and CoFe_2O_4 spinel ferrites exhibit high efficiency in the photocatalytic degradation of organic dyes. Owing to their magnetic properties, these nanoparticles can be easily separated from water and reused. In addition, composites combined with silver nanoparticles or other functional materials enhance antibacterial and catalytic activity, enabling the development of environmentally friendly water treatment technologies [111–113].

4.5. Biomedical Applications

Spinel ferrites such as MnFe_2O_4 are effectively utilized in targeted drug delivery systems due to their superparamagnetic properties, allowing precise control of drug release under an external magnetic field. Furthermore, ferrite nanoparticles serve as efficient contrast agents for magnetic resonance imaging (MRI) and are considered promising materials for biosensors and antibacterial medical coatings [93,95,107,114].

Spinel-structured magnetic ferrite nanoparticles (CuFe_2O_4 , MnFe_2O_4 , CoFe_2O_4 , MgFe_2O_4 , ZnFe_2O_4 , Fe_3O_4 , NiFe_2O_4 , and mixed ferrites) are extensively investigated as targeted drug-delivery platforms. Their tunable surface chemistry, (super)paramagnetic behavior, and the possibility of externally field-guided/targeted release broaden their biomedical applicability. Key functionalization approaches include polymeric coatings (e.g., PLA, PEG), bioconjugation with biomolecules (e.g., folate, doxorubicin), and hybridization with carbon nanomaterials; collectively, these strategies enhance colloidal stability, targeting capability, and multifunctionality of the system [115,116].

Specific examples include CuFe_2O_4 coated with poly(methacrylic acid) and lignin derivatives, which enables pH-dependent release of curcumin and exhibits pronounced cytotoxicity against breast cancer cells [117]. Folic-acid-modified MnFe_2O_4 systems provide sustained doxorubicin release and improve MRI contrast performance [118]. L-cysteine-functionalized CoFe_2O_4 demonstrates high drug-loading capacity and pH-responsive release, making it promising for theranostic applications; however, its relatively higher cytotoxicity requires further optimization [119]. MgFe_2O_4 -based composites can effectively load antibiotics and show strong antibacterial activity via pH-triggered release [120,121]. ZnFe_2O_4 nanoparticles deliver curcumin efficiently and induce anticancer effects while maintaining low toxicity and high biocompatibility [122,123]. Fe_3O_4 coated with chitosan and gallic acid achieves pH-sensitive therapeutic release without compromising biocompatibility [124]. Hollow NiFe_2O_4 nanospheres and coated NiFe_2O_4 carriers exhibit high loading capacity and a combined therapy–imaging (therapeutic–diagnostic imaging) function, although surface modification is needed to mitigate potential nickel-associated toxicity [125]. In addition, systems such as CaFe_2O_4 and Zn-doped MgFe_2O_4 show controllable release profiles and antibacterial efficacy, supporting the potential of mixed ferrites as “tailor-made” nanocarriers [123,126]. Overall, ferrite nanomaterials optimized via green synthesis and surface engineering are regarded as safe and effective multifunctional candidates for drug delivery [115–126].

4.6. Gas Sensors

Spinel ferrite nanomaterials are widely applied in sensors for detecting gases such as H_2S , NH_3 , and $\text{C}_2\text{H}_5\text{OH}$. Doping with metal ions significantly enhances their sensitivity and selectivity. An increase in operating temperature accelerates reaction kinetics and improves sensor response, while the magnetic properties enable easy recovery and reuse of the sensors [127].

4.7. Catalytic Applications

Spinel ferrite nanoparticles are extensively used in environmental remediation and organic synthesis processes due to their high catalytic activity and magnetic recoverability. Surface modification and composite structures can further enhance their adsorption and photocatalytic properties. Moreover, they are promising catalysts in photoelectrochemical systems such as water photolysis and hydrogen production [31,111,128].

4.8. Photoluminescent Applications

The photoluminescent properties of spinel ferrites can be tuned by varying their composition and particle size. Doping with rare-earth elements or transition metals enhances emission efficiency, enabling their use in display technologies, lighting devices, and bioimaging applications [32,93,108].

4.9. Data Storage Technologies

Core-shell structured spinel ferrite nanoparticles exhibit controllable coercivity, making them suitable for high-density magnetic recording systems. These characteristics render them promising materials for next-generation data storage devices [129].

4.10. Acid Mine Drainage (AMD) Treatment

Spinel ferrites are employed in the treatment of acid mine drainage through the efficient removal of heavy metals such as Zn, Cu, and As. This process results in the formation of chemically stable ferrite sludges, preventing the secondary release of toxic elements and ensuring environmental safety [130].

4.11. Voltage-Tunable Inductors

Ni-Cu-Zn spinel ferrites, when incorporated into magnetoelectric composites, enable voltage-controlled tuning of magnetic permeability. This significantly improves the tuning efficiency of inductors and offers high-performance solutions for modern microelectronic devices [131].

4.12. Magnetic Hyperthermia Using Spinel Ferrites

Magnetic hyperthermia aims to selectively damage tumor tissue by raising the local temperature to 40–45 °C using magnetic nanoparticles exposed to an alternating magnetic field (AMF). Heat is generated via Néel/Brownian relaxation and/or hysteresis losses. Surface functionalization improves tumor targeting, enhances treatment precision, and can reduce systemic side effects.

Among spinel ferrites, CuFe_2O_4 exhibits high saturation magnetization and may provide synergistic outcomes when combined with drug delivery or photothermal therapy. MnFe_2O_4 modified with PEG, folate, or thermoresponsive polymers enables controlled heating, temperature-triggered drug release, and enhanced MRI contrast, supporting multifunctional theranostic platforms. Owing to high magnetic anisotropy, CoFe_2O_4 can facilitate hyperthermia-triggered doxorubicin release and combined treatment strategies. SPIONs remain among the most clinically advanced candidates, offering efficient heating and chemo-hyperthermia synergy through tunable coatings. MgFe_2O_4 and ZnFe_2O_4 show favorable biocompatibility with effective heating, and their performance can be further improved by doping and surface engineering. NiFe_2O_4 can generate strong heating due to anisotropy, but requires biocompatible coatings to mitigate potential cytotoxicity. In addition, polymer- or silica-coated mixed ferrites can provide pH- and temperature-responsive drug release under AMF, enabling multimodal therapy with synergistic cytotoxicity in tumor cells. Overall, controlled synthesis and surface engineering are essential to design safe and efficient ferrite-based hyperthermia systems [132].

5. Advantages and Limitations

Spinel ferrites (SFs) have attracted considerable attention from the scientific community in recent years due to their unique combination of electrical, dielectric, electronic, mechanical, magnetic, optical, and catalytic properties [133]. As these materials simultaneously exhibit ferrimagnetic and semiconducting behavior, they can be classified as

magnetic semiconductors [134]. Spinel ferrites are distinguished by exceptional physico-chemical characteristics, including high Curie temperature, pronounced magnetocrystalline anisotropy, good thermal stability, and favorable chemical composition.

In addition, spinel ferrites possess excellent magnetic properties, controllable particle size and shape, high specific surface area, abundant active surface sites, high chemical stability, and ease of modification or functionalization [134]. These properties largely depend on the nature of the divalent cations (other than Fe^{3+}) in the spinel structure, their distribution between tetrahedral and octahedral sites, as well as the synthesis method, grain size, morphology, and calcination temperature employed [135–137].

In recent years, spinel ferrites have been extensively investigated for water treatment and environmental protection applications owing to their low production cost, high catalytic activity, attractive band gap energies, good selectivity, recyclability and reusability without significant loss of activity, and relatively low toxicity [138]. Figure 12 schematically summarizes the main advantages of spinel ferrite nanoparticles.



Figure 12. Various properties of spinel ferrite nanoparticles.

6. Future Directions and Challenges

Future research on spinel-structured ferrite nanoparticles should focus on achieving a deeper understanding of the structure–property–application relationships and on the deliberate control of these correlations. In particular, precise regulation of cation distribution between the tetrahedral (A) and octahedral (B) sites represents a key strategy for tuning magnetic anisotropy, coercivity, dielectric stability, and optical response, thereby enabling the development of highly functional materials. Moreover, nanoscale control over particle size and morphology can optimize superparamagnetic behavior and open new opportunities for applications in high-frequency electronics, gas sensors, and biomedical systems.

In forthcoming studies, the development of environmentally friendly, energy-efficient, and industrially scalable synthesis routes will play a crucial role. The design of hybrid and composite systems—including core–shell architectures, surface-functionalized structures, and multicomponent ferrites—can significantly enhance catalytic, photocatalytic, and sensing performance. In addition, data-driven modeling and machine learning approaches

are emerging as promising tools for predicting complex relationships between synthesis parameters and material properties.

Despite these prospects, several critical challenges remain for the practical implementation of spinel ferrite nanoparticles. These include their tendency toward agglomeration, broad particle size distributions, difficulties in ensuring synthesis reproducibility, and issues related to long-term chemical and thermal stability. For biomedical applications, comprehensive evaluation of toxicity, biocompatibility, and in vivo stability remains one of the major limiting factors.

7. Future Outlook/Challenges

Future direction: Establish robust structure–property–application relationships and precisely control cation distribution (A/B sites) as well as particle size and morphology to tailor ferrite performance.

Key challenges: Persistent agglomeration, broad size distribution, limited synthetic reproducibility, and insufficient long-term chemical/thermal stability.

Biomedical bottlenecks: Comprehensive evaluation of toxicity, biocompatibility, and in vivo stability, alongside optimization of safe surface coatings/functionalization, remains essential for translation.

8. Conclusions

This review systematically examined the synthesis routes, crystalline-structure characteristics, and structure–property–application relationships of spinel-structured ferrite nanoparticles. Various chemical and physicochemical approaches were compared in terms of their impact on particle size, morphology, cation distribution over A/B sites, and the degree of inversion, highlighting that these factors play a decisive role in determining magnetic, dielectric, optical, and catalytic properties.

The analysis indicates that the functional performance of spinel ferrites is governed not only by chemical composition but also by synthesis-dependent parameters such as crystal defects, oxygen vacancies, and surface state. Therefore, achieving application-specific properties requires precise control of cation distribution and nanoparticle size, mitigation of agglomeration, and improved structural homogeneity.

Despite the strong potential of spinel ferrite nanoparticles, several practical limitations remain. These include a tendency toward agglomeration, broad particle-size distributions, insufficient synthetic reproducibility, and challenges related to long-term chemical/thermal stability. For biomedical applications in particular, comprehensive assessment of toxicity, biocompatibility, and in vivo stability is essential and remains a key requirement for translation.

Overall, spinel ferrites can evolve into high-performance functional materials across diverse fields when synthesis is tightly controlled and surface engineering is effectively implemented. Future studies should strengthen property-by-design strategies by clarifying the links between cation distribution–defects–surface state–properties, adopting standardized characterization and measurement protocols, and addressing safety considerations more rigorously to accelerate reliable deployment of spinel ferrites in real-world applications.

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