

Synthesis and characterization of Li-ferrite doped Cdferrite

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Abstract:

This paper focused to prepared $Li_x Cd_{1-x} Fe_2 O_4$ complex at Varity compound (x = 0, 1,2,5,10,15, and 20 wt%), by using the solid state reaction. samples disc with diameter 1.5 cm and thickness 0.4cm prepared at temperature 1100 C° for two hours in air. The x- ray diffraction using to study the structure of all samples and the volume of crystals for composition using sherrer equation according to Hall method. The in dices of lattice increased linearly with increased Li in Li Cd Fe_2O_4 composites. The electric measurement for complexes in as a function of frequency with range (50 HZ - 1 M H Z). Real dielectric constant in lower frequency decreased with increased Li content in Li Cd $Fe_2 O_4$, Its' found the x= 15% from Li has upper Values at real dielectric constant and decreased with increasing frequency for all complexes prepared. The imaginary of dielectric constant decreased with increased Li of $Li_x Cd_{1-x} Fe_2 O_4$ samples in lowering frequent and decreased with increased frequency and the concentration x= 15% of Li has upper value at imaginary dielectric constant. The tan δ decreased with increased of Li concentration for Li cd Fe_2O_4 in lower frequency and concentration x= 15% of Li has upper tan δ Also, the tan δ , decreased with increased frequency for all samples. The alternative conductivity, will decreased with increased Li concentration in Li Cd Fe_2O_4 for x = 15% of Li has upper alterative conductivity. We noticed $\sigma_{a,c}$ increased with increased frequency for samples.

Keywords: Li-ferrite, Cd-ferrite, structure, Dielectric constant, SEM.

INTRODUCTION

The Ferrites material has used in different science and technology especially in communication instrument, electronic devices and computers. This device is produced with easy low cost and large aboundence, it is cheave of magnetic material {1}. In addition, this material is poly crystalline, this indicate that materials are dielectric it uses in much more applied in technology and industrial {2}. the dielectric properties of Ferrites depend on the prepared methods (sintering temperature) and centering time {3,4}. The lower

conductivity for these material effectly by large on the dielectric properties and magnetism {2}. Ferrites are lron complex have good electric properties, they have large electric resistant and lowering dielectric losses with magnetic properties, It leads to use in different technology devices from microwave to radio frequency {3,5}. The different in electric conductivity by is a function of electric and magnetic properties with chemical variable of structure. It depends on temperature {5}. However, the electric and magnetic properties make ferrite is very important in technology {6-8}. The metal oxides M Fe_2O_4 are



magnetic material have spinal cubic structure, It makes that's material used in electronic and electric devices, it has transmition in radio frequency $\{9\}$. According dielectric properties for this material we can understand the properties for this material we can understand the polarity and chemical structure $\{10\}$. For the recently in berview, the cadmium Ferrite Cd Fe_4O_4 has used in many applied in different field $\{11\}$. It has spinal structure with cadmium ions in square position space It lists as under antimagnetic materials $\{12\}$.

Concentration and ions levels for Iron and diffusion through octahedral structure surface, its' limited the magnetic properties with electric properties for ferrite molecule $\{13\}$. The ferrite molecule contains cadmium (Cd) has spinel structure, it has able to use in important industrial applicate. This because the magnetic and electric properties $\{14\}$.

The chemical stability for steel with good magnetic and mechanical for Cd Fe_2O_4 made that to use in many variety industrials applied especially in elastic magnetic and cost materials $\{15\}$. The Li Fe_2O_4 is one types of Ferrite, it is very important traditionally and it used in many applied a specially in oil cell, remote sensor instrument and electronic devices. These because it has higher chemical stability with less metal and large curie temperature $\{16 - 24\}$. on the other hand, it has steel structure that's uses into high frequency material $\{25 - 27\}$. The Li Fe_2O_4 are prepared with different methods. The method of preparing, controlling and temperature of sintering are very important for preparing Li Fe_2O_4 . The temperature must be lowering to prevent diffusion of Li and oxygen and exchange cd^{+2} , cu^{+2} , Ni^{+2} , and Zn^{+2} effected on the Li Fe_2O_4 properties {28}. R. K. Kotnla and et.al discussion the added small quantity of ionic Ni to exchange basic Li Fe_2O_4 leads to improvement the characteristic and lead to increasing the electric properties with each other, then the exchange Ni ions lead to decreased the energy levels, it indicate that it's very important in energy applied. S. S. Belled and et.al {29} show that exchange of Liferrite by Cd indicate that increased content ratio of Cd with homogenous grain size volume. In this paper we prepared Li Fe_2O_4 and cd fe_2O_4 using conventional method because it very important industrial applied for these materials especially in electronic devices. The Li Fe_2O_4 was added to Cd Fe_2O_4 with concentration (1,2,5,10, 15 and 20 wt%). The structure properties have studied such that ρ_{x-ray} and crystalline size using Debye scherrer and willumson – hall method. The XRD used to study the lattic parameters for $Li_xCd_{1-x}Fe_2O_4$.

Experimental:

The samples of Li Fe_2O_4 are preparing using the ceramic traditional. The Iron oxide Fe_2O_3 and Lithium carboniteLi2Co3 materials used with ratio (1:5), furthermore, The Cd Fe_2O_4 prepared with same technical. The materials are used are Fe_2O_3 and Cdo under ration (1:1) with aceton to mixed the containing in backage with good mixture the mixture reformed by bulk and treatment by heating at (1100 °C) for two hours and cooling for room temperature. After we produce powder Bulk another time for three hours and reformed with heating at (1100 °C) and cooling Bulk for room temperature to produce a powder of Li Fe_2O_4 . Next, we reformed the first powder of cadmium ferrite at treating under (1100 °C) for two hours and cooling for room temperature and reach to good powder of cadmium Ferrite. The Lithium added with different concentration (1,2,5,10,15, and 20 wt %) for Cadmium Ferrite by mixed one ration single and compose under pressure (10 ton) to produce samples have diameter a proximally (1.5) cm with width (0.4)cm and sintering to air to (1100 °C) for two hours. the x-ray diffraction has used to study the structure of crystal for all samples and show that a single phase



with characteristic (cu K_{α} radiation, $\lambda = 1.5405 \text{ A}^{\circ}$) type Philips X pert Diffractometer. The theoretical density ρ_{x-ray} of the all samples was calculated using the formula ($\rho_x = 8M / N a 3$) where (M) is the molecular weight, (N) is Avogadros' number and (a) is the lattice parameter. The experimental density (ho_{exp}) of all samples was measured in bidistilled water using Archimedes method. The porosity (P%) was calculated using the relation $P=(1-\rho/\rho x)$ 100%. The average crystalline size was calculated using Deby -Scherrer equation: $D = \frac{0.89\lambda}{\beta \cos \theta}$ were λ is the wave length of the x – ray ($\lambda = 1.5 \ 05 \ A^{\circ}$), β is the full width at half maximum in radians of the maximum intensity peak and (θ) is the angle at which the maximum peak occurs $\{30,31\}$. The average crystalline size for the samples can be determined by using Williamson -Hall equation {32}. $\beta \cos\theta = \frac{K\lambda}{R} + 4 \text{ s} \sin\theta$ where s is the microstrain in the sample. If $\beta \cos \theta$ is plotted with respect to 4 sin θ for all peaks. microstrain and crystalline size can be, determined from the slope and y - intercept of the fitted line respectively. the dielectric constant and dielectric loss tangent were measured in the frequency range 50 Hz - 1 M Hz at room temperature by using (a Hp 4 2 8 4 L C R meter).

Results and Discussion:

X – ray diffraction pattern of Lithium Ferrite powder Li Fe_5O_8 that has been prepared at a temperature of 1100 °C and for two hours using the dub`139le sintering methods shown in figure (1). Reflection planes of the spinel ferrite structure shown in x – ray diffraction that were compared with the standard card date (00-017-0114), (00-017-0938), and these reflections are (211), (220), (311), (400), (422), (511), (440), (620), (444), (730), (751), (840). Where it was found that the crystal structure is a cubic with a lattice spinel parameter a = 8.333 A° which corresponds to the value previously confirmed by S. Y. An {33}.



Also, cadmium ferrite Cd Fe_2O_4 was prepared with a temperature of 1100 °C for two hours, where figure (2) shows the x-ray diffraction pattern for Cd Fe_2O_4 , where the reflectance for planes of the spinel structure shown in the x – ray diffraction were compared with the standard card data (00 – 022 – 1063) and these reflections (220), (311), (222), (400), (331), (422), (511), (440), (620), (533). the crystal structure was found with a spinel cubic with lattice parameter a = 8.6996 A°, and this value corresponds to the value it found P.K. Nayak {34,35}.



Fig. (2): XRD of Cd Fe_2O_4

Figure (3) shows the x – ray diffraction patterns for the ferrite samples, $Li_xCd_{x-1}Fe_2O_4$ Which were compared with the standard card (00 – 022 – 1063). It



was found that all the peaks that appeared showed the structure of the spinel cubic of cd Fe_2O_4 . The addition of lithium in different concentrations in the $Li_r Cd_{1-r} Fe_2 O_4$, has an effect on the formation of Cdo and $Fe_2 O_3$ phases. It was also noticed that the crystallization of Cd Fe_2O_4 increased with the addition of lithium concentrations and the highest peak intensity was at x = 10 % and this was confirmed by researchers {36}. The lattice constants (a) of the spinel cube were calculated using d-spacing and (h K 1) Millers' indecis and using the relation ship a = d $\sqrt{h^2 + K^2 + L^2}$. The lattice constants changes with the lithium concentration in Li_x Cd $Fe_2 O_4$ shown in table (1), where we notice that the lattice constants decrease



and then fluctuate with the increase in the lithium concentration. This follows the law Vegards'. ionic radius of Cd^{2+} (0.97 A°). L i^+ (0.71 A°) and Fe^{3+} (0.65 A°). Lithium ions replace the $Fe_2 O_3$ ions on A - site, and this explains the decrease in the lattice constants with increased lithium concentrations, and this was confirmed by {37}.

Liof	aA°	D _{sh}	D_{w-h}	$ ho_x$	$ ho_{a pp}$	Porolsity%	Average grain
Addlitives		nm	nm	gm/cm ³	g/cm ³		Size µm
0	8.662	43.	42.529	5.888	3.852	34.6	2.168
		336					
1	8.681	48.041	42.0136	5.847	3.63	37.9	
2	8.620	47.974	44.724	5.974	3.64	39.1	
5	8.636	46.031	46.215	5.942	3.833	35.5	
10	8.649	39.068	37.472	5.92	3.604	39.13	2.019
15	8.553	21.491	22.007	6.12	4.068	33.5	1.907
20	8.612	40.534	39.613	5.98	3.91	34.6	1.313

Table (1): lattice constant and Average crystalline size of $Li_xCd_{1-x}Fe_2O_4$







Fig (4): Williamson- Hall analysis of the $Li_x Cd_{1-x} Fe_2O_4$

The average crystal size shown in table (1) and Fig (4)its quantities ranged from (43.336- 48.041. - 40.534 A°) where we notice that these quantities change with in the composition of Li^+ additives. The variation is due to different locations of the unit cell. The increase in the average crystal size is due to increased concentrations of Li^+ from (0-2%) and with the increase in the Li^+ additions, the average crystal size has decreased And this may be due to atomic radius of and cd^{2+} ions. Li^+ The increased of Li^+ concentration, in contrast, decrease in the amounts of cd^{2+} in the compositions, and this may lead to a change in different locations of the unit cell {38}. In addition. The increase in the average size of the crystal is because Li^+ ions are present in octahedral sites instead of tetrahedral sites and the decrease in the average crystal size with in creasing Li^+ con centrations is due to the compressive stress of the Li^+ ions {39}.

Density has an important role in controlling the properties of crystalline ferrites. Where the apparent density (ρ_{app}) and the x – ray density (ρ_x) were measured for the prepared samples. Methods were measured in the experimental. The apparent density and x – ray density with the additions of Li – ferrites were tabulated in table (1). It has been shown that the values of ρ_{app} are less than the values of the ρ_x and this may be due to the presence that were formed during the process of preparing samples and during the sintering process {40}. It was found that the ρ_{pp} decreased slightly with the additions of Li – ferrite, but increased at the two concentrations (x = 15, 20 %) of Li – ferrite. The explanation for this is that of volume ($0 < X \le 10$) the molecular weight of (Li –



Cd ferrite) decrease with the added Li -ferrite. Therefore, the cell unit size decreases and the rate of de crease in the molecular weight is greater than the increase in the volume of ($0 < X \le 10$), so the ρ_{app} decreases with Li^+ additions of the concentrations (0 $< X \leq 10$). As for (X = 15, 20 %) of (Li⁺) additions, the decrease in molecular weight is less than the increase in volume (X = 15, 20 %), so the ρ_{app} increases at (X = 15, 20 %). An increase in the (ρ_x) was observed with the addition of Li^+ . The increase in the ρ_x values was correlated with the lattice constant values of the prepared samples. In the composition L_i – Cd ferrite, the lattice constant decreases slightly with the increase in the Li^+ content, so the decrease in the values the lattice constant lead to an increase in the ρ_x of samples $\{14\}$. In addition, the percentage of porosity (**P**%) for the samples prepared was calculated using the apparent density and x - ray density values according to the relationship mentioned in the experimental and the variation in the porosity values reported in the table (1). where it was observed that the (P%) values increase linearly with the addition of (Li^+) because the rate of increase in the x - ray density with the (Li) additives is greater than the rate of apparent density, so the porosity (P%) increases with the increases in (Li) additives for (Li – Cd ferrites). As for the (x =15, 20%), the (P%) decreases, because the apparent density at addition of (Li){41}. one of the important factors that affect the electrical properties of ferrites is the grain diameter, so the surface morphology of the Li – Cd $Fe_2 O_4$ system has been studied using scanning electron technology, where grain growth is closely related to the movement of grain boundaries {42}. From the observation of the (SEM) images, Figure (5) shows that the grain size is irregular, and this is due to grain distribution. Average grain diameter was measured for the (x = 0, 10, 15, 20 %)of (Li^+) concentrations for the prepared samples and shown in the table (1). It was noted that there was a discrepancy in the diameter of the grains with the addition of (Li^+) , and the highest value of the diameter was at (x = 0) (Cd $Fe_2 O_4$), which reached (2. 186 μ m) which reached the lowest value for the diameter is when the (X = 20%) of (Li^+) in the Li – Cd $Fe_2 O_4$ system, and that was value (1.313 µm). The cause of the variation in the diameter of the grains is attributed to the diffusion coefficient and the concentration of ions {43}. Like was we can observe from table (1) that the diameters of the grains measured using SEM are greater than the dimeters of the particles calculated using the x - ray diffraction pattern. This clearly indicates that each particle detected by SEM consists of several crystalline granules {44}.



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 $X = 15 \qquad X = 20$ Fig (5): SEM for $Li_x Cd_{1-x} Fe_2 O_4$

Dielectric analysis:

Real and imaginative dielectric constant change with frequency for a wide range of compositions and with a frequency of (50 Hz–1MHz) shown in Figures (6,7). The real part of the dielectric constant gives the stored energy and the imaginary part gives the energy dissipated in the dominant field. we notice from the

two figures that the actual dielectric constant decreases with increasing frequency in all the prepared compositions and the decrease in the dielectric constant values rapidly in low frequencies, while the decrease is slow at high frequencies shown in table (2). The dispersion of the dielectric constant is due to the type of inter polar polarization {45,46}.

Table (2) Dielectric constant (ε) and Dielectric loss (tan δ) of the $Li_x Cd_{1-x} Fe_2 O_4$ With content Li – ferrite $0 \le X \le 20$.

content (w%)	x = 0	x = 1	x = 2	x = 5	x = 10	x = 15	X =20
ε at 5 kHz	60.49	10.98	10.58	9.65	21.26	24.87	12.86
ε at 100k Hz	29.58	6.90	7.56	6.77	10.52	14.09	7.93
ε at 500 KHZ	18.53	5.92	7.48	6.08	8.21	12.13	6.78
ε at 1MHz	15.23	5.40	7.54	5.64	7.23	10.95	6.25
Tan δ at 5 kHz	0.97	0.24	0.10	0.091	0.54	0.66	0.172
Tan δ at 100k Hz	0.475	0.167	0.101	0.107	0.304	0.218	0.171
Tan δ at 500 kHz	0.477	0.478	0.0047	0.0035	0.1707	0.142	0.0403
Tan δ at 1MHz	0.443	0.054	0.0146	0.0155	0.166	0.173	0.038

Large values of the dielectric constant ions, are at low frequencies due to Fe^{+2} ions, oxygen Spaces, grain boundary defects, and interstitial distribution {47}. The polarization process in a ferrite is similar to the

conduction mechanism {48}. The transmission of electrons between iron ions and ferric ions on a site of eight surfaces leads to the dis placement of electrons in the direction of the applied field, and these



electrons determine the polarization. This type of behavior was previously mentioned {49}. At high frequencies, polarization decreases and a constant value of pearls arrives. The reason for this is that at a certain frequency of the applied field the electron transmission does not Follow the alternating field. Therefore, there is a light decrease in the values of the dielectric constant at high frequencies. The loss factor $(\tan \delta)$ was changed with the frequency in the room temperature for all combinations prepared from the $Li_r Cd_{1-r} Fe_2 O_4$ system shown in figure (8). The loss factor represents the energy dissipation in the insulating system and the cause of this loss is the domain wall resonance. This loss is low at high frequencies and this is due to the obstacle in the rotation of the field wall in addition to that, the loss Factor is proportional to the imaginary part the dielectric constant. From all of the above, we can infer that the loss of the insulator in the ferrite is the result of electron transmission contributes to the insulation loss only in the low frequency and the electron transmission is the reason for the low loss factor in high frequencies $\{50\}$.

The alternating conductivity $\sigma_{a,c}$ with the frequency is shown in figure (9), and we notice that all the curves are very dispersed with frequency, and this behavior is very important in the ferrite. The reason for the electrical conductivity in ferrite is due to the transfer of electrons between ions for the same element in more than one valence state and randomly distribute it in crystal lattice sites {51}. The isolation parameters of the Li $Fe_2 O_4$ additives in Cd $Fe_2 O_4$ decrease with the addition of *Li* ions, and this is evident by the curves in the previous forms of the insulating properties of the $Li_x Cd_{1-x} Fe_2 O_4$ system.



Fig (9): conductivity $\sigma_{a,c}$ of $Li_x Cd_{1-x} Fe_2 O_4$

Conclusion:

In summary, we added Li to Cd - ferrite with concentration (0,1,2,5,10,15 and 20) wt % for $Li_x cd_{1-x} Fe_2 O_4$ that prepared by traditional method. The crystal structure for all samples are single phase spinel structure, The results show that lattice constant, crystal size and XRD density are increased with increased *Li* concentration in $Li_x Cd_{1-x} Fe_2 O_4$. Furthermore, the apparent density values are lower than XRD density because the porosity that form thought prepared the samples cress sintering processes. on the other hand, the morphology of the surface $Li_x Cd_{1-x} Fe_2 O_4$ studies and measuring the average of diameter for preparing composition samples, the measuring show that diameters decreased with increased Li in $Li_x Cd_{1-x} Fe_2 O_4$. The measurement of diameter the samples by SEM have large values that calculated using XRD, the dielectric constant and loses factor are calculated for all samples and show the lower value of dielectric constant at (x = 5wt %) for $Li_x Cd_{1-x} Fe_2 O_4$ and the losses factor is decreases for high frequency because the a badent of the resistant field wall.

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