

دراسة خواص اوكسيد الحديد النانوي المعوض بأيونات النحاس واليثيريوم

والمحضر بطريقة الاحتراق الذاتي للسائل الهلامي

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الملخص

في هذا العمل تم أستعمال طريقة المحلول الهلامي (sol gel) في تحضير اوكسيد الحديد النانوي المطعم بالنحاس والليثيوم والذي يحمل الصيغة الكيميائية $\text{Li}_{1-x}\text{Cu}_x\text{Fe}_5\text{O}_8$ حيث ان x هي نسبة الاضافة المولية ($x = 0.0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6$) ومن خلال الاحتراق الذاتي للجل المائي والذي حصل عند درجة حرارة 200°C ، ثم بعد ذلك قسمت العينات الى اربع مجاميع، المجموعة الاولى وهي التي لم يتم تليدها بينما المجاميع الاخرى؛ الثانية والثالثة والرابعة فقد تم تليدها بدرجة حرارة 500°C ، 700°C و 900°C على التوالي.

في هذا العمل تم دراسة البناء البلوري والاطوار البلورية السائدة بواسطة فحص حيود الاشعة السينية وكذلك باستعمل فحص المجهر الالكتروني و فحص الاشعة تحت الحمراء بواسطة موديل فورير الرياضي اما هيئة السطح وتضاريس السطح فقد تم دراستها باستعمال مجهر القوة الذرية، فحص الخواص السعوية بواسطة جهاز قياس السعة، المقاومة والمحث. وبصورة غير مباشرة ومن خلال تلك الفحوصات تم معرفة حجم الوحدة التركيبية للبلورة والكثافة النظرية وثابت الشبكة وكذلك حجم الحبيبة قد تم قياسها بواسطة فحص حيود الاشعة السينية بينما تشكل الكتل الماكروية المجهرية قد تم فحصه بواسطة المجهر مسح الالكتروني الماسح اما قياس حجم الحبيبات على سطح العينة ومعامل خشونتها قد تم قياسه بواسطة مجهر القوة الذرية ونوع الاواصر التي تربط الذرات في البلورة ونوع الاطوار البلورية قد تم فحصه بواسطة الاشعة تحت الحمراء في حين قياس الخواص السعوية وقابلية التوصيل للتيار الكهربائي المتناوب قد تم بواسطة فحص الخواص السعوية.



الدراسة الحالية تركز على قياس الخواص السعوية والتي تاخذ في حسابها تأثير تغيير محتوى النحاس في العينات ودرجة حرارة التلبيد بالاضافة الى تغيير التردد المطبق على العينات من مصدر الجهد الخارجي وكذلك خسارة الطاقة الكهربائية عند مرور التيار الكهربائي في العينات.
الكلمات الدالة : اوكسيد الحديد النانوي المطعم بالنحاس و الليثيوم ، المحلول الهلامي، الاحتراق الذاتي ، الخواص السعوية .

Synthesis and Characterization of Copper Substituted Lithium Nano Ferrites by Sol-Gel Auto combustion

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Abstract:

In this work sol-gel method used for preparing nanoscale iron oxide ferrites dopant by copper and lithium, which carries the chemical formula $Li_{1-x}Cu_xFe_5O_8$ where x is the added ratio as molar ratio ($x = 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6$ and 1), during the self- combustion for sol-gel which happen at temperature of $200^{\circ}C$, after that the samples were divided into four groups , the first group namely that have not been annealed while the other groups ; the second, third and fourth were annealed at $500^{\circ}C$, $700^{\circ}C$ and $900^{\circ}C$ respectively.

In this work the crystalline structure have a dominate phases which have been studied by using the X-ray diffraction as well as





examination of the electron microscope and examine the infrared - mediated model of Fourier Transform Infrared Spectrometer either the topography of the surface has been studied by using an atomic force microscope, examine the properties of electrical capacitive mediated capacitance measuring device, resistance and the inductor also studied. And indirectly through these tests was to know the size of the structural unit of the crystal density theory and fixed lattice, as well as the size of the grain has been measured by X-ray diffraction, while a microscopic blocs have checked mediated electron microscopy scan scanner, either measuring the size of the granules on the surface of the sample and the coefficient of roughness has been measured mediated by an atomic force microscope and the type of bonds that bind atoms in the crystal and the type of crystalline eccentric may checked mediated infrared while measuring the properties of capacitive and portability electrical conductivity of the alternating current it may have been mediated by checking capacitive properties.

he current study focuses on measuring the properties of capacitive, which takes into account the effect of changing the copper content in the samples and the degree of sintering temperature as well as the change altered applied to the samples from the external voltage source, as well as the loss of electric power when the power is in the passage of the samples

Keywords: Copper Lithium Nano Ferrites; Sol-Gel , Auto Combustion Technique ; capacitive properties.





1.Introduction:

Nano crystalline spinel ferrites have been investigated in the recent years by many researchers. Ferrite is a polycrystalline, magnetic components, they have low magnetic losses properties therefore its an excellent soft magnetic spinel ferrites, low dielectric losses, high mechanical hardness, high Curie temperature and high chemical stability. They play a useful role in many technological applications such as the electronic and telecommunication industries and because of their high initial permeability the nano ferrites are used in high density information storage devices. Depending on type of applications, magnetic ferrite nano particles are used in varieties of forms such as surface functionalized particles in biomedical applications, particles arrays in magnetic storage media and compacted powders in permanent magnets ferrite can be used at considerably high frequencies sintered material with high electrical





resistively. The high resistance of ferrite resistivity, makes eddy current losses extremely low at high frequencies [1].

The spinel ferrite structure with the chemical formula KFe_5O_8 , where K refers to the monovalent metal, can be described as a cubic close-packed arrangement of oxygen atoms, with K^{1+} and Fe^{3+} at two different crystallographic sites. These sites have tetrahedral and octahedral oxygen coordination (termed as A and B-sites, respectively), so the resulting local symmetries of both sites are different. The spinel structure contains two cation sites for metal cation occupancy. There are 8 of A-sites in which the metal cations are tetrahedral coordinated with oxygen, and 16 B-sites which possess octahedral coordination. When the A-sites are occupied by K^{1+} cations and the B-sites are occupied by Fe^{3+} cations, the ferrite is called a normal spinel. If the A-sites are completely occupied by Fe^{3+} cations and the B-sites are randomly occupied by K^{1+} and Fe^{3+} cations, the structure is referred to as an inverse spinel. In most spinels, the cation distribution possesses an intermediate degree of inversion where both sites contain a fraction of the K^{1+} and Fe^{3+} cations. Magnetically, spinel ferrites display ferrimagnetic ordering. The magnetic moments of cations in the A and B-sites are aligned parallel with respect to one another. Between the A and B-sites the arrangement is antiparallel and as there are twice as many B-sites as A-sites, there is a net moment of spins yielding ferrimagnetic ordering for the crystal. The choice of metal



cation and the distribution of ions between the A and B-sites therefore, offer a tunable magnetic system [2].

2.The Raw Materials:

The $\text{Li}_{(1-x)}\text{Cu}_x\text{Fe}_5\text{O}_8$ Nano ferrite samples were manufactured with a raw materials, the selected materials were very pure as mentioned in the table (1):

Table (1) : The Nano Ferrite Samples Raw Materials

Compounds	Chemical formula	Molecular Weight	% Purity	Supplier
Iron (III) nitrate	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	404	99	England
Cupper nitrate	$\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	241.63	≥ 97	Saudi Arabia
Lithium nitrate	$\text{Li}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	102.94110	≥ 99	Saudi Arabia
Ammonia solution	NH_3	17	33	England
Citric Acid	$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$	210.14	≥ 99	Germany
Distilled Water	H_2O	18		

3. Experimental Procedure:

The raw materials weighted according to the mixing ratio, beside it was taken of 50ml from distilled water as a solvent which separated into two half, first 25ml of distilled water was used to dissolving citric acid with continuous stirring at room temperature, the second half 25ml used to dissolving (Iron nitrate, copper nitrate and lithium nitrate) with



continuous stirring until to get homogenous solution , iron nitrite dissolved with molar ratio 0.2 mol/L and citric acid dissolved with 0.4 mol/L, the copper nitrate and lithium nitrate added with the ratio needed to complete the chemical composition, after that the solution of the citric acid was added to the metal nitrate solution , the changing in color notified , the NH_3 solution was added drop by drop to the final solution until the titration point was reached when Ph meter read 7 , the operation was done with continuous stirring at room temperature.

the heat temperature was raised after that and kept at 90°C , with continuous stirring to get the gel, when the magnetic bar stopped from movement that means the solution reached to the gel point due to the high viscosity of the sol-gel, the gel was removed from the hot plate, and inserted into hot oven at 120°C until the dry gel occur, the Nano ferrite was formed at 200°C .

The Nano ferrite samples were manufactured within the past listed raw materials in special procedure and conditions , the procedure were repeated to manufacture (23) Nano ferrite samples arranged in four groups ,the first group has the samples with copper molar ratio $x=(0,0.1,0.2,0.3,0.4,0.5,0.6)$, used fresh without any calcination temperature ,the second group has the samples copper molar ratio $x=(0,0.1,0.2,0.3,0.4,0.5,0.6)$ and the calcination temperature was 500°C , the third group has the samples with $x=(0,0.1,0.2,0.3,0.4,0.5,0.6)$ and the calcination temperature was 700°C and the fourth group has the samples





with $x = (0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6)$ and the calcination temperature was 900°C , all of the Nano ferrite samples were cooled to room temperature, the first and second group were directly prepared to the structural tests, the third and fourth groups were prepared for the electrical properties test after cooling to the room temperature, they were compacted in hydraulic press at 3 ton/cm^2 for a compression period about a minute, then they were sintered at 1000°C for an hour then they were cooled again to the room temperature, figure (1) clarify the raw materials & the detailed procedure.





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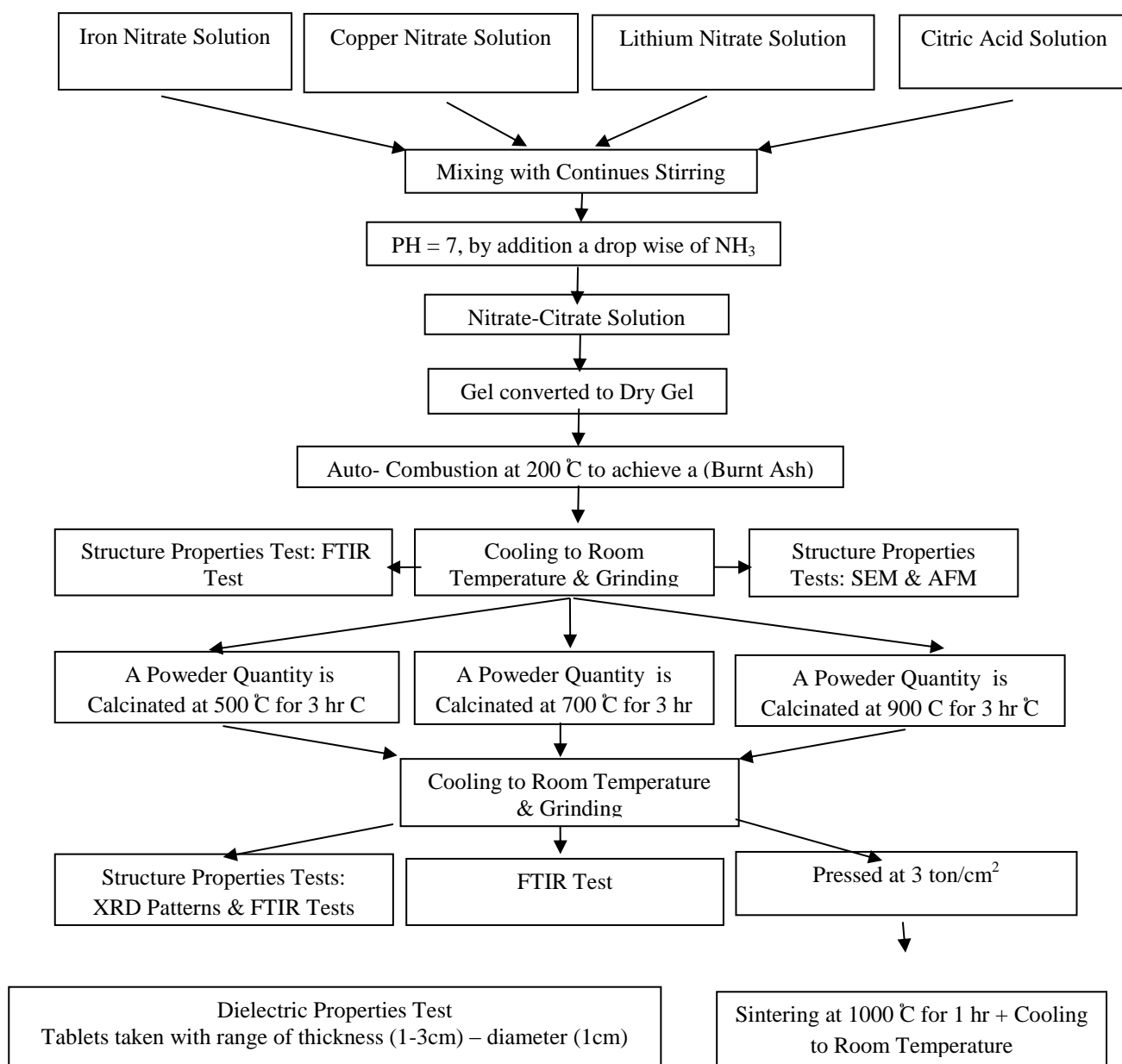
Figure (1): Experimental Procedure Details



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4.1 Dielectric Constant

In the Nano ferrite sample series, the variation of the dielectric constant of the composite with frequency is calculated in equation (1):

$$\varepsilon'r = C * d/A * C_o.....(1)$$

Where: $\varepsilon'r$ is the dielectric constant which is dimensionless, A is the cross sectional area of the Nano ferrite tablet in M^2 , d is the thickness of the tablet in M , C is the capacitance of the tablet in farad and C_o is the capacitance at the air as insulator medium in farad .The dielectric constant is the measure factor to calculate the imaginary dielectric constant and A.C. conductivity.

The capacitance of the tablets were measured by the LCR meter, recorded and used in the equation (1) to calculate the value of the dielectric constant, the dielectric constant is displayed within the curves as shown in figure (2) and figure (3). The real dielectric constant was notified as a continuous declining with increasing the frequency of the external applied source voltage. At fixed frequency the real dielectric constant of the calcined samples at $700^\circ C$ was higher than that calcined at $900^\circ C$, at high frequency values the convergences in the dielectric constant curves are notified to be at in a fixed dielectric constant value indicating that the charge storage capacity of these samples remained insensitive to the variations in the extended field at high frequencies. The changes in dielectrics were attributed to the tweaking in the polarizability of the copper-lithium ferrite as calcination temperature is increased, the baseline of the real dielectric curves tended to shift to higher values. It was observed that the dielectric constant is decreased with the increasing of copper content in ferrites; the variation in dielectric constant may be



explained on (the basis of space charge polarization). According to the Maxwell and Wagner two-layer model [3]. the Nano ferrite tablets act as multilayer capacitor, the space charge polarization is produced in a dielectric material due to the presence of higher and poorer electrical conductivity phases where they're the structure of the dielectric material, the grains of the Nano ferrite samples are the higher electrical conductivity phases and they're acted as the capacitor conductive slabs. When an external electric field is applied [4]. The electrons reach the grain boundary through hopping of the E.M.F. The value of (ϵ_r') is much higher at lower frequencies and decreased with the increasing frequency; the decreasing in dielectric constant is maintained rapidly from 25 KHz up to 1000 KHz, followed by slow decreasing up to 2 MHz, at very high frequencies, its value is so small that it becomes independent of frequency as prescribed. The assembly of space charge carrier's positions in a dielectric material takes a finite time to line up their axes parallel to the alternating external electric field. If the frequency of the external electrical field is increased, a point is reached where the space charge carriers cannot keep up with the field and the alternation of their direction lags behind that of the field. The reduction of the capacitance is resulted and the dielectric constant reduction is resulted of the material. The electron exchange between $Fe^{2+} \leftrightarrow Fe^{3+}$ results the local displacement of electrons in the direction of applied field that determines the polarization. The polarization is decreased with the increasing in value of frequency



and then reaches a constant value. It is due to the fact that beyond a certain frequency of external field, the electrons exchange $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ cannot follow the alternating field so the polarizability is become independent on the frequency [5].

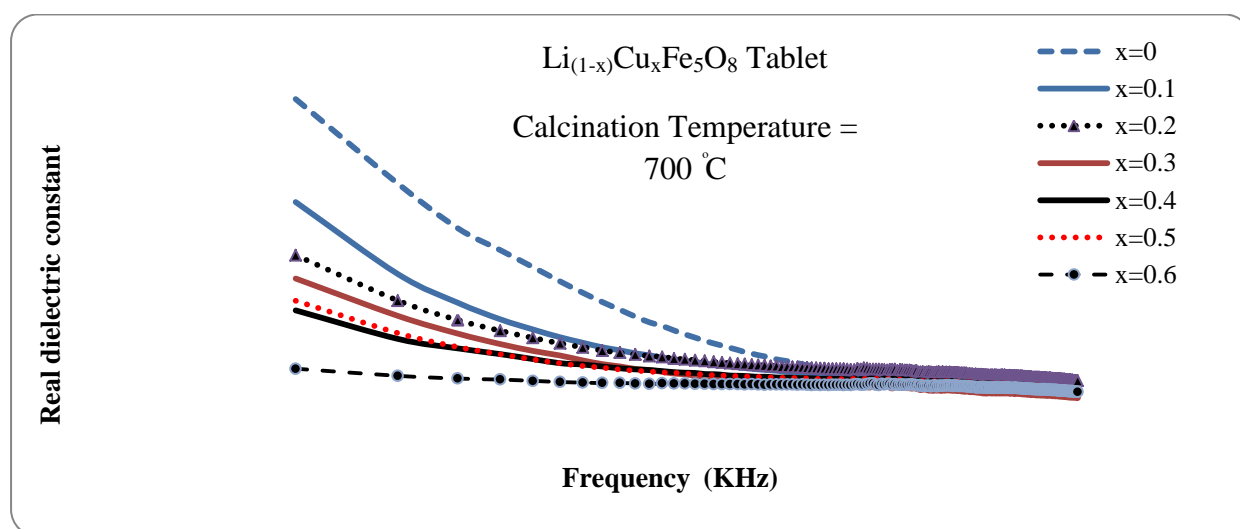


Figure (2): The Dielectric Constant of $\text{Li}_{(1-x)}\text{Cu}_x\text{Fe}_5\text{O}_8$ Tablet
Calcination at 700 °C as a Function of Frequency

The Dielectric constant (ϵ_r') has a large value at the lower frequency. That happen due to the predominance of species like Fe^{2+} ions, the grain boundary defects and the interfacial voids. The decreasing of dielectric constant in presence of ferric ions Fe^{3+} , creates lower hopping sites [6]. Therefore the electron pile-up at the grain boundaries tends to decrease. In addition, the Fe^{3+} ions are stated to be less polarizability than the Fe^{2+} ions. These effects are the main causes which shift the real dielectric constant curves to lower values. it was found that,

the composite samples with $x=0.0$ shows the higher ϵ_r' which was equal 21.512198 when the tablet calcination temperature was 700°C and equal to 19.16497 when the tablet calcination temperature was 900°C .

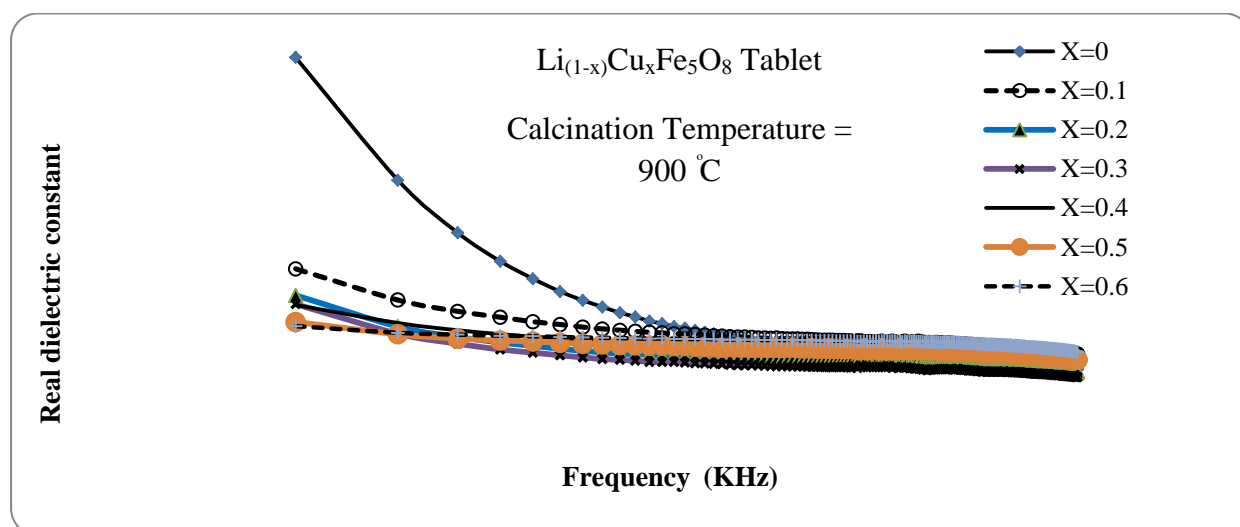


Figure (3): The Dielectric Constant of $\text{Li}_{(1-x)}\text{Cu}_x\text{Fe}_5\text{O}_8$ Tablet Calcinated at 900°C as a Function of Frequency

4.2 Dielectric Loss Factor

The data on the dielectric loss factor ($\tan \delta$) relates between the real and imaginary dielectric constants, it's the tangent of the phase lag angle between the two types of the dielectric constants and mainly used in A.C. power and in the telecommunications, the loss factor is an important role to determine the losses made within the flow of A.C. current in which they turn to heat by creating the eddy currents inside the conductive medium , it was studied by the effects of calcination



conditions, E.M.F frequency and Cu additions on the lossy characteristics of the Cu-Li ferrite samples[7]. The values of dielectric loss factors were obtained directly by the LCR meter in the test .Figures (4) and (5) give clear behavior about how the loss factor affected by the variation of copper content and the calcination temperature .It's found that the dielectric loss tangent is decreased with the increasing in the applied frequency. This behavior was similar to that of the normal dielectric behavior of all spinel ferrite samples. This behavior of $\tan\delta$ may be explained qualitatively by considering that the mechanism of polarization process in ferrite is similar to that the conduction process. in Cu-Li ferrites. The initial decrease of dielectric loss with an increase in frequency can be explained on the basis of Koops' model . In the low frequency region which corresponds to high resistivity (due to grain boundaries) more energy is required for electron exchange between Fe^{2+} and Fe^{3+} ions. Thus the energy loss is high. In the high frequency range which corresponds to low resistivity (due to the grains) a small energy is needed for electron transfer between Fe^{2+} and Fe^{3+} ions in the grains and hence the energy loss is small . The electron hopping occurs by electron transfer between adjacent octahedral sites (B-site) in the sub-lattice. Thus by electronic exchange ($\text{Fe}^{2+} + \text{Cu}^{2+} \Leftrightarrow \text{Fe}^{3+} + \text{Cu}^{+}$) one obtained local displacements of electrons in the direction of the applied electric field, these displacement determine the polarization of the ferrite[8].



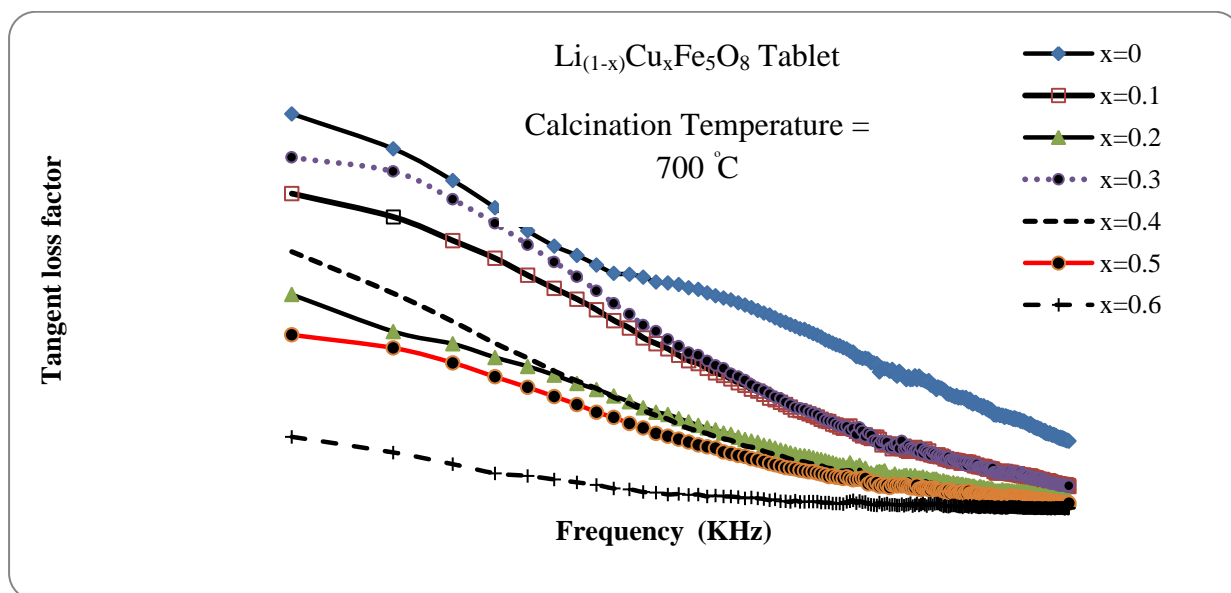


Figure (4): The Tangent Loss of The $\text{Li}_{(1-x)}\text{Cu}_x\text{Fe}_5\text{O}_8$ Tablet Calcinated at 700°C as a Function of Frequency

The effect of polarization is to reduce the field inside the medium. it was found that, the composite samples with $x=0.0$ shows a rapid decreasing in $\tan \delta$ (the electrical loss factor) which was equal to 1.253974 when the tablet calcination temperature was 700°C and equal to 1.205202 when the tablet calcination temperature was 900°C when the maximum $\tan \delta$ is observed when the jumping or hoping frequency of electrons between Fe^{2+} and Fe^{3+} becomes approximately equal to the frequency of the applied field, so at when the tablet calcination temperature was 700°C the tablet is less conductive then the calcined tablet at 900°C .

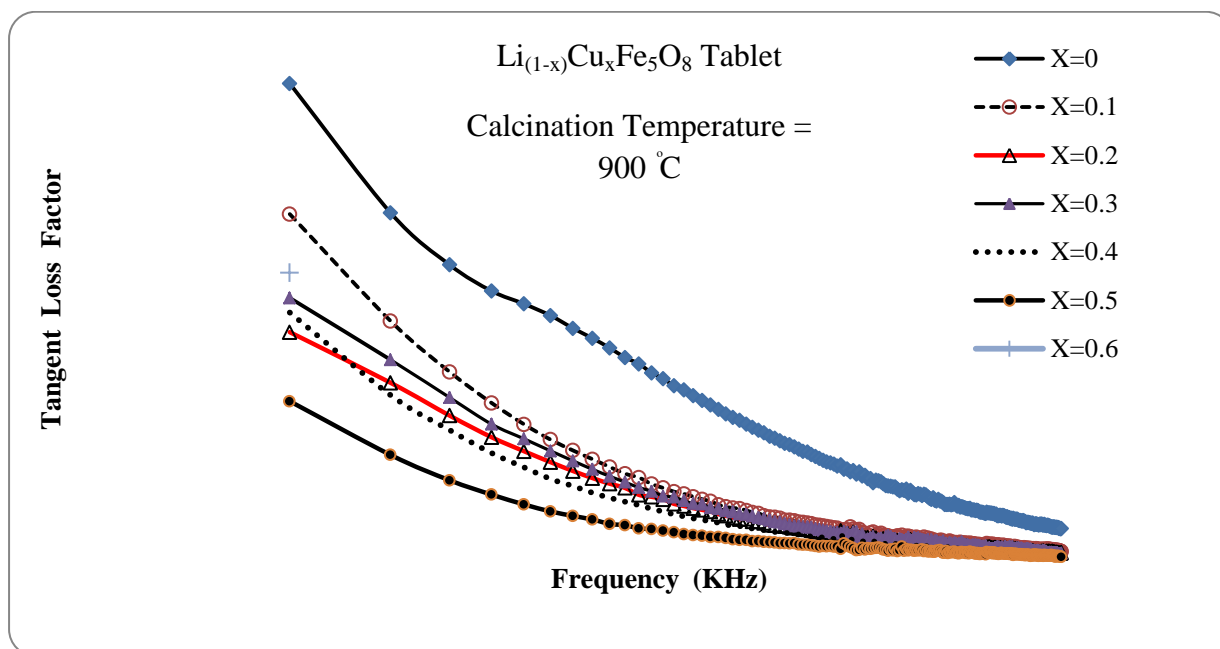


Figure (5): The Tangent Loss of Li_(1-x)Cu_xFe₅O₈ Tablet Calcinated at 900 °C as a Function of Frequency

4.3 Imaginary Dielectric Constant

It is clear that all the ferrite composite samples under study exhibit the imaginary dielectric constant (ϵ''_r) decrease as the frequency increases up to 5 MHz. Figures (6) and (7) shows clarify the behavior of the imaginary dielectric constant as a function of the frequency in which they were obtained by equation (2)

$$\epsilon''_r = \tan\delta * \epsilon'_r \dots\dots\dots(2)$$

Where ϵ''_r is the imaginary dielectric constant, $\tan\delta$ is the tangent loss factor and ϵ'_r is real dielectric constant, the equation elements are dimensionless, it's effected by the variation of copper content ratio in the nano ferrite sample and the calcination temperatures. The imaginary dielectric constant decreases rapidly in lower frequency region from 25 KHz up to 1000 KHz, the behavior almost approaches to frequency independent behavior.

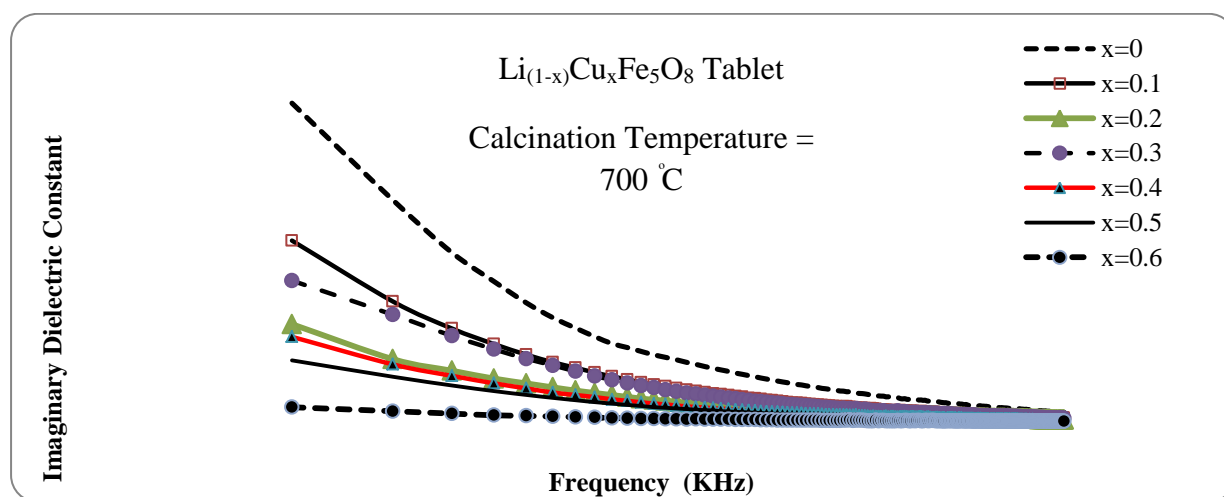


Figure. (6): Imaginary Dielectric Constant of Li_(1-x)Cu_xFe₅O₈ Tablet
Calcinated at 700 °C as a Function of Frequency

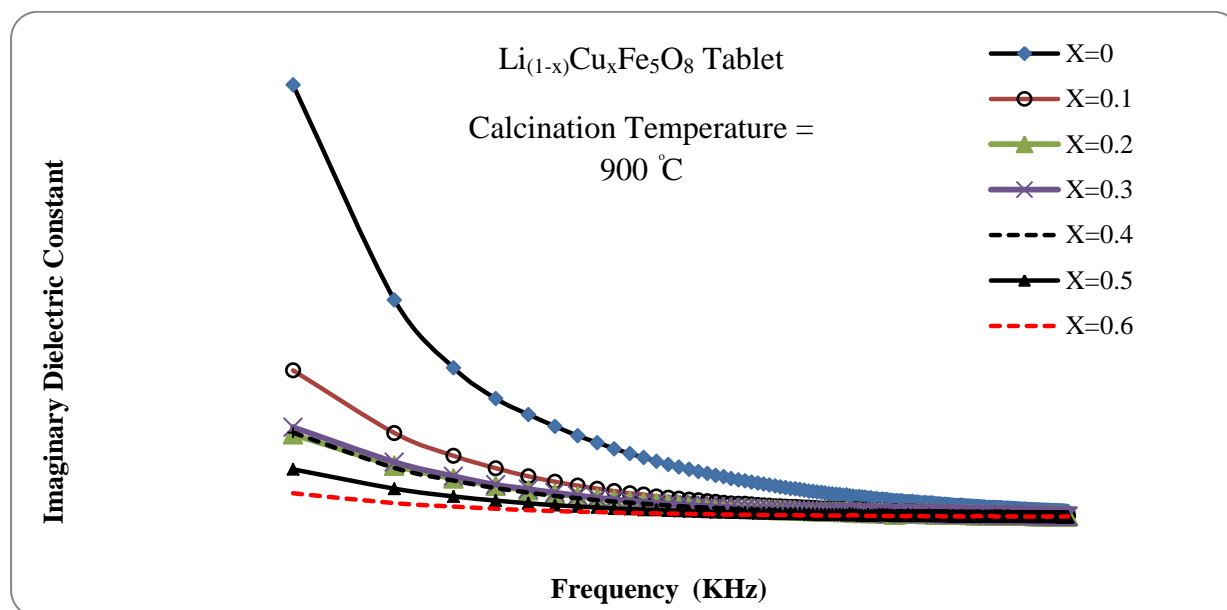


Figure (7): Imaginary Dielectric Constant of Li_(1-x)Cu_xFe₅O₈ Tablet

Calcinated at 900 °C as a Function of Frequency

The imaginary dielectric constant (ϵ''_r) is found to decrease faster than (ϵ'_r) in the lower frequency region, while the variation in (ϵ''_r) is the same as (ϵ'_r) in higher frequency region. it was found that, the composite samples with $x=0.0$ shows the higher ϵ''_r which was equal 22.71200132 when the tablet calcination temperature was 700 °C and equal to 18.47125 when the tablet calcination temperature was 900°C.

4.4 A.C. Conductivity

The A.C. conductivity (σ_{AC}) test detects the ability of the conductor to transfer the eddy currents in which they cause electrical energy losses , the more A.C. conductivity the more electrical losses and

the material is detected as insulator, the variation of the A.C. conductivity (σ_{AC}) with the applied frequency that ranges from 25 KHz up to 5 MHz was studied at room temperature for the all composite sintered samples which was contained ferrite nanoparticles ($\text{Li}_{1-x}\text{Cu}_x\text{Fe}_5\text{O}_8$), the test was guided by using the equation (3)

$$\sigma_{AC} = 2 * \pi * f * \epsilon''r \dots\dots\dots(3)$$

Where σ_{AC} is the A.C. electrical conductivity in $1/\Omega.\text{cm}$, f is the frequency of the applied voltage ranged from 20 KHz to 5 MHz, $\epsilon''r$ is the imaginary dielectric constant which is dimensionless. Figure (8) and figure (9) display the A.C. conductivity (σ_{AC}) for the given samples which show a continuous increasing value with the increasing of the applied frequency. Compositional dependence of A.C. electrical conductivity for Li-Cu ferrite compositions were studied at different frequencies and also for different calcinated temperatures. All samples exhibit normal behavior with the variation of the applied frequency. It is clear that the high value of the AC conductivity is obtained for the samples for the LiFe_5O_8 tablet in which was calcined at 700°C . This indicates that the AC conductivity depends strongly on the increasing in the Li^{+1} ions in the samples; the decreasing in A.C. electrical conductivity when the copper is added may be related to presence of Cu^{1+} , when copper is added and ferrous ions content as in the original LiFe_5O_8 sample in which were produced during sintering and prefers the occupation of tetrahedral sites (A) and Li ions prefers the occupation of



octahedral sites (B), while Fe-ions partially occupies the A and B sites. On increasing Cu^{1+} concentration at A-sites, Li ions concentration at B sites will decrease. This leads to migration of some Fe ions from A site to B site to balance the reduction in Li ions concentration at B sites. As a result the number of ferric and ferrous ions at B sites which are responsible for electrical conductivity in ferrites and increase consequently so the resistivity and decreases by increasing Cu^{1+} ion concentration. The A.C. electrical conductivity is decreased by the increasing of the calcination temperature because of decreasing the two main dependent factors in which they are the tangent loss factor and real dielectric constant values by increasing the calcination temperature, therefore; The conductivity changed from insulator to semiconductor behavior with increasing the calcination temperature[9]. The variation in resistivity can be explained as a consequence of microstructural and structural shifts resulting from changes in calcination conditions. Decreasing in porosity causes individual grains to come closer, while the increased average grain size reduces the grain boundary. These two characteristics combined increase the cross-sectional area of the conductor, thus causing a decrease in the total resistance of the sample. Additionally, the raise in calcination temperatures increases Fe^{3+} concentrations, allowing hopping-type conduction between Fe^{3+} and Fe^{2+} . The electric conductivity in ferrites can be explained by the Verwey-de Boer mechanism [10] in which electron exchanges takes





place between ions of the same element present in more than one valence state. Such ions are distributed randomly over crystallographically equivalent lattice sites and, in Li-Cu ferrites, the electronic hopping between Fe^{2+} and Fe^{3+} ions located on octahedral sites is the primary mechanism which allows electrical transport [11], in further comparison, it was found that the composite samples with $x=0.0$ shows a rapid increasing in σ_{AC} which was equal 0.000234 moh/cm when the tablet calcination temperature was 700°C and equal to 0.000116191 moh/cm when the tablet calcination temperature was 900°C . This is due to the existence of a maximum value of the divalent iron Fe^{3+} ions and Cu ions mixed together among all the mixed Li-Cu spinel ferrite in the composite samples. At high frequencies i.e. 1000 KHz to 5 MHz, the value of A.C. conductivity were hesitant in random values with a very small differential ranges for all compositional nano ferrite tablets, while the ϵ'_r , ϵ''_r and $\tan \delta$ were begun to be constant in these frequency period, this may be related to the hopping of charge carriers could not follow the applied field in the same applied frequency resulting in hesitant in the A.C. conductivity values and it could be solved by shaping the tablets with different diameters and thicknesses with repeating the A.C. conductivity test to the new dimensions to deal with the higher frequencies.



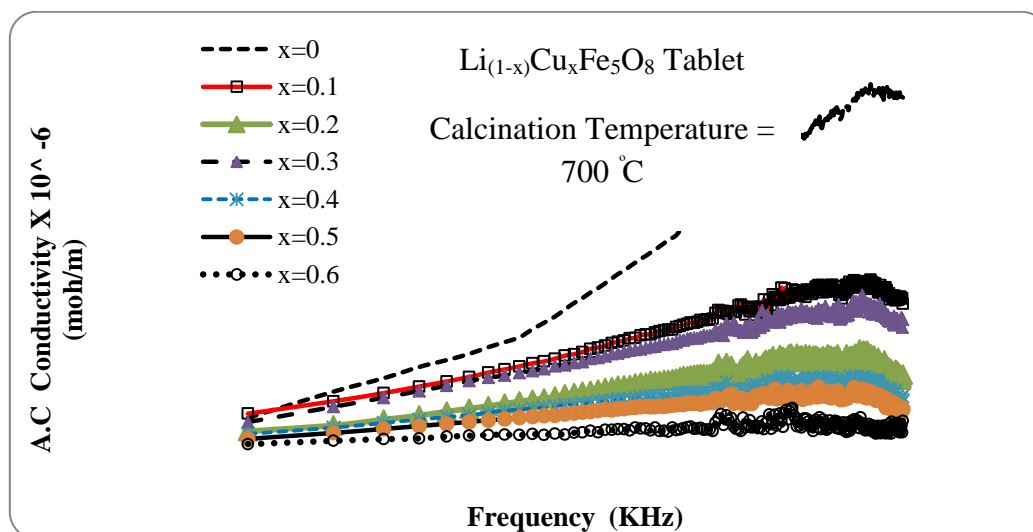


Figure (8): The AC Conductivity of $\text{Li}_{0.4}\text{Cu}_{0.6}\text{Fe}_5\text{O}_8$ and $\text{Li}_{0.3}\text{Cu}_{0.7}\text{Fe}_5\text{O}_8$ Tablet Calcinated at 700 °C as a Function of Frequency

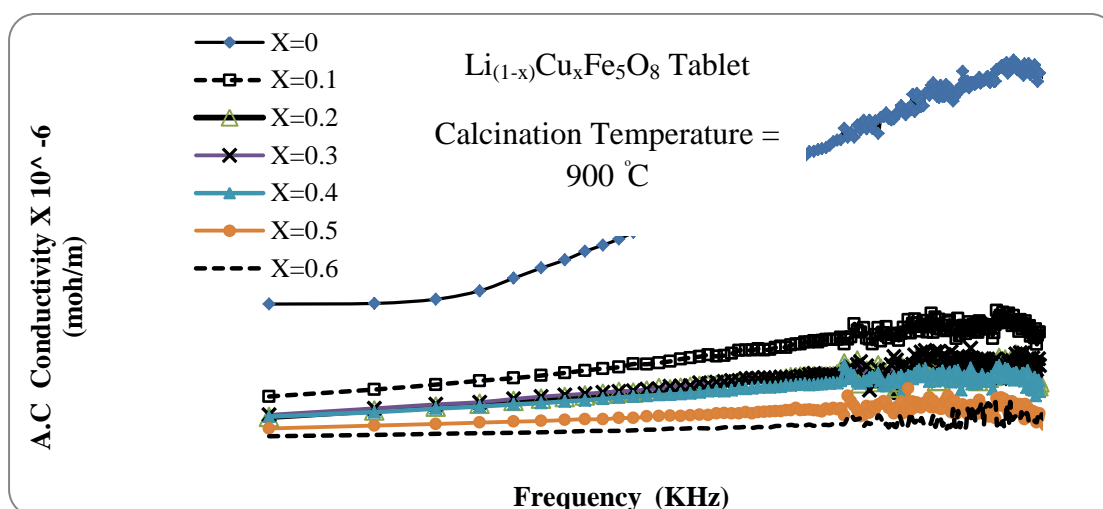


Figure (9): The AC Conductivity of $\text{Li}_{(1-x)}\text{Cu}_x\text{Fe}_5\text{O}_8$ Tablet Calcinated at 900 °C as a Function of Frequency



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