$Zn_xCo_yO_4$ spinel's synthesis and electrical characteristics investigated at various frequencies

Dr. Rasha Hosam Saleh*

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□ ABSTRACT □

$Zn_xCo_yO_4$ spinel samples were made using the thermal ceramic method, with (x = 0.2, 0.4, 0.6, and 0.8) as the molar ratios for samples. The molecular weights of the main compounds were used to determine the necessary weights of each material. The samples were sintered for 30 hours at 450^oC after the doping and heating processes were completed at the selected molar ratios and within the temperature range of 25–450^oC. Within the frequency range of 5Hz-1MHz, the electrical parameters of the resultant samples were diagnosed and quantified. These properties included electrical conductivity σ, electrical capacitance C, relative dielectric constant ε_r, and loss tangent tanδ. The findings demonstrated that, with respect to the variation in the percentage of zinc (Zn) in the samples, the highest electrical conductivity value is $5.50\times10^{+4}$ $(\Omega.cm)^{-1}$, the highest value of C is 84.57 pF, the highest ε_r value is 190.58, and the lowest value of tanδ is $2.34\times10^{+6}$.

Keywords: Spinel structural, Cobalt oxide Co_3O_4, Zinc metal Zn, Electrical conductivity, Electrical capacitance C.

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*Phd ─ Department of Chemistry ─ Faculty of Science ─ Tishreen University ─ Lattakia ─ Syria.

تحضير السباينل $Zn_xCo_yO_4$ ودراسة خصائصه الكهربائية عند ترددات مختلفة

د. رشا حسام صالح *

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□ ملخّص □

تم تحضير عينات السباينل $Zn_xCo_yO_4$ باستخدام الطريقة السيراميكة الحرارية، وفق ($x = 0.2, 0.4, 0.6, 0.8$) كنسب مولية للعينات. تم استخدام الأوزان الجزيئية للمركبات الرئيسية لتحديد الأوزان اللازمة لكل مادة. تم تلبيد العينات لمدة 30 ساعة عند 450 درجة مئوية بعد الانتهاء من عمليتي التشويب والتسخين عند النسب المولية المختارة وضمن نطاق درجة الحرارة 450−25 درجة مئوية. ضمن نطاق التردد من 5 هرتز إلى 1 ميجا هرتز، تم تشخيص وقياس العوامل الكهربائية للعينات الناتجة. شملت هذه الخصائص الناقلية الكهربائية σ، والسعة الكهربائية C، وثابت العزل الكهربائي النسبي ε_r، وظل الفقد $\tan\delta$. أظهرت النتائج أنه فيما يتعلق بالتغير في نسبة الزنك (Zn) في العينات، فإن أعلى قيمة للناقلية الكهربائي هي $5.50\times10^{+4}$ $(\Omega.cm)^{-1}$، وأعلى قيمة لـ C هي 84.57 pF، أعلى قيمة لـ ε_r هي 190.58، وأدنى قيمة $\tan\delta$ هي $2.34\times10^{+6}$.

الكلمات المفتاحية: بنية السباينل، أوكسيد الكوبالت، معدن الزنك، الناقلية الكهربائية، السعة الكهربائية.

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* دكتوراه — قسم الكيمياء— كلية العلوم — جامعة تشرين — اللاذقية — سورية.

Introduction

Mineral compounds known as spinel compounds have the crucial formula AB_2X_4, in which the polyhedrons and tetrahedrons that make up the complex structure are A and B, which are positive cations and are typically metals [1].

The atoms of spinel compounds are arranged regularly within the crystal lattice and they typically have a cubic crystal structure. As a result of this arrangement, the crystalline structure becomes more robust and stable and gains unique characteristics including stability, durability, and resistance to compression and expansion. Because of their great endurance and low energy absorption in the optical vacuum region, these compounds find extensive use in various technical and industrial applications. Since spinel compounds have regular crystalline structures that hinder the flow of electrons and, consequently, electric current, many of them also have good magnetic and electrical conductivity qualities [2].

Co_3O_4, or cobalt oxide, is a significant chemical compound. It is a powder that is black in color, exhibits good chemical stability, and may catalyze a variety of chemical reactions. Because of its superior electrochemical qualities, it is utilized in energy-related applications as solar cells, batteries, and capacitors [3]. Cobalt is present in the cobalt oxide compound at two different oxidation degrees: binary (Co^{2+}) and triple (Co^{3+}). Cobalt oxide is composed of cobalt binary oxide (CoO) and triple cobalt oxide (Co_2O_3). Because of its spinel structure, cubic crystal system crystallization, and vacuum group Fd-3m crystallization, it is known as mixed cobalt oxide [4].

One significant chemical element is zinc. It has a gray to silver hue. The hexagonal system is where it crystallizes. Among the transition metals is this one. It has several characteristics, including electrical and thermal conductivity. It is utilized in numerous technical applications, including batteries—of which the zinc-carbon battery is the most well-known example. Another name for them is dry cells [5].

Because of their exceptional qualities, spinel compounds comprising zinc Zn and cobalt oxide Co_3O_4 are widely used in numerous industrial and technical applications [6]. It can be utilized as thermal insulators and in thermally inert tools, such as fire-resistant materials, due to its magnetic qualities and high heat resistance [7]. Because of their unique electrical characteristics, they are also utilized in battery and capacitor applications, magnetic storage, and magnetic sensor applications [8].

Importance and Objectives of the Study

Because $Zn_xCo_yO_4$ spinel has so many potential uses in a variety of fields and fields of science, there is a lot of interest in studying its electrical properties. This is because of these materials' chemical makeup as well as their technological and electrical characteristics, which could result in advancements in energy transmission and storage.

Materials and Methods

1. Materials

- Cobalt oxide (Co_3O_4) with a purity of 99%.
- Zinc metal Zn, with a purity of 99%.

2. Sample Process

The spinel formula $Zn_xCo_yO_4$ is used to prepare the samples for the following molar percentages (x = 0.2, 0.4, 0.6, and 0.8) mol%. A sensitive balance of 0.0001gr was used to weigh the materials, and the required weights were determined from the initial based on the molecular weight, as indicated in Table (1):

Table 1: Weight of raw material calculated as a function of ratio x

n	unit wt	
	x	**y**
1	0.2	2.8
2	0.4	2.6
3	0.6	2.4
4	0.8	2.2

Subsequently, each sample was thoroughly mixed and ground for five hours in a mortar. A manual hydraulic press operating at 3 tons per square centimeter was utilized to put the final samples into tablet form.
In the first stage, the samples were heated in porcelain crucibles from room temperature to 150^oC, and they were then calcined for six hours at this temperature. The tablets were then allowed to cool down gradually in the lab. Next, the tablets were well ground, and the heating process was repeated until the temperature reached 250^oC. This process was then repeated until the temperature reached 450^oC, at which point the samples were sintered for 30 hours.
An LCR device was used to study the electrical properties of the manufactured samples within the frequency range of 5Hz-1MHz, while a digital biopsy was used to measure the discs' thickness and diameter.

3. Study Samples

A PM 6306 LCR meter was used to analyze the prepared samples; the condenser tip's diameter was 1 cm.

Results and Discussion

1. The impact of molar percentage on spinel's electrical conductivity (σ)

Using the equation $\sigma=1/\rho$, where σ is electrical conductivity and ρ is electrical resistivity, the variations in electrical conductivity $(\Omega.cm)^{-1}$ for synthesized samples in accordance with the $Zn_xCo_yO_4$ spinel formula within the frequency range (5Hz-1MHz) were investigated. The diameter of the capacitor pin is 1cm. High caliber. The equation below provides the particular electrical resistivity value:
$\rho=R\,A/d$, where d is the disc thickness (cm), A is the disc surface area (cm^2), and R is the electrical resistivity (Ω) [9].
As the amount of zinc in the samples increased, the electrical conductivity values increased, according to the data. This is due to the electrical conductivity properties of metallic zinc, which affected the properties of mixed cobalt oxide and led to an increase in electrical conductivity, as the entry of zinc atoms into the specific structure led to a reduction in vacuum. Located between the conductivity sector and the valence sector, there is less vacuum obstruction to the movement of electrons that enter the structure, which in turn leads to increased passage of electrical current and increased electrical conductivity values, as shown in (Fig. 1).
The obtained samples are classed as semiconductors based on the electrical conductivity values, with the greatest value being $5.50\times10^{+4}\,(\Omega.cm)^{-1}$ at the molar percentage (x=0.8).

The electrical conductivity's lowest value, which occurs at a ratio of x = 0.2, shows that there is a larger energy band at this ratio between the two compounds' conductivity and valence sectors.

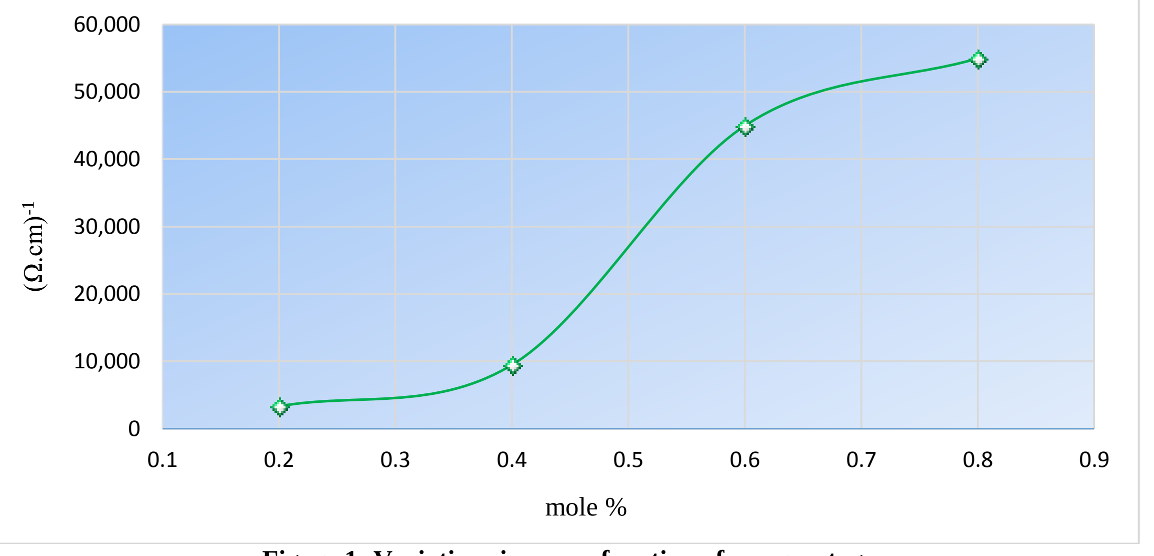

Figure 1: Variations in σ as a function of x percentage

2. The impact of frequency f on the spinel's electrical capacitance C

Within the frequency range of 5Hz-1MHz, the electrical capacitance changes C of the spinel samples generated in Pico farad pF unit using the formula $Zn_xCo_yO_4$ were examined. The findings demonstrated that, in general, electrical capacitance values fall as frequency increases and that, in the manufactured samples, and these values increase as doping % increases. Which indicates that increasing the molar percentage of zinc contributed to increasing the ability to retain an electrical charge, which led to an increase in electrical capacitance values due to the entry of zinc atoms into the spinel structure of cobalt oxide.

The electrical capacitance values at these molar percentages ranged between 84.57 and 6.51 pF, with the sample with the highest content of zinc having the biggest capacitance values at the molar percentage (x = 0.8%).

The variations in the electrical capacitance values C of the synthesized samples, at molar ratios (x = 0.2, 0.4, 0.6, and 0.8) mol%, are displayed in (Fig. 2).

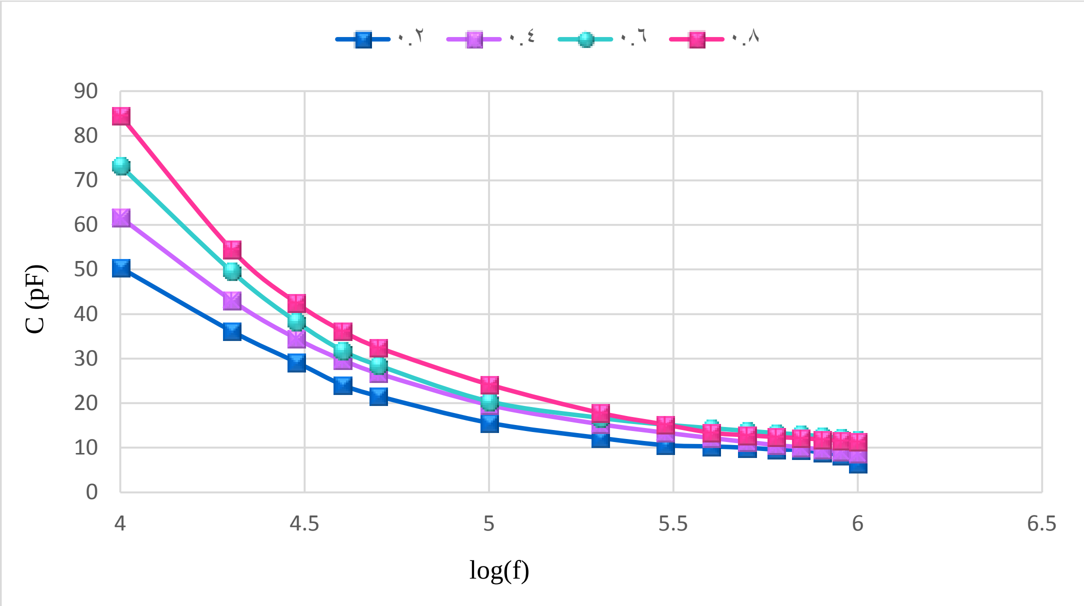

Figure 2: Variations in C as a function of frequency f

3. The impact of frequency f on the spinel's Relative dielectric constant ε_r

The electrical capacitance of the solid disk is calculated according to the equation:

$$C = \varepsilon_0 \varepsilon_r \frac{A}{d} \qquad (1)$$

Therefore, when the capacitance is given in pF and the geometric dimensions of the disk are given in cm, the relative dielectric constant is obtained from the equation:

$$\varepsilon_r = 11.3 \times 10^{12}\ C\ \frac{d}{A} \qquad (2)$$

The $Zn_xCo_yO_4$ spinel samples' results demonstrated that, generally speaking, the relative dielectric constant values decrease with increasing frequency and also decrease with an increase in the prepared samples' doping percentage. The samples with the highest dielectric constant values are those with increasing cobalt oxide concentrations, which is consistent with the results of electrical conductivity, due to its cubic structure and semiconductive properties in which the resistance to the movement of electrons varies according to the octahedrons and tetrahedrons that are present in the crystal structure, which decreases with increasing percentage of doping with metallic zinc.

The values of the relative dielectric constant at these molar percentages ranged within the range (14.68 - 190.58), with the largest value being for the sample containing the highest concentration of cobalt oxide, at the molar percentage (x = 0.2).

The variations in the relative dielectric constant values ε_r of the synthesized samples, are displayed in (Fig. 3).

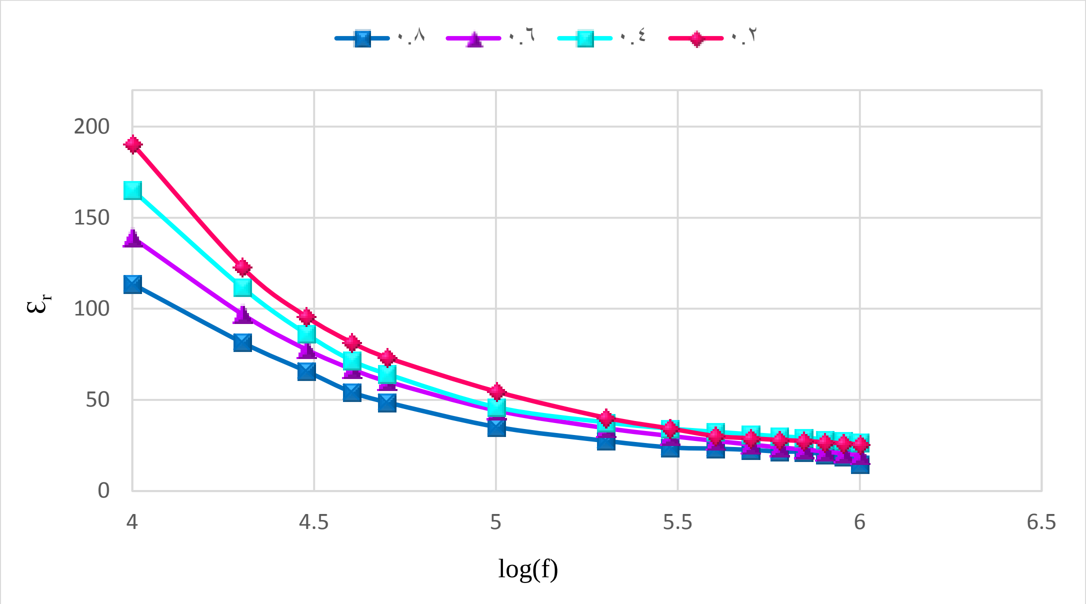

Figure 3: Variations in ε_r as a function of frequency f

4. The impact of frequency f on the spinel's loss tangent tanδ

Equation $\tan\delta = 1/(\rho.\omega.\varepsilon_0.\varepsilon_r)$ is clarifying the variations in the $Zn_xCo_yO_4$ spinel's loss tangent tanδ as a function of frequency f were examined. Where, $\omega = 2\pi f$, f is the frequency (Hz) and ω is the angular frequency (rad/sec).

The amount of energy lost in the insulating material is expressed by the value of the loss tangent, or tanδ. When the values are high, the insulating material's wasted energy is high, indicating that it is not a good insulator; conversely, when the values of the tangent of the loss angle are modest, the insulating material is a good insulator.

The findings demonstrated that when the proportion of zinc doping increases, the tangent values of the loss tangent increase. This shows that the samples with higher cobalt oxide concentrations are more suitable for electrical insulation, as evidenced by the higher loss coefficient values compared to the other samples. This is congruent with the electrical conductivity values in the samples: as the amount of doping in the spinel structure grows, the value of the loss coefficient increase, this result is consistent with the electrical conductivity values in the samples.

The samples' value of the loss tangent varied between $2.34\times10^{+6}$ and $8.70\times10^{+8}$, with the sample with the highest concentration of cobalt oxide at the molar percentage (x = 0.2) having the lowest value, as shown in (Fig. 4).

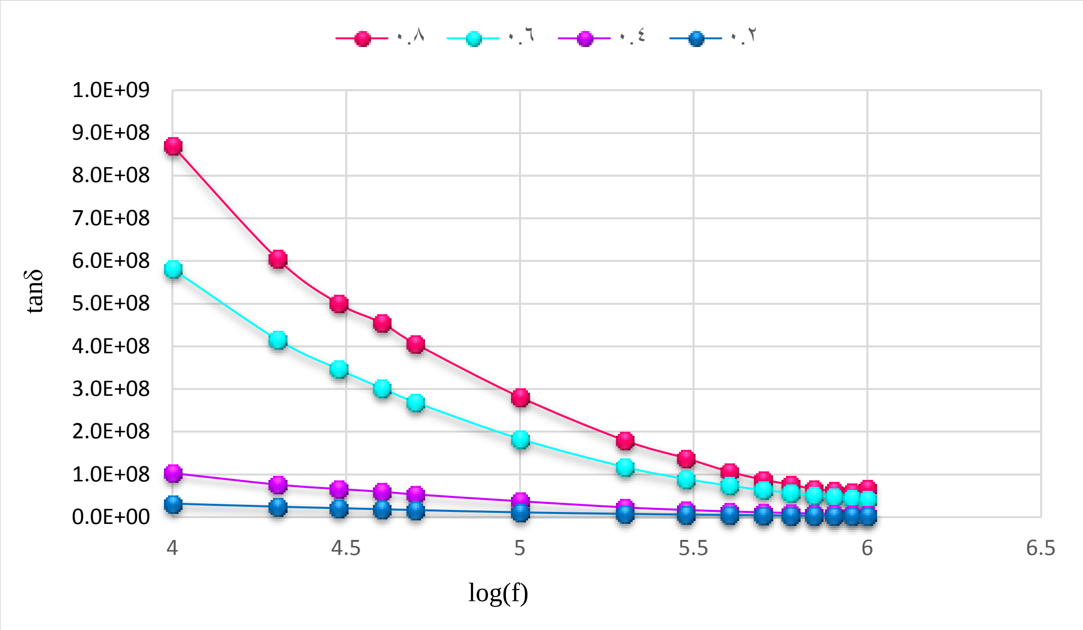

Figure 4: Variations in tanδ as a function of frequency f

Conclusions and recommendations

Conclusions

- The electrical conductivity levels rise, as the percentage of zinc in the samples increase
- at the molar percentage (x=0.8), the maximum electrical conductivity value is $5.50\times10^{+4}$ $(\Omega.cm)^{-1}$.
- As the percentage of doping in the prepared samples increases, the electrical capacitance values also increase.
- The electrical capacitance with the greatest value is 84.57 at molar percentage (x=0.8).
- As the percentage of doping in the samples increases, the values of the relative dielectric constant decrease.
- At the molar proportion (x=0.2), the relative dielectric constant has its highest value of 190.58.
- As the percentage of zinc doping increase, the loss tangent values also increase.
- At the molar percentage (x =0.2), the loss tangent has a minimum value of $2.34\times10^{+6}$.

Recommendations

using the created spinel's molar percentages in batteries and solar energy cells to develop and boost their capacity because of the electrical values and qualities attained.

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