Influence of cobalt substitution on the properties of
Ni$_{0.95-x}$Co$_x$Cu$_{0.05}$Fe$_2$O$_4$ ferrite nanoparticles

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Abstract
Cobalt substituted nickel copper ferrite samples with general formula Ni$_{0.95-x}$Co$_x$Cu$_{0.05}$Fe$_2$O$_4$, where (x= 0.00, 0.01, 0.02, 0.03, 0.04 and 0.05) were prepared by solid-state reactions method at 1373 K for 4h. The samples prepared were examined by X-ray diffraction (XRD), atomic force microscope (AFM), Fourier transform infra-red spectroscopy (FTIR) and Vickers hardness. X-ray diffraction patterns confirm the formation of a single phase of cubic spinel structure in all the prepared samples. XRD analysis showed that the increase in the cobalt concentration causes an increase in the lattice constant, bulk density ($\rho_b$) and the x-ray density ($\rho_x$), whereas porosity (p) and crystallite size (D) decrease. The Topography of the surface observed was found to be more uniform and homogeneous when the cobalt concentration increases, leading to a decrease in the roughness of the surface while average grains size increases. The FTIR spectra show two absorption bands, namely the high frequency band ($\nu_1$) in the range (1078-1081) cm$^{-1}$ and the low frequency band ($\nu_2$) in the range (418-459) cm$^{-1}$, which due to the vibrations of the tetrahedral and octahedral sites of Fe$^{3+}$–O$^2$ respectively, these bands confirm the spinel structure of the prepared ferrite nanoparticles. Vickers hardness was found to increase with cobalt concentration increases.

Key words
Ferrite nanoparticles, solid-state reactions method, cubic spinel ferrites, XRD, AFM, FTIR, Vickers hardness.

Article info.
Received: Dec. 2017
Accepted: Mar. 2018
Published: Sep. 2018

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Introduction

A ferrite is a type of ceramic compound composed of iron oxide (Fe₂O₃) combined chemically with one or more additional metallic element [1, 2]. The crystal structure of ferrites is divided into three types namely, spinel ferrite, hexagonal ferrite and garnet. Many ferrites are spinel ferrites. They have the general formula MFe₂O₄ in which (M = Cd²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Co²⁺ or a combination of these ions) and Fe occupy tetrahedral and octahedral cation sites of the crystalline structure, respectively, and O indicates the oxygen anion site. They are also called cubic ferrites [3]. Some ferrites exhibits hexagonal crystal structure, like barium and strontium ferrites BaFe₁₂O₁₉ (BaO:6Fe₂O₃) and SrFe₁₂O₁₉ (SrO:6Fe₂O₃), garnet ferrite has the formula R₃Fe₅O₁₂ where R is a rare earth ion like (Dy, Ln, Gd, Sm) [4].

In terms of their magnetic properties, the different ferrites are often classified into soft ferrites and hard ferrites, which refers to their low or high magnetic coercivity [5].

According to distribution of cations, spinel ferrites are classified in to three types; random spinel, normal spinel and inverse spinel. Among these ferrites, the inverse type ferrite is more interesting due to its high magneto crystalline anisotropy and high saturation magnetization [6].

Cobalt and nickel ferrites, belong to the category of inverse spinel ferrites. Therefore, by substituting the Co²⁺, Ni²⁺, and/or Fe³⁺ions by suitable cations, their structures undergo a change from inverse spinel to mixed spinel, leading to a corresponding change in the magnetic properties. Thus, by the choice of the cations as well as their distribution in tetrahedral and octahedral sites of the lattice, interesting and useful magnetic properties can be obtained [7].

Nickel ferrites and cobalt ferrites have been extensively studied in view of their good chemical and thermal stability, large permeability at high frequency, high electrical resistivity, mechanical hardness, magnetic anisotropy, high coercivity, moderate saturation magnetization, various exchange interactions and superparamagnetism etc. In addition to these, they exhibit ferromagnetism, originating from the magnetic moment of anti-parallel spins between Fe³⁺ ions at tetrahedral sites and Co²⁺ or Ni²⁺ ions at octahedral sites. Due to the magnetic properties, ferrite have various applications in numerous fields including microwave devices, recording media, magnetic fluids, gas sensors, high density information storage, ferro-fluids and catalysts [8]. Ni–Co ferrites are important electronic materials which are used in electronic devices suited for high-frequency applications in the telecommunication field. These materials are commercially used in high-quality filters, rod antenna radio frequency circuits, transformer cores, read/write heads for high-speed digital tape and operating devices [9].

The method of preparation plays an important role in the properties of the ferrite nanoparticles. Generally, ferrites are highly sensitive to preparation method, sintering conditions, amount of constituent metal oxides, various additives included as dopants and impurities [6].

Nickel Cobalt ferrite is synthesized by a variety methods including hydrothermal synthesis [10], sol–gel [6], high energy ball milling [11], microemulsion technique [12], chemical coprecipitation [13] and solid state reactions [14].
In this paper, cobalt–nickel–copper ferrite nanoparticles have been prepared by solid state reactions method. Solid-state reactions method is one of the most important methods used in the preparation of ferrites. This method is also called as ceramic method or high temperature method, because it involves heating the suitable constituents to high temperatures above 1273 K.

Solid-state reactions methods are the process of mixing materials in the form of fine powders by pressing them into a desired shape and volume, and then heating the compressed material in a controlled atmosphere to combine these grains together. Sometimes a subsequent phase is added after the heating process to incorporate the granules, with the aim of obtaining special properties or even improving existing properties [15]. The advantages of solid state reactions are that the process is very simple and is the preferred method when larger quantities of the products are required [16].

The present work deals with preparing of nanoparticles of nickel–cobalt–copper ferrite with general chemical formula Ni$_{0.95-x}$Co$_x$Cu$_{0.05}$Fe$_2$O$_4$, where (x=0.00, 0.01, 0.02, 0.03, 0.04 and 0.05) by solid-state reactions method and study the influence of substitution of cobalt on the (structural, topography and mechanical) properties for nickel cobalt copper ferrite nanoparticles.

**Experimental details**

Samples of nickel–copper ferrite substituted by cobalt having the chemical formula Ni$_{0.95-x}$Co$_x$Cu$_{0.05}$Fe$_2$O$_4$, where (x=0.00, 0.01, 0.02, 0.03, 0.04 and 0.05) were prepared by solid-state reactions method. Ferrite preparation requires high accuracy in all stages of synthesize in order to avoid contamination caused by impurities which affect the properties of the resulting ferrite, the following raw materials have been used, Fe$_2$O$_3$ (purity of 98%), CuO (purity of 99%), CoO (purity of 98%), NiO (purity of 99%). These oxides were mixed in agate mortar for 2 h. The homogeneous mixture was sintered at 973 K for 12 h and cooled slowly to room temperature. And then ground by a mortar of agate and continue to grind well until a homogeneous mixture is obtained. The pre-sintered powder were mixed with small quantity of polyvinyl alcohol (PVA) as an organic binder. The mixture was dried and pressed in pellets of 2 mm thickness and 10 mm diameter by applying a pressure of 5 tons. The samples were sintered again at 1373 K for 4 h and allowed to cool slowly. The specimens are then refined using soft silicon carbide sheets to be ready for testing.

The structural properties were obtained using X-ray Diffractometer (XRD-6000 from SHIMADZU) using Cu Kα radiation (λ = 1.5406 Å), using (2θ) = 10° - 80° in steps of 5 deg/s. The Fourier transform infrared (FTIR) spectra of the powders were recorded using FTIR spectrometer (FTIR 8400S from Shimadzu) in the wave number range (4000–400) cm$^{-1}$ to confirm the spinel structure of the samples. The surface Topography of the ferrite samples was observed with atomic force microscope (AFM) AA3000 from Angstrom Advanced Inc. USA.

The hardness of prepared samples were calculated by Vickers Hardness Test, with the insertion of a diamond pyramid with a square base and a vertical angle of 136° and applied load (500 g).
Results and discussion
1. Structural analysis

The XRD patterns of the ferrites compounds having the formula Ni_{0.95-x}Co_xCu_{0.05}Fe_{2}O_{4} (where x= 0.00, 0.01, 0.02, 0.03, 0.04, 0.05) prepared in powdered metallurgy at 1373 K for 4 h was shown in Fig.1. The sharp peaks in x-ray diffraction patterns confirm their polycrystalline structure with single spinel phase. The strong diffraction from the planes (220), (311), (222), (400), (422) (511), (440) and (533) indexed using Bragg’s law indicating the presence of cubic spinel structure [17], and the peaks match well with characteristic reflection of NiFe_{2}O_{4} (JCPDS card number 89-4927).

From results of x-ray diffraction the inter-planar spacing (d) values for Ni_{0.95-x}Co_xCu_{0.05}Fe_{2}O_{4} samples were calculated using Bragg’s law [18]:

\[2d \sin \theta = n\lambda\]  

Table 1 shows the Miller Indices (h k l) and the distance (d) obtained from x-ray diffraction results for Ni_{0.95-x}Co_xCu_{0.05}Fe_{2}O_{4} samples.

<table>
<thead>
<tr>
<th>(h k l)</th>
<th>X1=0.00 d (Å)</th>
<th>X2=0.01 d (Å)</th>
<th>X3=0.02 d (Å)</th>
<th>X4=0.03 d (Å)</th>
<th>X5=0.04 d (Å)</th>
<th>X6=0.05 d (Å)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>4.18358</td>
<td>4.06480</td>
<td>4.06419</td>
<td>4.16343</td>
<td>4.05214</td>
</tr>
<tr>
<td>(4 2 2)</td>
<td>3.41260</td>
<td>3.30019</td>
<td>3.25208</td>
<td>3.28516</td>
<td>3.31564</td>
<td>3.34863</td>
</tr>
<tr>
<td>(3 3 3)</td>
<td>2.93958</td>
<td>2.88770</td>
<td>2.90049</td>
<td>2.89363</td>
<td>2.90026</td>
<td>2.90147</td>
</tr>
<tr>
<td>(2 2 0)</td>
<td>2.47731</td>
<td>2.43805</td>
<td>2.45169</td>
<td>2.44960</td>
<td>2.44831</td>
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</tr>
<tr>
<td>(0 1 4)</td>
<td>2.38728</td>
<td>2.39193</td>
<td>2.38320</td>
<td>2.38183</td>
<td>2.38574</td>
<td>2.38647</td>
</tr>
<tr>
<td>(3 1 1)</td>
<td>2.07155</td>
<td>2.06113</td>
<td>2.08363</td>
<td>2.06679</td>
<td>2.06907</td>
<td>2.06497</td>
</tr>
<tr>
<td>(2 2 2)</td>
<td>1.80610</td>
<td>1.80799</td>
<td>1.81523</td>
<td>1.80337</td>
<td>1.80354</td>
<td>1.80196</td>
</tr>
<tr>
<td>(4 0 0)</td>
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<td>1.68102</td>
<td>1.67029</td>
<td>1.67324</td>
<td>1.67498</td>
<td>1.68316</td>
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<tr>
<td>(4 2 2)</td>
<td>1.59025</td>
<td>1.59025</td>
<td>1.58224</td>
<td>1.57538</td>
<td>1.58079</td>
<td>1.60263</td>
</tr>
<tr>
<td>(5 1 1)</td>
<td>1.53309</td>
<td>1.53559</td>
<td>1.54003</td>
<td>1.52214</td>
<td>1.52645</td>
<td>1.53586</td>
</tr>
<tr>
<td>(4 4 0)</td>
<td>1.45967</td>
<td>1.45717</td>
<td>1.46173</td>
<td>1.46173</td>
<td>1.45722</td>
<td>1.46787</td>
</tr>
<tr>
<td>(5 3 3)</td>
<td>1.36142</td>
<td>1.36886</td>
<td>1.36489</td>
<td>1.36649</td>
<td>1.36646</td>
<td>1.37500</td>
</tr>
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</table>
Fig. 1: The XRD patterns for Ni$_{0.95}$Co$_x$Cu$_{0.05}$Fe$_2$O$_4$ samples.
Lattice constant \( a \) for FCC system was calculated for (311) plane using the following relationship [19]:

\[ a = d \sqrt{h^2 + k^2 + l^2} \tag{2} \]

\( h, k, l \) are the Miller indices.

The lattice constant \( a \) increases with the increase of Co concentration in the \( \text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4 \) with \( x = 0.0, 0.01, 0.02, 0.03, 0.04, \) and 0.05 as shown in Fig. 2 and listed in Table 2. This difference can be attributed to the difference in the ionic radius of the component ions. The \( \text{Co}^{2+} \) ions have larger ionic radius (0.745 Å) than \( \text{Ni}^{2+} \) (0.69Å), \( \text{Cu}^{2+} \) (0.73Å) and \( \text{Fe}^{3+} \) (0.645 Å) ions. The substitution by the larger ions results in expansion of lattice then the lattice constant increased [20-22].

![Graph showing the relationship between the lattice constant and cobalt concentration.](image)

**Fig. 2: The relationship between the lattice constant and cobalt concentration.**

The crystallite size of the ferrite samples were calculated from XRD pattern and by using Debye–Scherrer equation by measuring the FWHM of the strongest intensity (311) peak [23]:

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \tag{3} \]

where 0.9 is constant depends on the shape of the crystal, \( \lambda \) is the x-ray wavelength and \( \beta \) is the full width half maximum (FWHM) in radians.

It is noted from Fig. 3 and Table 2 that the crystallite size gradually decreased with increasing Co concentration. Decrease in the crystallite size may be due to the substitution of larger \( \text{Co}^{2+} \) ions (0.745Å) with smaller \( \text{Ni}^{2+} \) ions (0.69Å) [21, 24].

![Graph showing the variation between the crystallite size and cobalt concentration.](image)

**Fig. 3: The variation between the crystallite size and cobalt concentration.**

The bulk density \( \rho_m \) was determined using formula [21, 25]

\[ \rho_m = \frac{m}{V} = \frac{m}{\pi r^2 h} \tag{4} \]

where \( m, V, r, \) and \( h \) are mass, volume, radius, and thickness of the pellet of sample, respectively.

Fig. 4 and Table 2 showed that the bulk density increases as the cobalt concentration increases. This is due to
the atomic weight of cobalt (58.933 amu), which is greater than the atomic weight of nickel (58.693 amu) [26].

The x-ray density $\rho_x$ of the prepared samples was calculated using the equation [22, 27]:

$$\rho_x = \frac{9 M}{N_A a^3}$$  \hspace{1cm} (5)

where $M$ is the molecular weight of the samples, $N_A$ is the Avogadro’s number ($6.023 \times 10^{23}$ atom/mole). Calculated values of x-ray density are shown in Fig. 5 and listed in Table 2. It is observed that the value of x-ray density increase with increasing concentrations of Co$^{+2}$ ion. This behavior may be due to the fact that the density and atomic weight of Co atoms are (8.90 g/cm$^3$ and 58.933 amu), which are greater than those of Ni atoms (8.908 g/cm$^3$, 58.693 amu) and Fe atoms (7.874 g/cm$^3$, 55.845) [22, 28].

In general, the x-ray density of nano-ferrites is higher than bulk density due to the existence of pores which depends on the sintering condition [21, 22, 25].

The porosity $P$ of ferrite samples were determined using the relation [22, 29]

$$P = \left(1 - \frac{\rho_m}{\rho_x}\right) \times 100\%$$  \hspace{1cm} (6)

Fig. 6 and Table 2 show the increase in cobalt concentration leads to decrease in porosity due to the larger ionic radius of Co compared to Ni. This clearly indicates that Ni$^{+2}$ entering the A-sites and replacing the Co$^{+2}$, this result is consistent with the researchers [30, 31].
The relationship between porosity and cobalt concentration.

Table 2: Structural parameters of Ni_{0.95-x}Co_{x}Cu_{0.05}Fe_{2}O_{4} (x = 0.0–0.05).

<table>
<thead>
<tr>
<th>Co concentration (x)</th>
<th>a(Å)</th>
<th>D (nm)</th>
<th>(\rho_{m}) (g/cm(^3))</th>
<th>(\rho_{s}) (g/cm(^3))</th>
<th>P %</th>
<th>Vickers Hardness (N/mm(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>6.87054</td>
<td>43.493</td>
<td>1.02</td>
<td>1.28574</td>
<td>20.66825</td>
<td>509</td>
</tr>
<tr>
<td>0.01</td>
<td>6.88029</td>
<td>37.932</td>
<td>1.06</td>
<td>1.58586</td>
<td>33.1593</td>
<td>519.3</td>
</tr>
<tr>
<td>0.02</td>
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<td>1.11</td>
<td>1.56509</td>
<td>37.932</td>
<td>29.07756</td>
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<td>0.03</td>
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<td>1.60365</td>
<td>26.41786</td>
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<tr>
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<td>1.23</td>
<td>1.62836</td>
<td>24.46388</td>
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</tr>
<tr>
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<td>30.002</td>
<td>1.36</td>
<td>1.63473</td>
<td>16.80583</td>
<td>554.9</td>
</tr>
</tbody>
</table>

2. AFM analyses

The Topography of the surface of the Ni_{0.95-x}Co_{x}Cu_{0.05}Fe_{2}O measured by Atomic force microscope AFM and average grains size, average surface roughness and average square root were determined. Figs. 7-9 show the two-dimensional images, three-dimensional pictures and distribution of the grains respectively.

It is clearly observed the uniformity and homogeneity of the particles. It has been found that average square root and the average roughness decreases with increasing cobalt concentration while average grains size increases as listed in Table 3.

This is due to cohesion between the cobalt and nickel grains. This uniform and homogeneous distribution increases with the increased concentration of the cobalt. This results in a strong cohesion between the granular boundary and this increases its movement, causing growth in the size of the granules and thus reducing the internal and surface defects in the structural structure. This improves the mechanical, electrical and magnetic properties. This is consistent with the porosity results achieved in the results of x-ray diffraction [32].

The average grain size of the samples obtained from AFM images is larger than the crystallite size calculated using XRD measurement, which indicates that each grain is formed by aggregation of number of nanocrystals [6].
Table 3: Values obtained from AFM analysis.

<table>
<thead>
<tr>
<th>Co Concentration (x)</th>
<th>average roughness (nm)</th>
<th>average square root (nm)</th>
<th>average grains size (nm)</th>
</tr>
</thead>
<tbody>
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<td>0.00</td>
<td>1.93</td>
<td>0.777</td>
<td>85.46</td>
</tr>
<tr>
<td>0.01</td>
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<td>0.02</td>
<td>0.665</td>
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</tr>
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<td>95.03</td>
</tr>
<tr>
<td>0.04</td>
<td>0.395</td>
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<td>97.85</td>
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<tr>
<td>0.05</td>
<td>0.283</td>
<td>0.453</td>
<td>109.84</td>
</tr>
</tbody>
</table>

Fig. 7: AFM images of Ni_{0.95-x}Co_xCu_{0.05}Fe_{2}O_{4} in two-dimensional (2D).
Fig. 8: AFM images of Ni$_{95.5}$Co$_x$Cu$_{0.5}$Fe$_2$O$_4$ in 3 dimension.
Fig. 9: Particle size distribution of Ni\textsubscript{0.95-x}Co\textsubscript{x}Cu\textsubscript{0.05}Fe\textsubscript{2}O\textsubscript{4}\ sample.

3. FTIR analysis

FTIR spectra for all specimens (Ni\textsubscript{0.95-x}Co\textsubscript{x}Cu\textsubscript{0.05}Fe\textsubscript{2}O\textsubscript{4}) were shown in Fig. 10. The normal and inverse cubic spinel have two fundamental absorption bands – the first band is due to tetrahedral and the second is due to octahedral complexes [33]. The FTIR spectra shows two main absorption bands of the cobalt ions on both tetrahedral and octahedral positions \(v_1\) and \(v_2\) in the range of (1078-1081) cm\(^{-1}\) and (418–459) cm\(^{-1}\), respectively except the sample
(Ni_{0.91}Co_{0.04}Cu_{0.05}Fe_{2}O_{4}) showed an additional absorption range near the range (2300 cm^{-1}). It was also observed that the absorption nature at the range of (1078 cm^{-1}) is wider and very strong, but the absorption at the range of (418 cm^{-1}) is less than (1078 cm^{-1}). The cause of the absorption near the range of (481 and 1078) cm^{-1} is due to the expansion of the vibration of Fe^{3+}O^{2-} multi-polarization. These bands confirm the spinel structure of the prepared ferrite nanoparticles.

The IR spectrum gives information about the change in the molecular structure of the ferrite due to changes in the band length when adding cobalt. It is observed that the width of infrared beam depends on the cation distribution of the material. On this assumption, the beam width of the octahedral sites was found to be almost constant for all samples, but the tetrahedral sites increased with the replacement of cobalt with nickel ferrite [34, 35].

4. Hardness results

Fig. 11 shows the relationship between Vickers hardness and cobalt concentration, it observed when Cobalt concentration increases the hardness values increase. The reason for increase in hardness values is due to the increase in bulk density and decrease of porosity which pre-defined in x-ray diffraction results. A decrease in porosity leads to increased durability of the sample i.e. increased hardness. Low porosity materials have high mechanical resistance ie high hardness.
In contrast, high porosity materials have low mechanical resistance and this is consistent with the findings of the [36, 37] for materials and other composites.

![Fig. 11](image)

**Fig. 11**: Relationship between hardness and cobalt concentration of \( \text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4 \) sample.

**Conclusions**

1. Nickel–copper ferrite substituted by cobalt having the chemical formula \( \text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4 \), where \( x = 0.00, 0.01, 0.02, 0.03, 0.04 \) and \( 0.05 \) were successfully prepared by solid-state reactions method.
2. XRD analyses showed that the crystalline structure of all prepared samples was cubic spinel structure with preferential orientation in the (311) direction.
3. Increasing the concentration of cobalt in the compound results in increased X-ray density, bulk density, average grain size and Vickers hardness, while porosity, average surface roughness and average square root decreases.
4. The FTIR spectra shows two main absorption bands of the cobalt ions on both tetrahedral and octahedral positions \( \nu_1 \) and \( \nu_2 \) in the range of \((1078-1081) \text{ cm}^{-1} \) and \((418–459) \text{ cm}^{-1} \), respectively.

**References**