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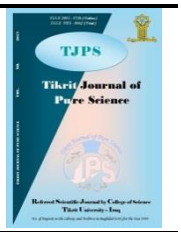

Fabrication and study of the structural and spectral properties of the composites (y) $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 + (1-y) PZT prepared by the powder technology method

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Abstract

Ferrite-ferroelectric composites holds following chemical formula (y) $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 + (1-y) PZT with different weight rations (y=0.15, 0.30 and 0.45) are prepared by the powder technology method at annealing temperature (1000 °C) for (12hrs). The structural properties are also studied representing by the lattice constants (a, c), particle size (D_t), apparent density (ρ_a), and X-ray density (ρ_x). It has been shown that increasing the concentration of the ferrite phase contributes to an increase in the lattice constant, apparent density and X-ray density which in turn contribute in decreasing porosity that plays a role in improving the physical properties of the samples as well as the weight ratios of the mixture after the final sintering with reference to x-ray diffraction technique. In addition, the spectral properties were also studied represented by the vibration force constant and regions of optical absorption in terms of IR technique.

1. Introduction

The development and technical progress are in need of new engineering materials that have unique properties, which lad to the emergence of materials called (composite materials) which are a mixture of two or more materials, one of which is called the 'matrix material' and the other called the 'reinforcement material' [1]. The matrix materials are ceramic, metal or polymeric, and the reinforcement materials are either fiber bristles, plates or sheet piles [2]. Each of these materials is different in mechanical, physical or chemical properties, thus producing a new material with different properties than the properties of its constituent materials. The new material possesses properties that cannot be obtained from traditional materials such as ceramic, polymeric and metallic materials. Composite materials are of low cost and high mechanical properties, with the possibility of changing or developing these materials, in addition to their ease of transport and light weight [3]. Examples of these composites are the two- phase composites made of ferrite. Ferroelectric are new materials that possess general physical properties combining the properties of mixed materials and have the advantage that its mixture does not cause any chemical reaction. Among the properties of these materials are that they have double qualities by being ceramic and metallic, metallic and non-metallic, or polymeric metallic, etc. The composite materials are characterized by the following properties [4]:

1- Easy to form complex shapes with large dimensions and sizes.
2- Its service ageing is greater than the rest of the traditional materials.
3- Its durability is very high compared with the durability of traditional materials.
4- Greater resistance to chemicals.
5- Very Light weight without affecting durability characteristics.
6- Massive resistance against the spread of cracks that appear as a result of vibrations.
7- High thermal (heat) resistance.

S. S. Chougule, et. al [5] were able to prepare two-phase composites from Ferrite materials with chemical formula $Ni_{0.8}$ $Zn_{0.2}$ Fe_2O_4 and Ferroelectric materials with chemical formula $Pb(Zr_{0.52}$ $Ti_{0.48})$ O_3 with weight ratios (0.15, 0.30, 0.45 and 1) which were prepared by the traditional double sintering method for Ceramic. The prepared composites phases, were analyzed by using the X-ray diffraction technology method. As the results show that the

Ferrite phases were of a Cubic Spinel type and the structure of Ferroelectric phases were of tetragonal crystalline structure. Also, they were able to study the dielectric properties represented by the dielectric constant and the amount of dielectric loss within the frequency range of (20 Hz- 1 MHz).

While the Magnetoelectric composites are able to be prepared by Rani [6] with chemical formula (y) $Ni_{0.8}$ $Zn_{0.2}$ Fe_2O_4 + (1-y) $Ba_{0.9}$ $Sr_{0.1}$ $Zr_{0.04}$ $Ti_{0.96}O_3$ prepared by the solid state reactions, it has been shown from the results that both phases of Ferrite and Ferroelectric existed by using the X-ray diffraction technology and they were also able of calculating the Lattice parameters.

The aims of the present work are:

1- Preparing the ferrite composite holding the following chemical formula (y) $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 and the ferroelectric composite PZT and the two-phase composites of ferrite - ferroelectric with the following chemical formula (y) $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 + (1-y) PZT (where y= 0, 0.15, 0.3 and 0.45) named as (Z_0, Z_1, Z_2, Z_3 and Z_4) respectively,which have been prepared by the powder technology method.

2- Studying the samples structural properties represented by the lattice constant, particle size, the apparent density and the X-ray density, by using X-Ray Diffraction (XRD) technique.

3- Understanding and studying the spectral properties represented by the force vibration constant, bond length, and optical absorption regions in terms of IR technique.

2- Experimental details

2-1- Preparation of composites

The samples were prepared in three stages: the first stage includes the ferrite phase preparation steps which has the following chemical formula $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 with the use of manganese dioxide (MnO_2), zinc oxide (ZnO) and iron oxide (Fe_2O_3) and with 99.5% purity and of Indian origin, the primary materials are weighed by using a sensitive balance. The weights of the composites in this reaction are determined based on the molecular weight of each element as follows:

M.wt for MnO_2= 1*54.938+2*15.999 = 86.936 gram/mole

M.wt for ZnO= 1*6537+1*15.999 = 81.369 gram/mole

M.wt for Fe_2O_3= 2*55.847+3*15.999 = 159.691 gram/mole

The method of solid state reactions (powder technology) at annealing temperature degree of 1200 °C, where the primary material is mixed well with a purity of 99.52%. It is grinded well by an agate mortar pestle for a period of 3 hours to obtain a homogenous mixture of the elements (Mn-Zn-Fe-O) with fine and homogenous granular size. The mixture is then heated to a temperature of 800 °C for a period of 4 hours after placing it in a crucible inside an electric furnace. After taking it out of the furnace, we heat it again for 2 hours so that it becomes a homogeneous mixture. After that, we sieve the sample with a (75) mm diameter sieve to get soft granules. Then, the bonding material which is polyvinyl alcohol (PVA) is added with a ratio of (2-3) drops for mix consistency. This material evaporates during the final heating. The samples are compressed under pressure (5 tons) for a period of (5 minutes) for the purpose of obtaining the sizes and forms of the required samples. After that, the compressed samples are heated at 1200°C for 12 hours using an electric furnace with a temperature reaching up to 1300°C. Then the samples are cooled inside the furnace until it reaches room temperature in order to obtain the complete integrated ferrite phase, and increasing the annealing temperature degree will less the pores and increase the size of the granules (particle size).

By following the same previous steps, the second stage of the fabricating stages can be achieved, which is the method of preparing the ferroelectric phase holding the chemical formula pb ($Zr_{0.52}$ $Ti_{0.48}$) O_3 using the primary materials PbO, ZrO_2, and TiO_2 with a purity of 99.5% and which is of Indian origin. The weights of the composites involved in this reaction are also determined by depending on the molecular weight and according to the same previous procedure used to calculate the required weights in the ferrite phase. They are mixed for 3 hours in order to homogenize the powder which is composed of Pb-Zr-Ti-O, and then the powder is treated with initial heating stages at a temperature of 800 °C for 4 hours. After that, a bonding material is added and the sample is compressed in a mold with a diameter of 1 cm at a pressure of 5 tons. Then, it is thermally treated at 950 °C for 12 hours to obtain the required phase. After finishing these two stages, the third phase starts in order to obtain the required ferrite - ferroelectric composites by taking the weight ratios of the ferrite phase (15%, 30%, and 45%) with the correspondance weight ratios of the ferroelectric phase (85%, 70%, 55%) then these ratios are mixed for 3 hours and heating it as final heating at a temperature of 1000˚C for two hours.

2-2- Characterization techniques:

This part includes the practical techniques used in studying the structural properties represented by the lattice constant, physical density, and density in terms of X-ray diffraction technique.

2-2-1 X-ray diffraction (XRD): -

The x-ray diffraction technique is used to study the structural properties of the prepared samples represented by the lattice constant, where the lattice constant of the ferrite composites with a crystalline crystal structure is calculated using the following mathematical equation [7].

$$d = \frac{a}{\sqrt{h^2+k^2+\ell^2}} \quad \ldots\ldots\ldots(1)$$

Where:

a: Lattice constant.

d: the distance between the planes.

(hkl): Miller indices.

The lattice parameters (a, c) for ferroelectric composites of quadratic structure is also calculated using the following mathematical equation [8].

$$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{\ell^2}{C^2} \quad \text{.....(2)}$$

The calculation of the particle size of all the samples is conducted using the Debye - Scherrer equation which is clarified by the following mathematical equation [9]:

$D_p = K\lambda / (\beta \cos \theta)$ ……(3)

where:

K: shape constant of the value 0.9 (assuming the particles are spherical in shape).

λ: The X-ray wavelength which equals (1.5418 Å).

β: Full Width at Half Maximum (FWHM) of the diffraction peak, in radian.

θ: Bragg angle, which is converted from degree to a radian after multiplication by π / 180.

As for the measurement of the apparent density (physical) of all the samples for ferrite - ferroelectric composite, this is conducted by using the following mathematical formula [10].

$\rho_a = m/V$ ……(4)

where

m = mass of the sample

V = Volume of the sample in which $V = \pi r^2 h$

Where *r* represents the sample radius, and *h* is the thickness of the sample.

The density in terms of x-ray diffraction and the density of the two-phase ferrite ferroelectric composites are calculated using the following relations:

For ferrite phase having cubic spinel structure, the X-ray density is calculated by [11]:

$$\rho_{x_1} = \frac{8M}{Na^3} \quad \text{……(5)}$$

Where: -

M = is the molecular weight in gram unit.

N_A = Avogadro number which is equal to 6.023×10^{23}

For ferroelectric phase having tetragonal structure, the x-ray density is calculated by [8]: -

$$\rho_{x_2} = \frac{M}{Na^2 c} \quad \text{…….(6)}$$

For ferrite-ferroelectric composites [12]: -

$$\rho_{x_3} = \frac{M_1+M_2}{V_1+V_2} \quad \text{……(7)}$$

Where: -

M_1 = y (Mol. Wt. of ferrite).

M_2 = (1-y) (Mol. Wt. ferroelectric).

$$V_1 = \frac{M_1}{\rho_{x_1}}$$

$$V_2 = \frac{M_2}{\rho_{x_2}} \quad \text{…….(8)}$$

Where: -

y = molecular weight i.e. 0.15, 0.30 and 0.45.

The porosity was then calculated by referring to the following relation [13]:

$$P = \frac{\rho_x - \rho_a}{\rho_x} \times 100\% \text{ …. (9)}$$

Where: -

ρ_x = X-ray density

ρ_a = apparent density

2-2-2 IR Spectroscopy: -

The Force of Infrared Radiation (FTIR) is calculated based on the following mathematical relationship [14].

$F = 4\pi^2 c^2 \upsilon^2 M$

Where: -

F = force of Infrared Radiation.

c = light speed (3×10^8 m/sec).

M = Mass decreases between ions O^{-2}, Fe^{+3}.

υ_1, υ_2 = are equal to the absorption ranges frequency.

3- Results and Discussion

3-1 XRD analysis: -

Table (1) shows Miller parameters (hkl) and the spacing (d) between adjacent (hkl) lattice planes which are obtained from the X-ray diffraction that results for Z_0, Z_1, Z_2, Z_3 and Z_4, which clarify the x-ray diffraction manner of ferrite - ferroelectric composites holding a chemical formula with weight ratios for the ferrite phase (0, 0.15, 0.30, 0.45, 1) and the corresponding weight ratios for the ferroelectric phase (0, 0.55, 0.70, 0.85, 1) named according to the following forms (Z_0, Z_1, Z_2, Z_3 and Z_4), respectively.

Table (2) provides information on the lattice, density and porosity of all composites. From this table, it is noticed that the lattice constants (a-c) for the PZT stage in the composites series (y) $Mn_{0.6}$ $Zn_{0.4}$ Fe_2O_4 + (1-y) PZT has been found to be slightly related to the change in the percentage of delivery in either of the two stages when compared with the lattice parameters in the main stages. The comparison of the lattice parameters refers to the individual ferrite phase in the composites (Z_0, Z_1, Z_2, Z_3 and Z_4), but the lattice constant for the ferrite phase continues to increase with the increase of Zn^{+2} concentration in MZFO while the lattice constant (a-c) for ferroelectric phase continues to decrease. The decrease of the c/a ratio in the case of PZT refers to the fact that the crystalline lattice PZT is tense in the composite. The x-ray density as well as the physical density data of composites under study show decrease while the percentage of porosity is shown to be increased if compared to the percentage of porosity of the ferrite – ferroelectric phases separately as shown in table (2). It may be attributed to the small percentage of ferrite in the composite and the probable dispersal of the density and porosity in the composite [15].

Table (1): Miller parameters (hkl) and the spacing (d) which were obtained from the X-ray diffraction results for Z_0, Z_1, Z_2, Z_3 and Z_4.

(hkl)	$d_{Std.}$ (Å)	d_{Z_0}(Å)	d_{Z_1} (Å)	d_{Z_2} (Å)	d_{Z_3} (Å)	d_{Z_4} (Å)
(100)	4.03	4.05	4.03	4.02	4.03	4.05
(220)	1.42	1.42	-	1.41	1.46	1.42
(110)	2.85	2.84	2.87	2.85	2.93	2.84
(311)	2.54	2.49	2.57	2.50	2.50	-
(222)	2.71	2.72	-	2.77	2.72	-
(200)	2.03	2.05	2.01	2.05	2.05	2.05
(422)	1.90	1.70	1.90	-	1.92	-
(211)	1.65	1.64	1.65	1.65	1.65	1.64
(511)	1.62	1.60	-	1.64	-	1.62
(301)	1.28	1.24	1.24	1.28	1.28	1.24
(111)	2.34	2.30	2.31	2.33	2.29	2.30
(002)	2.07	2.05	2.07	2.05	2.05	2.01
(400)	2.35	-	2.35	2.33	2.33	2.35
(022)	1.44	-	1.44	-	1.42	1.44
(103)	1.30	-	1.30	–	1.32	-
(101)	2.89	2.84	-	2.93	2.83	2.84
(004)	1.03	1.03	1.05	–	–	1.05
(210)	1.80	1.81	1.80	1.80	-	1.81
(201)	1.82	1.81	–	1.80	-	1.81
(112)	1.67	-	–	–	1.46	1.67
(310)	1.23	–	1.25	1.25	1.23	1.23
(001)	4.14	4.12	–	4.14	–	4.12
(012)	1.60	1.62	–	1.60	-	1.62
(300)	1.35	1.30	1.35	–	–	1.30
(020)	2.15	–	-	2.15	2.19	2.15

Table (2): Lattice constant, X-ray density, physical density and porosity for the composites Z_0, Z_1, Z_2, Z_3 and Z_4.

Composition (y) Mole%	Lattice parameters of the phases						
	Ferrite	Ferro electric					
Samples	a (Å)	a (Å)	c (Å)	c/a	ρ_x g/cm^3	ρ_a g/cm^3	P%
$YMn_{0.6}Zn_{0.4}Fe_2O_4$+(1-y)PZT							
Z_0	8.4135	------	_____	_____	5.2365	5.3040	14.28
Z_1	7.7671	4.0590	3.3007	0.8131	6.2140	5.6993	19.28
Z_2	6.8211	4.0150	3.2812	0.8172	6.0641	4.9705	20.03
Z_3	9.1825	4.0051	4.1175	1.0280	5.9055	4.2690	21.71
Z_4	-------	4.0281	5.5753	0.7284	6.3560	6.7508	12.94

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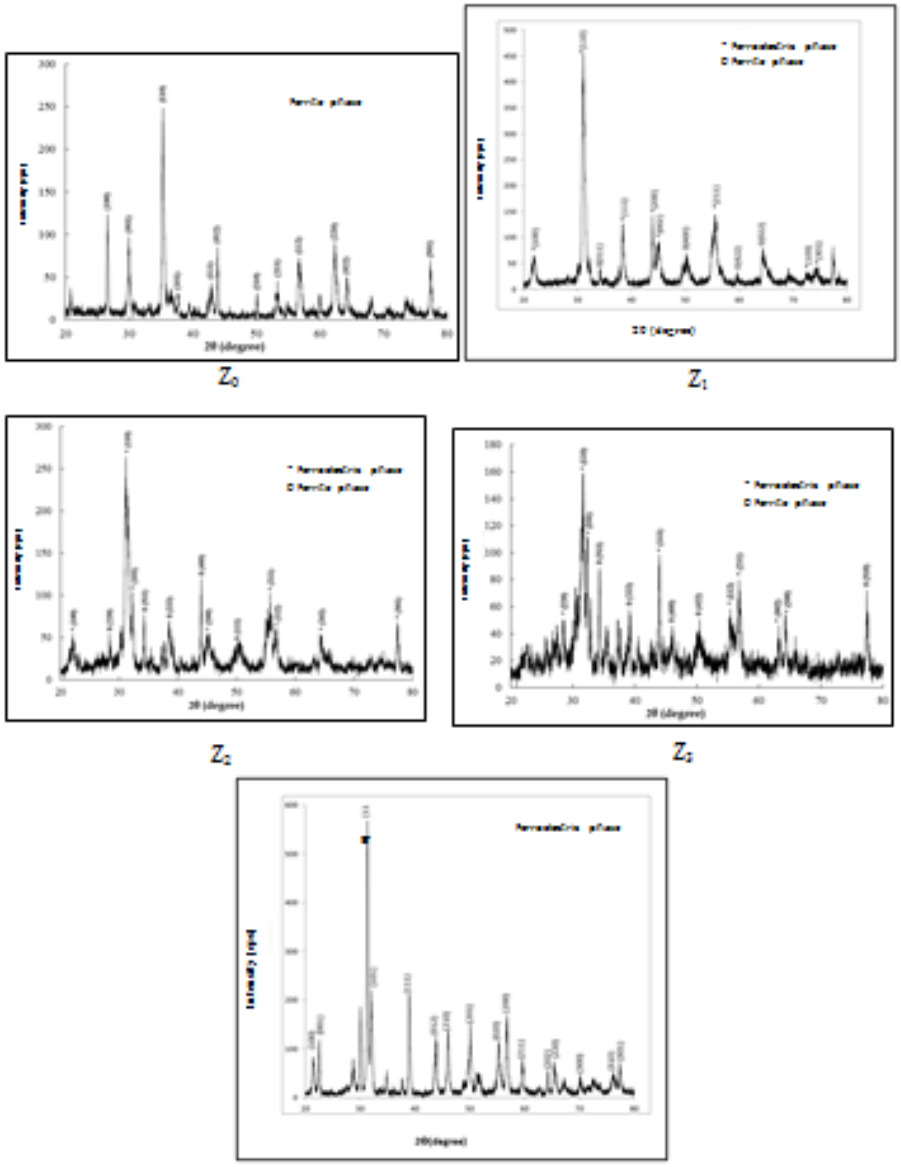

Fig (1) X-ray Diffraction patterns of Z_0, Z_1, Z_2, Z_3 and Z_4 samples.

3-2 FTIR analysis: -

Fig. (2) shows the IR spectrum resulting from the two- phase of ferrite-ferroelectric. It is clear that there are three absorption regions in the range 594 cm^{-1}, 634 cm^{-1}, and 974 cm^{-1} for Z_1, Z_2 and Z_3 respectively, that are the result of the lattice vibration which is in turn the result of the chemical bonds of the two phases for the ferrite. These are Mn-O within the range (415-422 cm^{-1}), Zn-O of the value 422 cm^{-1}, and Fe-O. In addition, there is the ferroelectric phase which is a result of the lattice vibration which is in turn a result of the chemical bonds Zr-O and Ti – O. There is a also the appearance of an absorption region within the range (3437 – 3464 cm^{-1}) which is result of the moisture H-O-H [16]. As for F_0, F_1, F_2, F_3 and F_4 it is interpreted on the basis of the mathematical relationship $F=4\ \pi^2c^2\upsilon^2M$ which depends on the absorption regions for Z_0, Z_1, Z_2, Z_3 and Z_4 composites, as shown in table (3).

Table (3): - IR Spectrum results for Z_0, Z_1, Z_2, Z_3 and Z_4 respectively.

Samples	V_1 (cm^{-1})	V_2 (cm^{-1})	V_3 (cm^{-1})	V_4 (cm^{-1})	V_5 (cm^{-1})	F_0 (dyne/cm)	F_1 (dyne/cm)	F_2 (dyne/cm)	F_3 (dyne/cm)	F_4 (dyne/cm)
Z_0	459	569	634	-----	------	$9.22x10^7$	$9.79x10^7$	$2.94x10^7$	------	------
Z_1	594	634	1101	1327	1487	$2.31x10^7$	$1.82x10^7$	$1.33x10^7$	$1.06x10^7$	$1.51x10^7$
Z_2	634	974	1157	1381	1545	$5.27x10^7$	$8.61x10^7$	$2.94x10^7$	$9.42x10^7$	$1.34x10^7$
Z_3	415	571	634	904	1157	$3.39x10^7$	$4.44x10^7$	$10.32x10^7$	$3.17x10^7$	$5.89x10^7$
Z_4	602	------	------	------	------	$2.56x10^7$	$2.903x10^7$	------	------	------

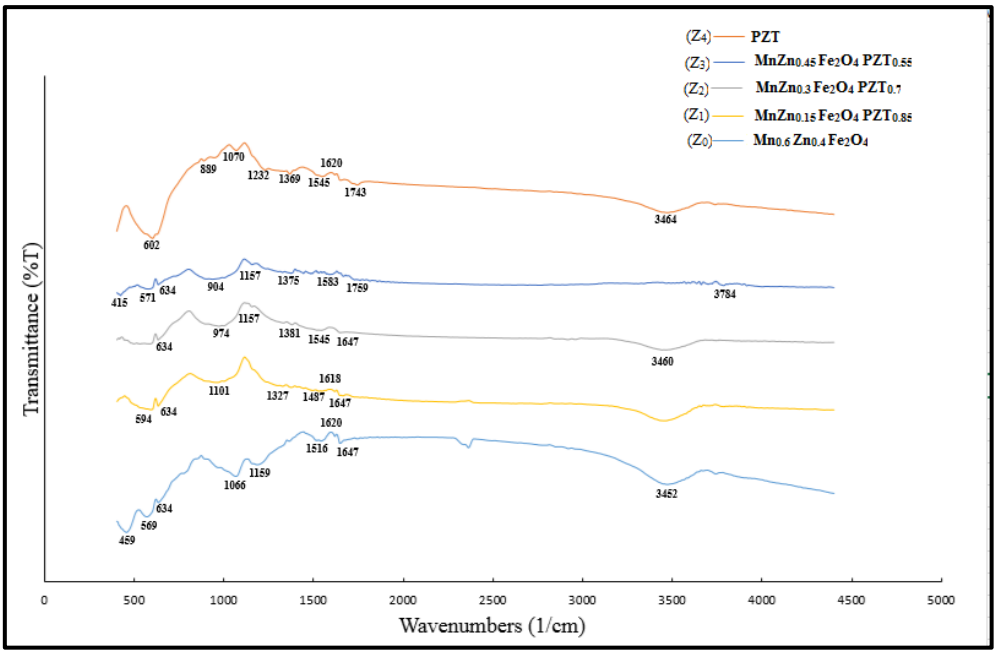

Fig. (2): - IR spectrum results for Z_0, Z_1, Z_2, Z_3 and Z_4 respectively

4- Conclusions

The results in this work show many points which could be taken under the consideration in the practical applications and usages of this prepared composite under the work circumstances and preparation method, and the following are the most important conclusions which were obtained from this work: -

1- The X-ray diffraction test results, show that the Crystalline structure of the composite Z_0 is a face-centered Cubic Spinel structure and the composite Z_4 is a tetragonal structure and the Z_1, Z_2, and Z_3 two-phase composites is a (face-centered cubic spinel-tetragonal) structure.

2- The lattice constant for the ferrite phase and ferroelectric phase increases with increasing in the concentration of Zn^{+2} and the reason behind this increases is that the radius of the substitutional ions Zn^{+2} (0.38 ˚A) is bigger than the atomic radius of the ions Mn^{+2} (0.74 ˚A) while the lattice constant for the ferrite-ferroelectric two-phase composites, the lattice constant for the ferrite phase is continuously increases with increasing in the concentration of Zn^{+2}, while the lattice constant for the ferroelectric phase is continuously decreases.

3- The physical density which is calculated from the X-ray diffraction results is decreasing while the porosity is increasing if it is compared with the porosity for the ferrite and ferroelectric phases separately.

4- Studies of FTIR spectra indicate that cubic for Z_0 sample and Z_4 sample. Z_0 sample show that the absorption range at the high frequency (4000 cm^{-1}) is bigger than the absorption at a lower frequency (400 cm^{-1}), while it has been shown for the Z_1, Z_2, and Z_3 composites show two-phases, that there are three absorption regions for the both phases of ferrite and ferroelectric in the range (450 – 3452) cm^{-1}, also it has been shown that there is an absorption region for all samples.

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تصنيع ودراسة الخواص التركيبية والطيفية لمركبات $(y)Mn_{0.6}Zn_{0.4}Fe_2O_4+(1-y)PZT$ المحضرة بطريقة تكلوجيا المساحيق

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[2] قسم الفيزياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

الملخص

تم تحضير مركبات الفرايت–الفيروكهربائية ذات الصيغة الكيميائية $(y)\ Mn_{0.6}Zn_{0.4}Fe_2O_4 + (1-y)\ PZT$ وبنسب وزنية مختلفة (y 0.30, 0.45 = 0.15,) بطريقة تكنلوجيا المساحيق عند درجة تلدين (1000 ℃) لمدة (12hr). كما تم دراسة الخواص التركيبية المتمثلة بثوابت الشبيكة (a, b) والحجم الحبيبي (D_t) والكثافة الفيزياوية الظاهرية (ρ_a) والكثافة بدلالة حيود الاشعة السينية (ρ_x) أذ تبين ان زيادة تركيز نسبة طور الفرايت يساهم في زيادة ثابت الشبيكة والكثافة الظاهرية والحقيقية التي بدورها تساهم في تقليل المسامية التي تلعب دور في تحسين الخواص الفيزياوية للنماذج وكذلك النسب الوزنية للخليط بعد التلبيد النهائي بدلالة تقنية حيود الاشعة السينة. كما تم دراسة الخواص الطيفية المتمثلة بثابت القوة الاهتزازية ومناطق الامتصاص البصرية بدلالة تقنية IR.