

Fabrication And Studies Of structural, Dielectrical Properties Of ($Ni_{0.95-x}Co_xCu_{0.05}Fe_2O_4$) Composites by Powder Technology Method

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Abstract

In this paper, the preparation of compounds, $Ni_{0.95}Co_xCu_{0.05}Fe_2O_4$) by using powder technology method where ($X = 0, 0.01, 0.02, 0.03, 0.04,$ and 0.05) annealing at 1100 C temperature for four hours. A series of samples were carried out for the study of structural properties by measuring x-ray diffraction (XRD). It included the study of the lattice constant (a), the particle size (D), the porosity (P) and the virtual density by x-ray diffraction, (x-ray density) as well as the study of Dielectric properties using the LCR , the study of the loss of dielectric meter tand and the electric conductivity (σ_{ac}). The results of X-ray diffraction showed that all samples had a crystalline,) and also showed that the Lattice constant (a) increases as the content of cobalt increases the density of the particles calculated by X-ray diffraction increases with the cobalt content, while the porosity and particles size decreases as the cobalt content increases, and the electrical show that the real and imaginary dielectric and electrical conductivity using current decreases as frequency increases, the real electrical dielectric constant increases as the cobalt content increases, while the imaginary part of dielectric constant and electrical conductivity decreases as the cobalt content increases.

Key Word : Dielectrical Properties , XRD , Powder Technology Method , Ni , Co & Cu Ferrites .

1- Introduction

The ferrites are one of these materials that continue to evolve. These materials are included in the components of the construction of the circuits that work at high and low frequencies[1]. The ferrites have crystalline structures in various forms, such as soft and hard ferrites are the basis of the magnetic materials of electrical devices[2]. The only fret found in nature is magnetic iron oxide (Fe_3O_4), which contains one type of positive ions (iron) [3]. Ferrites have magnetic properties that enable us to distinguish it from other materials such as paramagnetic materials, diamagnetic materials, and ferromagnetic materials in terms of their magnetic arrangement [4].

2- Experimental details

2-1 Preparation of Samples

(NiO, CoO, CuO, FeO) were used in the preparation of nitrate compounds ($Ni_{0.95-x}Co_xCu_{0.05}Fe_2O_4$), with a 75 micrometer Size measured sieves, with purity ranging from 98-99%. The Samples were prepared in different proportions ($X= 0.00, 0.01, 0.02, 0.03, 0.04$ and 0.05). This was done by calculating the weight ratios of the materials used in the preparation of the models using a sensitive balance. The materials were then mixed together using a mortar with mortar and grinding to obtain Homogeneous mixture for modeling preparation. The material was then filtered at a temperature of (700 C) using an electric furnace for 12 hr in order to obtain the frit and remove the impurities in it and obtain oxides of the original oxides. The mixture is then re-milled and, after a homogeneous mixture, the mixture is sifted in order to obtain smooth granules and after the process of sieving, the material is placed in the vinyl liquor and the mixture is then extracted to obtain the models using a hydraulic piston for 7 min. Finally, samples are modeled at a temperature of ($1100\text{ }^\circ\text{C}$) for 4 hr and configuration chemical was based on the ratios of preparing for the proposed by the sintering. So using an electric oven with a

temperature higher than $1100\text{ }^\circ\text{c}$ type ELF and the origin of England, and then the models are left until cool until the room temperature reaches. Finally, we will get solid samples but their surface is not smooth. Because of the heating process, the diameter of the sample will be slightly reduced Because increasing the temperature increases the size of the granules.

2-2-1 Structural Properties

The study of structural properties helps in the interpretation of many of the results that accompany the physical properties according to the changing conditions of preparation and other effects. The most important tests that are concerned with the study of structural properties are:

1- (X-ray) diffraction analysis

X-ray diffraction has enabled the study of the crystalline structure of materials at the atomic level [5-7]. The rate of interstitial distances of the crystal levels can also be identified [6-10], as are some synthetic parameters such as Grain size and the curved width at the middle of the top and the fixed lattice [11,12].

X-ray is an electromagnetic beam of high energy and short wavelength [9,10], wavelength is within range ($0.1-10$) A between ultraviolet rays and kamma [5,13].

Calculations

1. Grain Size

The grain size can be found by using the Debai-Scharr [14]:

$$D = 0.9 \lambda / \beta \cos(\theta) \text{ ----- (1)}$$

(β) represents the radial angle at the middle of the maximum intensity (FWHM).

2- Lattice constant

The splicing constant can be calculated by using the following mathematical relationship [15]:

$$a = d (h^2 + k^2 + l^2)^{1/2} \text{ ----- (2)}$$

As:

a:Lattic constant.

d: Distance between levels.

(h k l): Miller coefficients.

3- Physical density

Physical density is calculated by the following mathematical relationship [16]:

$$P_a = m / v \text{ ----- (3)}$$

m: mass of samples (gram).

V: Volume of Samples (cm^3).

4- Virtual density

Virtual density can be calculated by the following mathematical relationship [17]:

$$\rho_{x\text{-ray}} = 8M / NA a^3 \text{ ----- (4)}$$

M: Molecular mass

NA : Avocado number (6.023×10^{23}).

After calculating the density, porosity can be calculated through the following relationship [18]:

$$P = (1 - \rho_a / \rho_{x\text{-ray}}) \times 100\% \text{ ----- (5)}$$

2-2-2 Electrical Properties of Ferrite

The Ferrite has electrical properties at the high frequency and heat of various types such as the electric insulation constant and the resistance of the Oum, which led to the expansion of its use in all areas, we will clarify in this chapter some of these characteristics.

1- Losses Angle And Dielectric Losses.

When an electrical field is exposed to any insulating material, a certain amount of electrical energy will dissipate in that material. This energy is transformed from electrical energy to thermal energy. This phenomenon is called the phenomenon of loss of energy insulation. i.e, (the average time of electrical energy discharged by heat when the material is exposed to an alternating electric field), it can be said that the amount of loss in the insulation capacity when the insulator is under a certain voltage effect, i.e, a voltage called Dielectric Losses [19].

2- Electrical Conductivity

Permission is defined as the ability of insulating materials to store electrical charges. If we have two parallel metal plates, the space of each other is (A) and the distance between them is (d) they will be expanded (C). If we impose that the space is air, so:

$$C = \epsilon_0 \epsilon_r A / d \text{ ----- (6)}$$

As:

ϵ_0 : vacuum tolerance and value (8.854×10^{-12} F / m)

In the case of placing an insulating material in the empty space, the amplitude of the expanded in the case of laying the insulation will increase to become:

$$C = \epsilon A / d \text{ ----- (7)}$$

ϵ : the thickness of the insulating material that separating the two plates [20].

Electrical Measurements

Electrical measurements shall include the measurement of the static insulation of both types of real (ϵ') and imaginary (ϵ'') As measured by alternating conductivity measurement ($\text{ac}\sigma$)

As well as measuring the shadow angle of loss, as a function of frequency change (10 Hz -200000 Hz) and at room temperature. After measuring the thickness (d) and the radius (r), we can find the space in order to find the real isolation constant (ϵ') through the following mathematical relationship [21]:

$$\epsilon' = C d / \epsilon_0 A \text{ ----- (8)}$$

C: Expandable capacity.

D: thickness for each model.

ϵ_0 : Static smear-free space 8.85×10^{-12} pf / cm.

A: The space of the prepared models was found by ($A = r^2 \pi$).

As for the calculation of the imaginary isolation constant it is by the following mathematical relationship [20]:

$$\epsilon'' = \epsilon' \tan \delta \text{ ----- (9)}$$

As for the alternating electrical conductivity, it is calculated by the following relationship [22]:

$$\text{ac}\sigma = \omega \epsilon' \epsilon_0 \tan \delta \text{ ----- (10)}$$

σ

Ω : angular frequency ($2\pi f$).

Results and discussion

Results of X-Ray Diffraction

Figure 1 shows the results of x-ray diffraction of the fractite compounds ($\text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4$) prepared in powdered and plastic technology at a temperature of 1100°C and for (4 hr). Through x-ray diffraction, it was confirmed that the thermal reactions and the conversion of the used oxides to the fry required for ($\text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4$) were made to values ($X = 0.00, 0.01, 0.02, 0.03, 0.04, 0.05$).

It is noted that all models have a faceted FCC structure to get properties of the following ($a=b=c$, $\alpha=\beta=\gamma$) as shown in Fig. 1). Also, good diffraction peaks at specific levels are shown as shown in Table 1. Note that the higher the cobalt content, the lower the intensity of some diffraction peaks This is due to the spread of positive ions and to different locations of A-site and B-site within the lattice [21].

(Figure 1)) (a-f) shows x-ray diffraction of ($\text{Ni}_{0.95-x}\text{Co}_x\text{Cu}_{0.05}\text{Fe}_2\text{O}_4$ ($X = 0.00, 0.01, 0.02, 0.03, 0.04, 0.05$)). The impurities in these materials and the purity of play a major role in the control conditions the appropriate, terms of the time of the burn temperature for materials ferrites. I have been observed that these results match the card (ASTM) to check substances and also match these results with the results of researchers [21,22] .

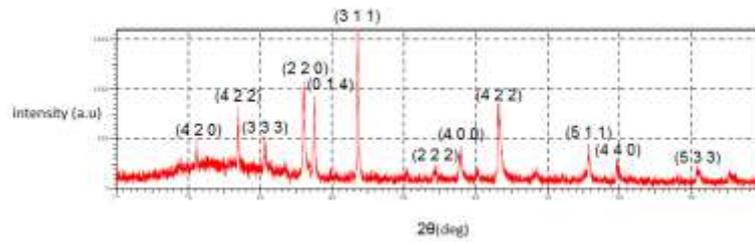


Figure (a) shows x-ray diffraction at X = 0.00

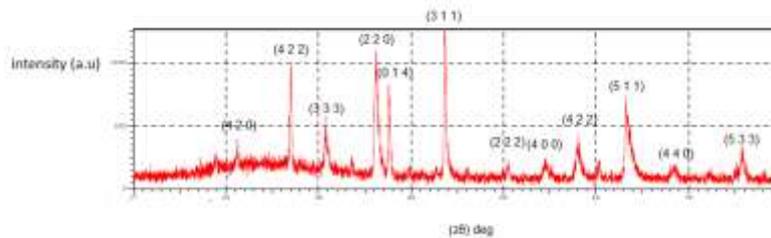


Figure (b) shows X-ray diffraction at X = 0.01).

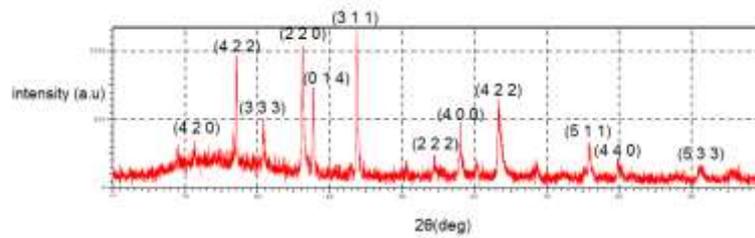


Figure (c) shows x-ray diffraction at X = 0.02)

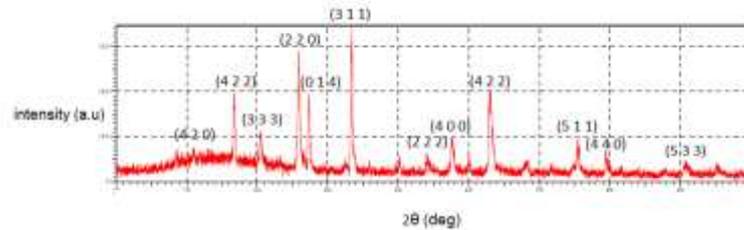


Figure (d) shows x-ray diffraction at (X = 0.03)

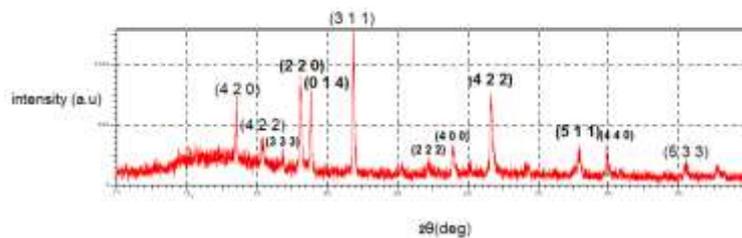


Figure (e) shows X-ray diffraction at (X = 0.04)

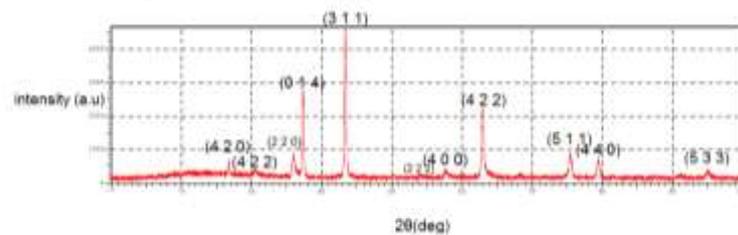


Figure (F) shows X-ray diffraction at (X = 0.05).

Table shows the coefficients of Miller (h k l) and the distance (d) obtained from X-ray diffraction results for Ni_{0.95-x}CoxCu_{0.05}Fe₂O₄ models.

h k l	X ₁ =0.00 d (Å)	X ₂ =0.01 d (Å)	X ₃ =0.02 d (Å)	X ₄ =0.03 d (Å)	X ₅ =0.04 d (Å)	X ₆ =0.05 d (Å)
4 2 0	4.22299	4.18358	4.06480	4.06419	4.16343	4.05214
4 2 2	3.41260	3.30019	3.32508	3.28516	3.31564	3.34863
3 3 3	2.93958	2.88770	2.90049	2.89363	2.90026	2.90147
2 2 0	2.47731	2.43805	2.45169	2.44960	2.44831	2.47987
0 1 4	2.38728	2.39193	2.38320	2.38183	2.38574	2.38647
3 1 1	2.07155	2.06113	2.08363	2.06679	2.06907	2.06497
2 2 2	1.80610	1.80799	1.81523	1.80337	1.80354	1.80196
4 0 0	1.68216	1.68102	1.67029	1.67324	1.67498	1.68316
4 2 2	1.59025	1.59025	1.58224	1.57538	1.58079	1.60263
5 1 1	1.53309	1.53559	1.54003	1.52214	1.52645	1.53586
4 4 0	1.45967	1.45717	1.46173	1.46173	1.45722	1.46787
5 3 3	1.36142	1.36886	1.36489	1.36649	1.36646	1.37500

Structural Parameters Calculation

1. Grain Size

The particle size D (μm) was calculated for all samples using the relationship (1) So depending on the values of FWHM and cos² highest the top of all samples. The granular volume was gradually reduced with the cobalt concentration increased as shown in Figure (2).

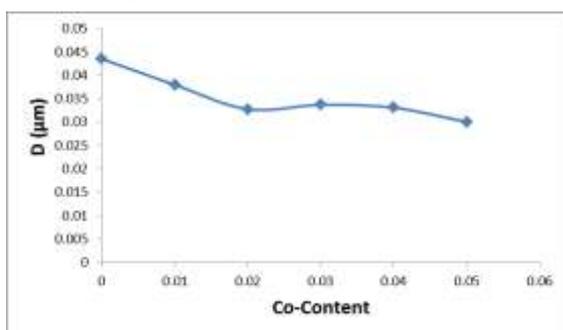
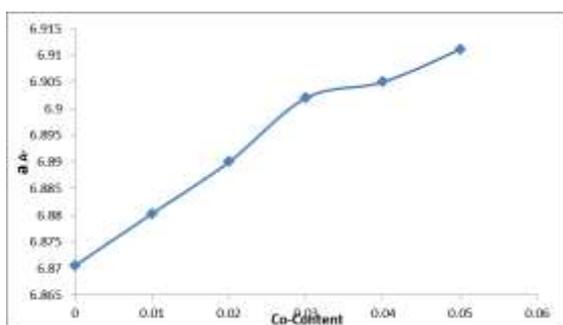


Figure (2) shows the relationship between grain size and cobalt content

2. Lattice Constant

The splicing constant (a) was calculated for all models using the relationship (2). The splicing constant was gradually increased with the cobalt concentration increased as shown in Table 2), which is shown in Fig. (3). This can be explained on the basis of ionic and cobalt ion substrates. The diameter of the cobalt ion (0.125nm) is greater than that of the nickel ion (0.124nm).



(Fig. 3) shows the relationship between the cobalt constant and cobalt concentration

3. Physical Density

The physical density was calculated by using the relationship 5 and Figure 4 showed that the physical density increases as the cobalt concentration increases. This is due to the atomic weight of cobalt (58.9332 amu), which is greater than the atomic weight of nickel (58.71 amu).

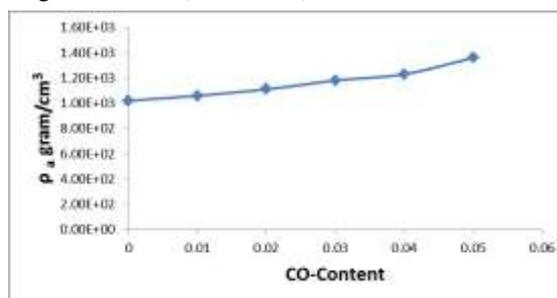


Figure (4) shows the relationship between physical density and cobalt concentration

3. Virtual Density

The virtual density was calculated by X-ray (X-ray) by using the relationship (6). It was found that the apparent density of X-ray diffraction increased as the cobalt concentration increased due to the density dependence on the molecular weight of the compound as shown in Fig.

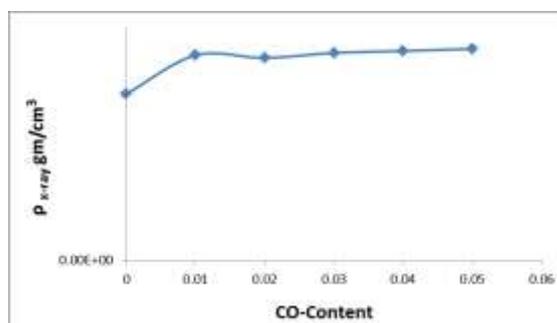
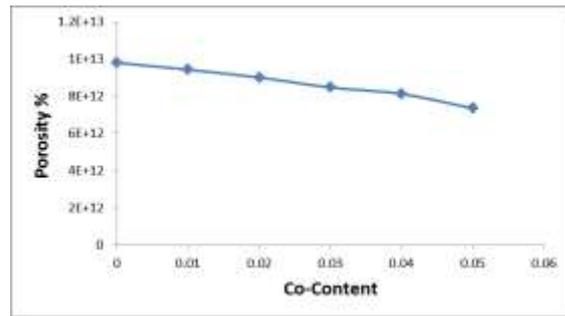


Figure (5) shows the relationship between the apparent density and cobalt concentration

4. Porosity

After calculating the physical density and density in terms of X-ray diffraction, it is easy to calculate the porosity of all models using the relationship (7), that

the increase in cobalt concentration leads to a decrease in porosity as shown in Figure (6).



Figure(6) shows the relationship between porosity and cobalt concentration.

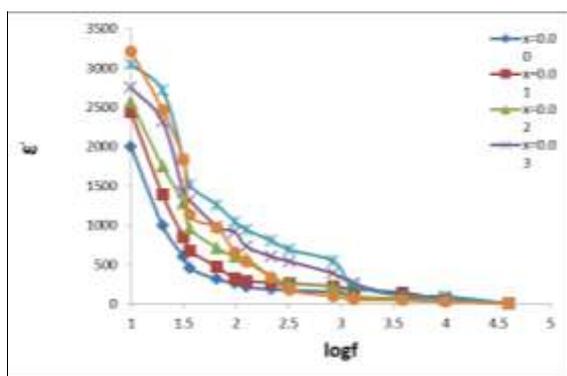
Table 2 shows the results obtained from X-ray diffraction of Ni_{1-x}Cu_xCo_{0.05}Fe₂O₄ models.

Co- concentration	Grain Size (μm)	Lattice constant(A°)	P _a (gm/cm ³)	x-ray(gm/cm ³) ρ	Porosity %
0.00	0.043	6.87054	1.02	1.28574	9.80392
0.01	0.037932	6.88029	1.06	1.58586	9.43396
0.02	0.032732	6.89	1.11	1.56509	9.00901
0.03	0.033666	6.902	1.18	1.60365	8.47458
0.05	0.033258	6.905	1.23	1.62836	8.13008
0.06	0.030002	6.91113	1.36	1.63473	7.35294

Electrical Results

1. Real Static electrode insulation:

Figure 7 shows the change in the true electrostatic insulation as a frequency function in the 100 Hz-1 MHz range for Ni_{0.95-x}Co_xFe₂O₄ and for all X values. (10) It is clear from the figure that the values of the static insulation constant at low frequencies are high but rapidly decreases at high frequency until they reach a constant value. It was found that the increase in the true electrical insulation of the models with increased cobalt concentration indicates a better homogeneity of the compound, increased density and shrinkage of pores. Co + 2 is a non-magnetic material, which increases the electrode polarization and thus increases the real electrical insulation constant with increasing cobalt concentration.

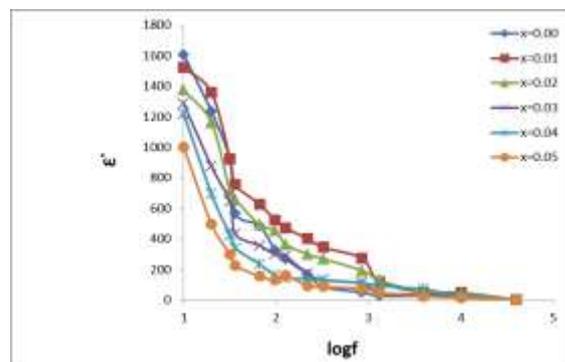


(Fig. 7) shows the relationship between the constant and the true electrical insulation frequency

2. Loss factor and angle of loss

Figure (8) and (9) shows the change in the loss factor and the angle of loss with the frequency at room temperature of the Ni_{0.95-x}Co_xCu_{0.05}Fe₂O₄ frit.

The loss coefficient can be found using the relationship (11) by the values of the true electrical insulation constant and the angle of loss. The loss factor is the measure of the loss of energy within the insulating medium and is an important part of the basic loss in the fracture. Of Fig. (8) and (9) shows the maximum values of the loss factor and the loss angle at the low frequencies due to a strong correlation between the conductivity mechanism and the insulation behavior in the fret, where the frequency of the electric charge carriers between Fe²⁺ and Fe³⁺ is approximately equal. The amplitude of the field is obtained. As shown in Figs. 8 and 9, when the cobalt concentration increases, the loss factor decreases and the loss angle remains due to the degree of homogeneity and nature of the crystalline structure of the ferrite produced as well as the granular growth and the process of removing the pores.



(Fig. 8) shows the relationship between the imaginary dielectric constant with frequency

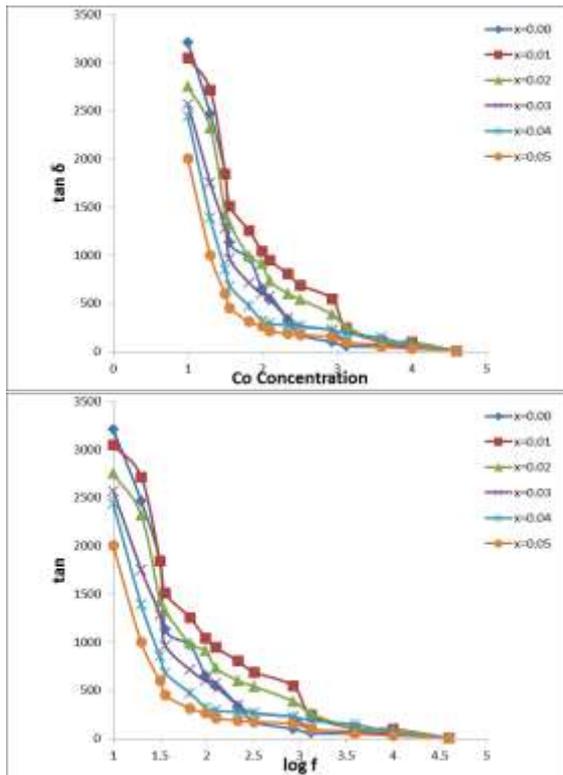


Figure (9) shows the relationship between the shadow of the dielectric loss with frequency

3. Alternating electrical conductivity:

Frites generally have low electrical conductivity ranging $\Omega^{-1} \cdot \text{cm}^{-1}$ (10^{-9} - 10^{-4}) at very low frequencies and by increasing the frequency of the outer field

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applied to the ferrite material, the electrical conductivity will increase and Figure 10 shows the change in the values of the electrical conductivity with the frequency. , And it is clear that the values that determine the values of the imaginary isolation and frequency, as the rest of the variables are fixed amounts, and since its value is low for frequency, frequency plays a large role in determining the increase of connectivity. The increased concentration of cobalt blocks the movement of the electron between Fe^{+2} and Fe^{+3} in the octagonal locations, causing a decrease in alternating conductivity [30-32].

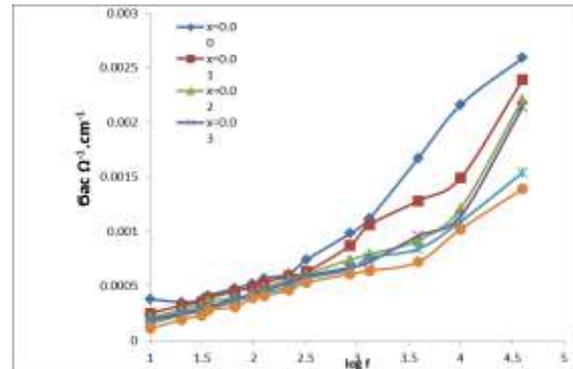


Figure (10) shows the relationship between alternating electrical conductivity with frequency

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تصنيع ودراسة الخصائص التركيبية والعزلية للمترابك $(Ni_{0.95-x}Co_xCu_{0.05}Fe_2O_4)$

بطريقة تقانة المساحيق

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قسم الفيزياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

الملخص

تم في هذا البحث تحضير مركبات نيكول – كوبالت – نحاس فرايت ذات الصيغة الكيميائية $(Ni_{0.95}Co_xCu_{0.05}Fe_2O_4)$ باستخدام طريقة تكنولوجيا المساحيق حيث $(X=0.00,0.01,0.02,0.03,0.04,0.05)$ عند درجة حرارة التلدين $1100^{\circ}C$ لمدة أربع ساعات. حيث تم اجراء مجموعة من الفحوصات لدراسة الخواص التركيبية وذلك عن طريق قياس حيود الأشعة السينية (XRD) فقد تضمنت دراسة ثابت الشبكة (a) والحجم الحبيبي ($D_{\mu m}$) والمسامية والكثافة الظاهرية بدلالة حيود الأشعة السينية، وكذلك دراسة الخواص العزلية باستخدام جهاز (LCR) اذ تضمنت الدراسة قياس ثابت العزل الكهربائي الحقيقي (ϵ') وثابت العزل الكهربائي الخيالي (ϵ'') ودراسة خسارة العازل ($\tan \delta$) والتوصيلية الكهربائية (σ_{ac}). لقد أظهرت نتائج حيود الأشعة السينية أن جميع النماذج ذات تركيب بلوري متمركز الواجهة (FCC) من خلال الحصول على خواص نظام (FCC) وهي $(\beta=\gamma=\alpha)$ و $(a=b=c)$ وكذلك أظهرت أن ثابت الشبكة (a) يزداد كلما زاد تركيز الكوبالت والكثافة الظاهرية التي تم حسابها عن طريق حيود الأشعة السينية تزداد مع زيادة تركيز الكوبالت أما المسامية والحجم الحبيبي ينخفضان كلما زاد تركيز الكوبالت. كما أظهرت الفحوصات العزلية أن ثابت العزل الكهربائي الحقيقي والخيالي والتوصيلية الكهربائية باستخدام تيار متناوب تنخفض كلما زاد التردد أما تأثير تركيز الكوبالت فإنه يؤدي إلى زيادة ثابت العزل الحقيقي وانخفاض قيم ثابت العزل الخيالي والتوصيلية الكهربائية.