Physical, Structural and Dielectric Parameters Evaluation of New Mg_{1-x}Co_{x}Ni_{y}Fe_{2-y}O_{4} nano-ferrites Synthesized via Wet Chemical Approach

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ABSTRACT: The surfactant assisted co-precipitation method has been used to synthesize the Co^{2+} and Ni^{2+} substituted magnesium nano-ferrites. The Co^{2+} and Ni^{2+} ions substitute the Mg^{2+} and Fe^{3+} ions from the magnesium ferrites, respectively. To study the effect of substituted ion concentrations on the dielectric performance of the MgFe_{2}O_{4} spinel ferrites. The substituted magnesium nano-ferrites with the compositions Mg_{1-x}Co_{x}Ni_{y}Fe_{2-y}O_{4} (x, y = 0.0, 0.1, 0.2, 0.3, 0.4, and 0.5) were synthesized to notice the effect of substituted ions concentration on the dielectric properties. Thermogravimetric analysis of the precursor precipitates was taken to investigate the stable spinel phase formation temperature. The X-ray diffraction analysis of the all substituted samples confirms the successful formation of the stable spinel phase and also exposes the effects of substituted ions on the crystal structure and unit cell constants. The presence of a characteristic absorption band (400 cm\(^{-1}\), 550 cm\(^{-1}\)) of spinel ferrites in the Fourier Transform Infra Red (FT-IR) spectrum of substituted magnesium nano-ferrites also confirms the formation of desire phase. The effect of temperature on the conductivity of the all substituted samples was observed within the temperature range of 25°C–300°C by two-probe direct current electrical resistivity measurements. Dielectric measurements of the substituted magnesium nano-ferrites were carried out to study their magnetic behavior. The inverse relation of the dielectric parameters of the all substituted magnesium nano-ferrites with frequency shows their excellent potential for devices that operate at higher frequencies.

KEYWORDS: Magnesium nano-ferrites, Cobalt-nickel substitution, X-ray diffraction;

INTRODUCTION

Nano-science is the understanding and explaining the behavior and properties of the materials at the nano-scale (10\(^{-9}\)-th of meter). While the nanotechnology is concerned with manufacturing of devices and structures at nano-scale by applying the understanding of nano-science. A nanotechnologist has to be restricted in the nano-range (1–100 nm), least in one dimension, during the synthesis of materials. At nano-scale, the properties of the materials are addressed by quantum law instead of classical laws. Therefore, properties of the nanomaterials are significantly different from the same materials but out of the nano-scale. Moreover, nanomaterials also possess larger surface area to volume ratio due to which unique properties induces in these materials. It is observed that both these effects are prominent below the 100 nm. On the other hand, classical laws are used to addresses the properties of the bulky materials. That is way nanomaterials exhibited properties that are not exhibited by their bulky homolog [1]. Like the property of surface plasma resonance that is exhibited by nanomaterials, but not shown by bulk material [2]. Nanomaterials because of their unique but unpredictable properties (electrical, optical, electronic, etc.) have caught the attention of the researchers of the all fields [3]. By controlling the particle size morphology and interaction between the particles, one can synthesize nanomaterial...
with desired properties. Nanomaterials are different from their bulk homologue due to their higher active surface area and retention of at least any one dimension in the nano-range [4].

The magnetite (Fe$_3$O$_4$), hematite (Fe$_2$O$_3$), and spinel ferrites (MFe$_2$O$_4$) are common ferromagnetic forms of the iron oxides. The extensive use of these ferrites is due to their high electrical resistivity, high corrosion resistance, unique magnetic, and optical properties [5,6]. Among magnetic ferrites the spinel ferrites, with general formula MFe$_2$O$_4$, are n-type semiconductor has numerous applications. These applications are due to their unique dielectric, electromagnetic and optical properties [7–9]. These properties merely depend on the arrangement of metal cations in tetrahedral (A) and octahedral (B) sites in the spinel ferrite structure [10]. The magnetic nanomaterials because of their tunable magnetic properties are widely being in various fields as in magnetic resonance imaging, in contrast agents [11], in storage devices, etc.[12]. Ferrites because of their high electrical resistivity acts as a magnetic semi-conductors and electric insulators [13]. The spinel nanoferrites also have their applications in many other fields, such as in sensory devices, data storage devices, magnetically warming instruments, and in Ferro-fluid’s technology [14–16]. The properties of nanoferrites materials can be changed by substitution of different transition metal cations in the crystal lattice [17]. The magnetic properties of a ferrite material mainly affected by the size of the crystal’s unit cell, the size of the cation being substituted, and the distribution ratio of the cations. The magnetic properties of the substituted nanoferrites further can be changed by changing the size of the substituted metal ions [18].

Additionally, the preparation methods, substituent nature and its concentrations are very key parameters and they strongly affect the electro-magnetic properties of the ferrites. Similarly, other factors such as structure, size, impurities, and temperature, and pH conditions also affect the magnetic properties of the ferrites. For the synthesis of spinel iron oxides, various methods are used such as co-precipitation [19], micro-emulsion method [20], sol-gel method [21], and microwave assisted hydrothermal method [22]. Specific reaction conditions tailored the specific structure, control the particle size and distribution of ions within the unit cell. It is necessary to control the oxidation and stop agglomeration of nanoparticles hence the particles are coated with a surfactant. The main purpose of using a surfactant is to control the size of nanoparticles [20,23].

Previously, several researchers investigated the effects of substitution of various metals on the electromagnetic and dielectric properties of magnesium nanoferrites. The magnesium ferrites are soft magnetic ferrite that have a partial inverse spinel structure as its Mg$^{2+}$ and Fe$^{3+}$ cations are distributed at both tetrahedral and octahedral sites [24]. These ferrites also show low saturation magnetization, high resistivity, and have reproducible and homogeneous properties. Magnesium nanoferrites can be used in magnetic sensors, heads, transformers, and AC power distributors [25].

In this work, we prepared Ni$^{2+}$ and Co$^{2+}$ substituted magnesium nanoferrites with different substituent’s concentration by simple co-precipitation method. The effect of nature and concentration of the substituent on the lattice parameters of the magnesium nanoferrites is observed. Moreover, we also observe the effect of the substituted ions on the magnetic properties of the nanoferrites. The substitution of transition metal cations in the magnesium ferrite shows positive impact on the electromagnetic and dielectric properties of the substituted magnesium nanoferrites. The improved electro-magnetic performance of the substituted nanoferrites enables them to be used in high frequency and switching devices.

**EXPERIMENTAL SECTION**

**Chemicals and materials**

The following chemicals with analytical grade purity chemicals were purchased from the different companies and used without any further purification. Ferric nitrate nonahydrate [Fe (NO$_3$)$_3$]$\cdot$9H$_2$O, 96%] magnesium nitrate hexahydrate [Mg (NO$_3$)$_2$$\cdot$6H$_2$O, 99%], nickel nitrate hexahydrate [Ni (NO$_3$)$_2$$\cdot$6H$_2$O, 97%, Merck], and cobalt nitrate hexahydrate [Co(NO$_3$)$_2$$\cdot$6H$_2$O, 97%], were purchased from the Merck. The Cetyl trimethylammonium bromide (CTAB) (>99%) was purchased from the Dae-Jung. The aqueous ammonia (NH$_3$$\cdot$H$_2$O, 33%) and methanol (CH$_3$OH, 99%) were purchased from BDH.

**Synthesis procedure**

Cobalt-nickel substituted magnesium nano ferrites (Mg$_{1-x-y}$Co$_x$Ni$_y$Fe$_2$O$_4$, $x,y = 0.0, 0.1, 0.2, 0.3, 0.4, and 0.5$) were synthesized by using a surfactant-assisted coprecipitation method. The required amounts of metal salt’s solutions were mixed in a large beaker. Then, the mixture was stirred contentiously on hot plate at room temperature. During the stirring operation to form 0.1 M CTAB was added into the mixture and further stirred for 45 minutes. Finally, aqueous ammonia (NH$_3$OH) was slowly added into the above stirring mixture to maintain the pH at approximately 10–11. The mixtures were further stirred continuously for 4 hours. Due to the OH$^-$ ions of ammonia, metal hydroxide were formed as precipitates. The obtained light brown precipitates were filtered and washed with doubly deionized water. Finally, precipitates were also washed methanol to reduce the organic impurity. The resulted precipitates were dried over the night at 100°C in oven. The entire synthetic process is depicted in Scheme 1.

**Characterization techniques**

Thermogravimetric analysis (TGA) of the un-annealed precipitates was performed on thermal analyzer (SDT Q600 V8.2 Build100). This analysis helps to find the suitable temperature required for the development of the crystalline spinel structure. The XRD analysis of the substituted magnesium nanoferrites was completed by using Philips X Pert PRO 3040/60 diffractometer. The Fourier Transform Infra Red (FT-IR) spectrum of the samples was also obtained at room temperature by using Nicolet
RESULTS AND DISCUSSION

Thermogravimetric analysis

Thermogravimetric analysis of the precursor precipitates was carried out between 298 and 1,273 K temperatures. Resulted thermo-gram is shown in the Figure 1. Thermogram shows a prominent mass loss that occurs in three temperature zones. The first temperature zone is from 300 to 469 K where maximum mass loss (1.2 mg) occurred. In this temperature zone, water desorption from the pores of un-annealed sample of MgFe$_2$O$_4$ takes place and anhydrous sample formed. The second temperature zone is from 568 to 659 K where 0.92 mg weight loss is observed. In this temperature zone, the hydroxide of the samples converted into oxide of ferrites. The third temperature zone is from 846 to 950 K where 1.18 mg weight loss is observed. In this temperature zone, rearrangement of oxides occurs to form the spinel ferrites [26].

Structural analysis

The crystallinity and phase purity of the all prepared compositions (Mg$_{1-x}$Co$_x$Ni$_y$Fe$_{2-y}$O$_4$) of the substituted magnesium nanoferrites were studied using X-ray diffraction analysis. The X-ray diffraction spectrum was obtained in the 2θ range of 20°–80° at room temperature. The X-ray diffraction patterns of all the six substituted magnesium nano ferrites samples are shown in Figure 2. The diffraction peaks at 30°, 35°, 43°, 57°, and 63° are due diffraction from the (220), (311), (400), (511), and (540) planes of the spinel magnesium nanoferrites, respectively. All these diffractions have accordance with cubic crystal system with space group Fd-3m according to the ICDD# 01-073-2440. The high intensity peak, at 2θ =35° arises due to diffraction from (311) plane, confirms the formation of spinel ferrite phase. The diffraction planes (220) and (400) at 30° and 43° are characteristic peaks of cations in tetrahedral (A) [27] and octahedral (B) sites [28], respectively. The Mg$^{2+}$ ions preferentially reside at B sites (octahedral sites), while ferric (Fe$^{3+}$) ions distributed almost equally at A sites (tetrahedral sites) and B sites (octahedral sites) [29,30].

The peaks positions in the substituted magnesium nanoferrites show slightly shifting due to differences in the sizes of the substituted ions (Co$^{2+}$, Ni$^{2+}$) and already occupied ions (Mg$^{2+}$, Fe$^{3+}$). It was also observed that doping of Co$^{2+}$ and Ni$^{2+}$ in the magnesium nanoferrites increases the intensities of the (220) and (400) planes. The increase in intensity of the (400) plane at 43° is prominent after the substitution of ions. It reflects that substitution of ions preferably takes place at octahedral sites (B sites). The lattice parameters of the un-substituted and submitted magnesium nanoferrites were determined by using Nelson–Riley standard refine-

![Scheme 1. Schematic illustration for synthesis of spinel ferrite nanoparticles.](attachment:image.png)
ment method [31]. The lattice parameter value for un-substituted magnesium nanoferrites is 8.3831 Å. This value increases up to 8.3843 Å in Ni-Co substituted magnesium nanoferrites. This increase is due to the substitution of large sized Ni^{2+} (0.69 Å) and Co^{2+} (0.72 Å) ions at the place of small sized Fe^{3+} ions (0.67 Å) and Mg^{2+} (0.66 Å) ions, respectively. The incorporation of larger cations at the place of smaller cations increases the size of the unit cell along with its parameters. The crystallite size of the synthesized sample was calculated by using following relationship:

\[ S = \frac{0.94 \times \lambda}{\beta \times \cos \theta} \]  

(1)

Here's' is the crystallite size, \( \lambda \) is wavelength (1.54Å) of the X-rays, while \( \beta \) is the FWHM in radian. The range of the crystallite size of the synthesized material was from 17–23 nm.

**FTIR study**

The FT-IR spectrum of the un-substituted magnesium ferrite was obtained at room temperature in the wave number range of 800 to 400 cm\(^{-1}\). The FT-IR spectra of the substituted and un-substituted magnesium nanoferrites are shown in Figure 3. The presence of the characteristic absorption band in the fingerprint region of the FT-IR spectrum of un-substituted magnesium nanoferrites confirms the formation of spinel structure [32,33]. The first absorption band for the tetrahedral stretching vibrations (\( \nu_1 \)) is observed at 550 cm\(^{-1}\). The second absorption band for the octahedral stretching vibrations (\( \nu_2 \)) sites is observed at 400 cm\(^{-1}\) [34]. The substitution of metal ions in the crystal lattice changes the nature of association between the atoms as a result lattice vibrations also changed. The cation-oxygen association, bonding force, and mass of the cations alter the level of lattice vibrations. The shifting of Fe^{3+} ions near to the oxygen ion at the tetrahedral site shifts the tetrahedral stretching vibrations (\( \nu_1 \)) band toward the higher frequency region.

This shifting of the vibration band toward higher wave number indicates stronger attraction forces between Fe^{3+} ion and oxygen ion. The absorption band at 1,652.1 cm\(^{-1}\) also specifies the existence of absorbed water molecules [35]. The relatively wider absorption band near the higher wave number ends (3,500–3,200 cm\(^{-1}\)) is a characteristic vibration band of O-H stretching [36].

**Dielectric properties**

Figure 4(a) is showing the effect of applied AC frequency on the dielectric constant of the all substituted nanoferrites samples. For all substituted magnesium nanoferrites, the value of dielectric constants decreases with an increase in the with applied AC frequency. This result indicates the common dielectric behavior of synthesized substituted magnesium nanoferrites. The falling curve with high steepness at low frequency region indicates the rapid
depression in the dielectric constant. The depression in the value of the dielectric constant is less prominent at higher AC frequency. The decrease in the dielectric constant is due to the electronic hopping between Fe$^{2+}$ and Fe$^{3+}$ that occurs at the adjacent octahedral sites (B sites). The electrons are transferred in the direction of applied field as a result of ferrites get polarized. This polarization of the ferrites drops the field inside the medium at higher frequency. That is the reason for the reduction of dielectric constant value with frequency [30].

According to the Koop and Maxwell Wagner model polarizations, at the interfaces and boundaries of the particles affect the dielectric properties of the magnetic material [37]. This is because the non-conducting grain boundary divides the large transmitting grains of the ceramics and ferrites. The grain boundaries are mostly formed during sintering operation as a result of the oxidation or reduction of crystallites in the porous materials [38].

Figure 4(b) is showing the effect of Co$^{2+}$ and Ni$^{2+}$ ion concentration on the dielectric constant. The result indicates that the dielectric constant value constantly decrease with increasing concentration of the substituent ions. On octahedral site (B sites) Fe$^{3+}$ ions at octahedral sites (B sites) play a basic role in the process of dielectric polarization. The substitution of Fe$^{3+}$ ions with Ni$^{2+}$ ions at octahedral sites decreases the dielectric polarization. Hence, the replacement of Fe$^{3+}$ ions at octahedral sites (B sites) with Co$^{2+}$ and Ni$^{2+}$ ions decreases the dielectric polarization. This reduction in dielectric polarization further reduces the dielectric constant with increasing concentrations of substituent’s cations. The increasing concentration of the substituted ions decreases the electronic hopping between Fe$^{2+}$ and Fe$^{3+}$ occurs at the adjacent octahedral sites (B sites).

Figure 5(a) shows the variation of dielectric loss of the all substituted magnesium nanoferrites with increasing frequency of the applied AC. The AC filed is applied externally in the broad frequency range (1 MHz to 3 GHz). The dielectric loss actually is the measure of the loss of energy as a result of heating the dielectric material in the applied AC field. After a certain frequency, the changes in external field polarity could not relate to the moving frequency of the charges. Figure 5(b) is indicating the dielectric loss with increasing concentration of the Co$^{2+}$ and Ni$^{2+}$ ions. The substitution of Co$^{2+}$ and Ni$^{2+}$ ions decreases the electronic conductivity in the substituted nanoferrites. Due to the inverse relationship of conductivity with resistivity, the depression in the dielectric loss can be understood by following relationship [39]:

$$\varepsilon'' = \frac{1}{\omega \varepsilon_0 \rho} \quad (2)$$

$$\omega = \text{angular frequency}$$

$$\rho = \text{resistivity of the material.}$$

At natural frequency, ions moves with vibrations and change their positions. A stage is reached when both applied and natural frequencies become equal, then maximum energy is transferred.

The variation of the dielectric tangent loss (tan δ) of the substituted magnesium nanoferrites with frequency is shown in Figure 6(a). It is observed that the value of tangent loss like other dielectric parameters also decreases with increase in frequency. A stage when hopping frequency of electrons, between Fe$^{2+}$ and Fe$^{3+}$ cations that are present at adjacent B sites, be higher than the applied electric field frequency then tangent loss be the highest. When an electric field of frequencies that is higher than the oscillating frequency of hooping of electrons is applied, then two states cannot follow hence minimum tangent loss occurs [40,41].

The value of tangent loss be high in the presence of low frequency applied field, but it becomes low at higher frequency. As a result the, power loss is also extreme at lower frequency.
frequency, but it is least at higher frequency [42]. Naturally, the anisotropic behavior is observed in spinel nanoferrites and they exhibited magnetic crystalline field in GHz region. It is found that the replacement of ions also shifted the peaks toward the low frequency region [43]. So, the resonance frequency decreases with increase in substitution of the other ions. The effect of the Co\(^{2+}\) and Ni\(^{2+}\) ion concentrations on the dielectric tan loss of the magnesium nanoferrites is also shown in Figure 6(b). The Figure 6(b) indicates that tan loss decreases with an increase in the concentration of the Co\(^{2+}\) and Ni\(^{2+}\) ions in the magnesium nano-ferrite.

**DC electrical resistivity**

By the study of dielectric and electric properties, appreciable amount of information can be collected about the band gap, activation energy, and behavior of electric charge carriers. Electrical resistivity of ferrites depends upon many parameters, such as density, chemical composition, and grain size. The DC electrical resistivity measurements were carried out by simple two probe method within the temperature range of 25°C–300°C [44]. The resistivity is given as follows:

$$\rho = \frac{RA}{l}$$

(3)

Here “\(\rho\)” is the resistivity, “R” the resistance, “A” the area of the pellet, and “l” is the thickness of the pellet.

$$\rho = \rho_0 e^{\Delta E/kT}$$

(4)

In Equation (4), “\(\Delta E\)” is the equation is activation energy and “\(k\)” is the Boltzmann constant.

The effect of DC electrical resistivity of the prepared samples was observed in temperature range of 300–373 K. The change in the resistivity as a function of absolute temperature for all substituted magnesium nanoferrites samples is shown in Figure 7. The decrease in the DC resistivity of the all samples with increasing absolute temperature is reveal-

![Figure 5](image1.png)

**Figure 5.** (a) Dielectric loss for Mg\(_{2-x}\)Co\(_x\)Ni\(_{y}\)Fe\(_{2-y}\)O\(_4\) \((x, y = 0.0-0.5)\) ferrite nanoparticles (b) Effect of Co-Ni contents/ mole on the dielectric loss.

![Figure 6](image2.png)

**Figure 6.** Tan loss for Mg\(_{2-x}\)Co\(_x\)Ni\(_{y}\)Fe\(_{2-y}\)O\(_4\) \((x, y = 0.0-0.5)\) ferrite nanoparticles (b) Effect of Co-Ni contents/ mole on the tan loss.
CONCLUSION

Pure spinel phase Co-Ni substituted magnesium nanoferrites were synthesized using micro-emulsion scheme. The XRD pattern of the magnesium nanoferrites confirmed the formation of crystalline spinel phase in all substituted magnesium nanoferrites. The grain size of the substituted nanoferrites was from 17 to 23 nm. The FT-IR spectrum of all samples consists of two prominent and characteristic absorption bands of 800 to 400 cm$^{-1}$.

ing their semiconductor nature. The activation energy that is required for the hopping of electron in the substituted magnesium nanoferrites were calculated by multiplying the slope of the curves with Boltzmann’s constant. The activation energy values decrease from 0.36 to 0.23 eV with increase in the concentration of the substituent.

ACKNOWLEDGMENT

The authors are thankful to the University of Gujrat, Allama Iqbal Open University Islamabad, University of Okara and The Islamia University of Bahawalpur for supporting this research project Pakistan.

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