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Effects of the Ferrits Addition on the Properties of Polyethylene Terephthalate

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Abstract:

The products of composites material are very sensitive to many variables, such as manufacturing process, additive materials, etc. Filler or additive plays a major role to determine the formation of the properties and behavior of the composites. In this study, polyethylene terephthalate-based compounds were produced and characterized. The work is concerned to prepare samples of Polyethylene terephthalate (PET) - zinc-ferrite (ZnFe_2O_4) with different addition ratio as zinc-ferrite (1, 2, 5, 10, 15 and 20) wt% obtained from mixing the solution with a hot pressing method applied under optimum conditions. The densities of the composites for all samples were calculated. Through the work the diffusion of zinc-ferrite into the grain of Polyethylene terephthalate has been noted. Structural properties are studied by using X-ray powder pattern, the results of the XRD diffraction analysis showed that the structure for pure PET has four broad peaks at the ($2\theta=16.46^\circ, 17.45^\circ, 22.72^\circ, 25.98^\circ$). The non-crystalline behavior of the polymer and the ceramic compound indicates the presence of a crystalline phase with a single cubic structure with a space group $fd-3m$ (227)) ($a=b=c=8.44 \text{ \AA}$, $V=601.45 (\text{\AA})^3$). While adding zinc-ferrite to PET in different concentrations to obtain (PET / ZnFe_2O_4) composites material will increase the intensity of the X-ray peaks, and change the location of the peaks with the addition of zinc-ferrite. It is noted that X-ray diffraction patterns for PET / ZnFe_2O_4 (20%) indicates that there was no apparent variation of the patterns of pure Zn-ferrite ceramic diffraction which showed that the crystalline structure remains stable in PET / ZnFe_2O_4 . In addition, the electrical properties of the compounds represented by the dielectric constant (real and imaginary), dielectric loss ($\tan\delta$) and the A.C conductivity as a function of a range of frequencies (50Hz-1MHz) have been measured. The results indicated that these properties increase with increasing concentrations of ceramic addition. On the other hand, these properties decrease with increasing frequency due to the change in polarization mechanisms. It has been proven that these prepared superposed materials possess good stability properties in a wide range of frequencies, making these polymeric overlays of wide use in many applications. As for the measurements of Shore D hardness, it has been shown that the hardness of the compounds increases with increasing ceramic concentrations.

Key words: AC conductivity, Dielectric properties, PET/ ZnFe_2O_4 , Shore D, XRD.

Introduction:

Composites are important and necessary materials today because of their benefits. Composites of polymer based such as matrix ceramics as a filler receive increased care due to their electrical and optical properties, such as angular acceleration for accelerometers, acoustic emission of sensors, integrated separation of capacitors, electronic packaging in some potential applications and optoelectronics as device elements

such as LEDs and solar cells and laser diodes. Ceramic materials are usually brittle, and electrical insulation strength is low and requires high temperature. But then, the polymers are flexible and easy to handle easily at low temperatures and are displayed in a dielectric breakdown¹. The incorporation of inorganic fillings into polymer matrices were the subject of many studies²⁻⁶. Gorrasi et al.³ prepared and studied the physical

properties of a PET-based composite material reinforced with nanotubes scattered in fancy locations for packaging applications. According to these authors, the behavior of the compound was similar to that observed in the literature. Kilinc et al.⁴ examined the retardants of flame and tensile properties of the PET composite material containing boron and phosphorous additives, which were processed by the extrusion and then formed into the injection machine, authors reported high flame retardation and strength improvement after additions. However, other researchers have investigated properties derived from the addition of Fe_2O_3 , TiO_2 , CaCO_3 , ZnO and TiO_2 nanofillers⁵. Chae et al.⁶ examined the biological and thermal properties of highly concentration ferrous nanoparticles with PET, their results showed that nanoparticles ferrite played an important role in crystallization of PET polymer, due to heterogeneous nucleation. As a result, other properties were affected, such as the thermal stability of the matrix, heat permeability and low propagation of the decomposition products. Rupture energy and tensile strength of hybrid poly (methyl vinyl siloxane) fortified with the short PET fibers and wollastonite bristles were studied by Fu et al.⁷, in which authors observed an increase in strength with an increase in the wollastonite concentration, from 5 to 45 wt.%. The opposite effect is related to the change in the interface properties between the PET fibers and the matrix by the incorporation of the wollastonite bristles. However, the interfacial friction stress caused by the shrinkage of the matrix during the curing process may be causing the compound to be reinforced⁷.

Zn- Ferrite is one of the most important non organic complete, it is used in many applied material devices since 60 years ago. The general formula is $\text{Zn}_x\text{Fe}_{3-x}\text{O}_4$ which consists of Fe and Zn with oxygen atoms⁸. The Ferrite complex is prepared from reaction oxide of Iron and Zinc oxide with higher temperature. The spinal Zinc Ferrite is a sold material and non-Solute in water, acid and Alkaline and acid materials (9). Zinc ferrite has good higher reflection to use Dyes (experiments) in much more applicaions that have stability heating¹⁰. On the other hand, it was added to many paintings to produce forcing corrosion by environmental effect¹¹. Zinc ferrite has high electric resistant and has high dielectric properties and optical heat, the magnetic properties is good practice such as resistant of damage and good emission. The coefficient of heat extended hourly and has energy transfer with good efficiency. All these properties made Zinc Ferrite complex stable with different applications in technology material, sensor,

microwave, and for battery and magnetic mater¹²⁻¹⁵. Although the ceramic has higher solid electric penetration, and corrosion, it is usually active for electric and heating transfer and has high resistant for higher temperature compared with polymer¹⁶. The mechanical properties for ceramic material were very complex with ductile and easy to break under any strains compared with polymer which has light weight, lower density and elastic but usually tearing materials^{17, 18}.

However, the produced decomposition material designed for valise application resulted in enforcing the polymer by ceramic mater to get good dielectric properties and mechanical force. It was important in many electronic instruments needed in material stability and has higher dielectric with a good control in heat treatment to produce good surface and lower vacancies^{10, 11}. D.K. Das et.al¹⁸ mixed ceramics material with a higher dielectric constant (PZT) in polymer matrix to produce a composite material which has a large dielectric constant. Li – Zhang et.al¹⁹ prepared composite material produced in that phase from polymer and ceramic for a small microwave antenna, due to the good electro chemical and polarity properties. The permittivity of composite was increased from 3.45 to 11.87 whereas the $\tan\delta$ remained constant at 0.0016. Samara Jasim Mohammed²⁰ studied some dielectric properties for polymer – ceramic composite, (PZT)- (PVC) using solid solvent for preparing composite samples. It was noticed that the dielectric constant value decreased with ratio of Ceramic (PZT). The decreasing of dielectric is small at low ratios 15% and 30%, while it increased largely at 0.45% and 60% ratio for PZT. However, the tangent of angle $\tan\delta$ was calculated for polymer and composite, it has been shown that $\tan\delta$ increased with increasing the ratio of (PZT). Samara Jasi Mohammed et.al²¹ prepared micro polymer composite material adding Titian di oxide (TiO_2) with grain size (75-150) μm to epoxy rating for different concentrations. The strength of the fracture and hardness were studied for composites materiales and it was observed the increasing of the hardness with the wieght ratios of ceramics. Kesarwani et.al²² prepared composite material from $\text{CaCu}_3\text{Ti}_{14}\text{O}_{12}$ (CCTO) ceramic and PET polymer in different concentrations, the x-ray diffraction, dielectric constant and $\tan\delta$ were investigated. It was shown that adding 10% from PET to CCTO ceramic decreased ($\tan\delta$) to 0.0015. Teskmes et.al²³ fabricate a nano and micro composite material with large thermal conductivity. In this study, polyethylene terephthalate-based compounds were produced and characterized. This work is concerned with preparing samples of

Polyethylene terephthalate (PET) - zinc-ferrite (ZnFe_2O_4) with different addition ratios as zinc-ferrite (1, 2, 5, 10, 15 and 20) wt% obtained from mixing the solution with a hot pressing method applied under optimum conditions. The aim of this research is to enhance the properties of Polyethylene terephthalate (PET) by adding different materials.

Material Methods:

The experimental process starts with preparing the $\text{Zn Fe}_2\text{O}_4$ complex material by solid state reaction method²⁴. ZnO and Fe_2O_3 were used as elementary materials to prepare the ZnFe_2O_4 by weighting equivalent quantity in molaric ratio (1:1). Then, they were mixed with each other by adding acetone to give a good homogenous mixed material. The resulted homogenous material was heated in electric furnace to sintering in air at 1100 C° temperature for 2 hours and cooled to room temperature. The homogenous material produced Zn-ferrite phase using x-ray diffraction.

The PET polymer was dissolved in dichloromethane and then the zinc – ferrite was added to polymer in volume fractions (1, 2, 5, 10, 15 and 20) wt % and mixed well in a mortar. These composites were reformed by using a hot pressing method.

The density of the composites was measured by Archimedes Method¹². The weight of the dry samples was estimated by a sensitive balance and the samples were immersed in distilled water and boiled for (4 hr). The samples were immersed in water for (6 hr) and then studied. The weight of each sample was suspended in its submerged basket with distilled water by the sensitive balance. The samples were then dried and the water was removed from the surface of the samples. The samples were weighed directly by a sensitive balance after drying. This process must be completed quickly to reduce the error rate. The density of the composites has been calculated from the following relationship²⁵.

$$\rho_{\text{Bulk}} (\text{gm/cm}^3) = \frac{w_d}{w_s - w_n} \times D$$

Where: D is the density of distilled water (1gm/cm^3), w_d (gm) is the weight of the dry sample, w_n (gm) is the weight of the sample suspended and immersed in distilled water, and w_s (gm) is the weight of the sample in saturation condition with distilled water.

The structure of composite materials were investigated by x-ray diffraction with cu $\text{K}\alpha$ radiation source ($\lambda = 1.54056\text{\AA}$) for a scan range 2θ from (10° - 80°). Dielectric constant, dielectric loose tangent and Ac conductivity for composites were studied by using LCR meter type (8105G(120) 5M Hz, Taiwan, LRC) within the frequency range

(50 Hz-1MHz). The hardness shore D was measured to different samples using achiness shore device establishment.

Results and Discussion:

The densities of the composites were measured and calculated by Archimedes method. The spread of zinc – ferrite in the PET polymer led to a change in the density of the coposite. There are two reasons for changing the composite density; the first is due to the large molecular weight of ferrite compared with the polymer. The second reason is the large volum of PET particles. The reason for the discrepancy in the values of the densities is the additions create a dangling bonds that reduce the density of the composite in some samples (2 & 3%), and this is a result of the stability of the crystal structure²⁶, as shown in Fig. 1 and Table 1.

Table 1. Density of composite with Zn-Ferrite wt%

Zn- Ferrite wt%	Density of composite(gm/cm^3)
0	1.0802
1	1.1208
2	1.1064
5	1.112
10	1.182
15	1.23
20	1.286

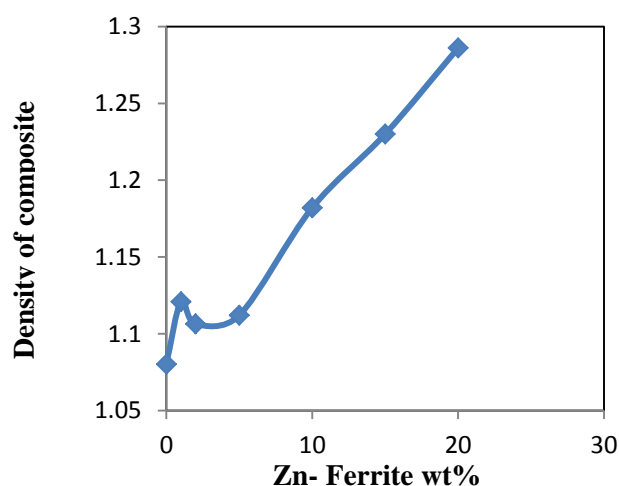


Figure 1. The densities of the PET/ Zn- ferrite as a function of addition Zn- Ferrite wt%

The structure of composite materials is investigated and illustrated in Fig. 2. The x-ray diffraction diagram for pure crystalline compound Zn-ferrite that the pattern of diffraction X-rays of the pure crystal Zn-ferrite includes 12 basic narrow peaks that are due to the crystalline behavior of the Zn-ferrite. Where the X-ray diffraction pattern indicates the presence of a crystalline phase with

one cubic structure with a space group $fd-3m$ (227)) ($a = b = c = 8.44 \text{ \AA}$, $V = 601.45 \text{ \AA}^3$), this was calculated using Brack's law²⁷.

As for Fig. 3, it shows the X-ray diffraction patterns of the pure polymer, where it shows that there are four broad peaks at ($2\theta = 16.46^\circ$, 17.45° , 22.72° , 25.98°). This indicates that the composition is polycrystalline and after its comparison with the primary sources, which showed the pattern is due to the pure polymer (PET), which promotes the amorphous behavior of the polymer. This is consistent with long-term structural disorder²⁸.

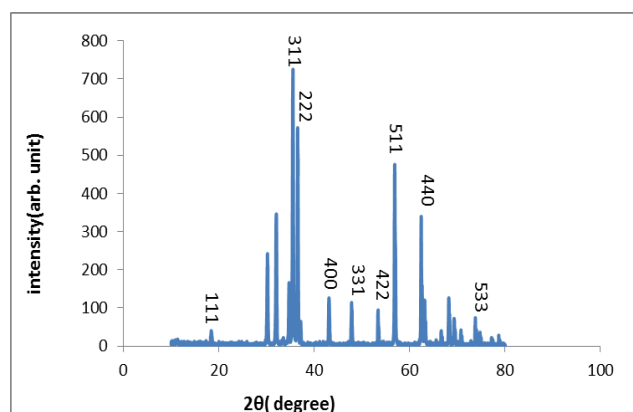


Figure 2. represents the x-ray diffraction of the pure crystalline compound of Zn- ferrite

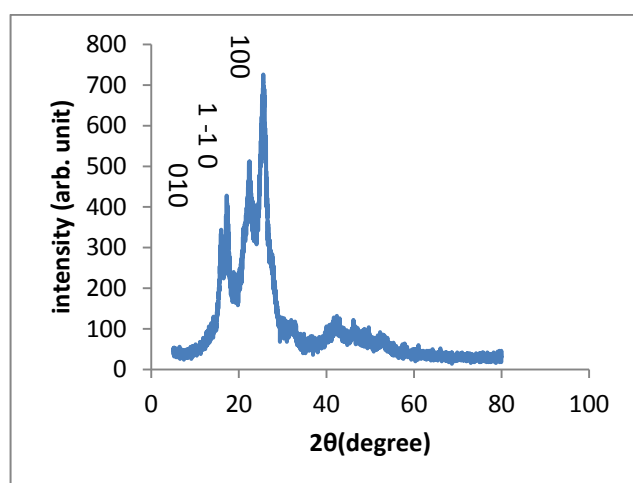


Figure 3. represents the x-ray diffraction of the pure polymer, where it shows that there are four broad peaks at ($2\theta = 16.46^\circ$, 17.45° , 22.72° , 25.98°) XRD of PET

All diffraction peaks in this paper are in good agreement with the data base comments card No-(00-022-1012)²¹ as well as diffraction patterns of x-ray ceramic– polymer composites (PET/ ZnFe_2O_4 with (1,2,5,10,15,20 wt%). There are differences in x-ray diffraction pattern of the pure compound, Zn-ferrite which also shows that the cubic structure of the compound Zn-ferrite is stable in the composite. However, we noted from the x-ray

diffraction diagram of the (PET/ ZnFe_2O_4) polymer –ceramic composite that there is no undesirable interaction and this is evident in Fig. 4 that the stronger peak occurs at the angle $2\theta=25.9^\circ$ of polymer (PET). The intensities of the diffraction peaks for the polymer (PET) are dispersing with the increasing of the ceramic Zn-Ferrite concentration. In addition to this, it is observed from Fig. 4 that there is a decrease in the intensity of the peaks and their width, this indicates that the increase in the crystal structure occurs whenever the percentage of ferrite increases²⁹.

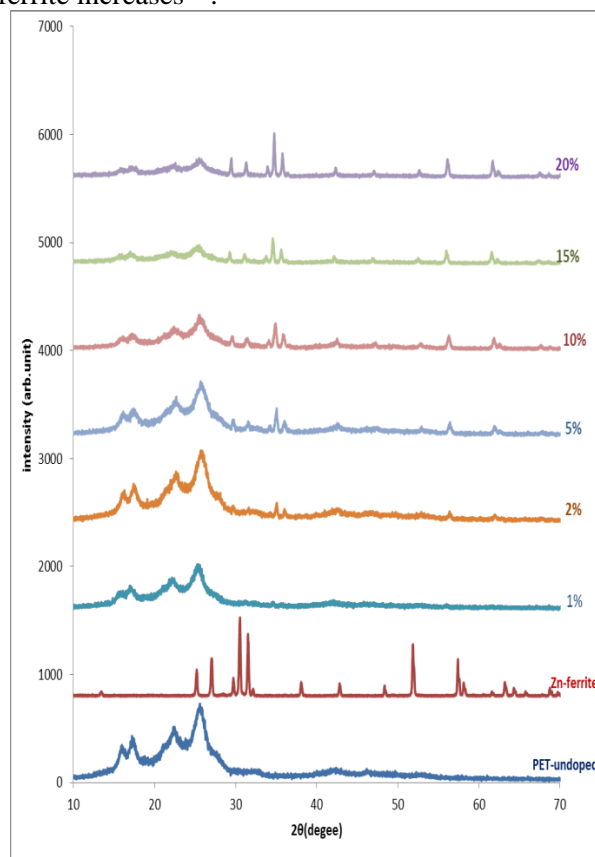


Figure 4. represents the x-ray diffraction of the polymer –ceramic composites

Analysis of Dielectric properties:

The variations in the real part (ϵ') and imaginary part (ϵ'') of dielectric constant for polymer-ceramic (PET/ ZnFe_2O_4) samples as a function of frequency at room temperature are shown in Figs. 5 and 6, it has been observed that a decrease in dielectric constant (ϵ') and loss (ϵ'') leads to decreasing frequency at room temperature without the effect of addition ZnFe_2O_4 . From these Figures, an increase in the dielectric constant (ϵ') and a loss (ϵ'') at low frequencies at 50000 Hz at room temperature are observed due to the lack of work of polarization mechanisms at these low frequencies. The dielectric constant (ϵ') and a loss (ϵ'') decrease sharply at the range of frequency over (500000 Hz). The real part (ϵ') of the dielectric

constant (ϵ') gives a part of the volume of energy stored inside the material when exposed to an electric field, and the most likely place to store this energy is within the grain sites (between the grains). The imaginary part of the dielectric constant (ϵ'') indicates the absorption and attenuation of energy across the interfaces under an external electrical field. Examples of interfaces are grain boundaries, localized defects and localized charge densities at the defect sites and at grain boundaries³⁰.

On the other hand, we can note from Fig. 7 that the lost tangent will decrease with increasing frequency. This is possible to be produced from the decrease in electrical conductivity of the composites with increasing frequency as shown in Fig. 8. Also, it has been noted that the frequency depends on the dielectric constant and ($\tan\delta$) and it increases with the increase of the Zn-Ferrite content. This increase in dielectric constant, ($\tan\delta$) and dielectric loss with increased Zn- Ferrite content explains the charging polarization at interface and the dipole polarization intrinsic with in composites in applied fields³⁰.

These phenomena appear in heterogeneous systems such as polymer compositions due to the accumulation of electrical charge at the interface of the boundary and the formation of large dipoles. Therefore, the degree of polarization with composite increases with increasing Zn-Ferrite content of ceramic and this enhances the dielectric constant of the composites. Varying the logarithm of Ac conductivity as a function of frequency for PET/

ZnFe₂O₄ is shown in Fig. 8. This Figure illustrates that the increase ceramic ZnFe₂O₄ produce increasing the AC conductivity of the composite due to the decreasing in the crystallization degree of the composites which is confirmed by x-ray diffraction analysis. The increasing of the non-crystallized regions will led to produce a faster polymer chains . This will make the movement of ions easy between chain so the ac conductivity of the (PET/ZnFe₂O₄) will increase. Fig. 8 shows that the AC conductivity decreases with increasing frequency for the PET/ ZnFe₂O₄, and the ratio of the weight (20 wt%) of the ceramic ZnFe₂O₄ indicates significant increase in the AC conductivity at high frequencies, which related to the relaxation (such as Deby) that occurs in such materials.

Hardness shore D as a function of Zn-ferrite concentration are shown in Fig.9 and Table 2, where it is noted from the Figure that the values of shore hardness D increase sharply from 68.7 to 74.8 when adding Zn - ferrite 0 to 15 wt%, but decrease gradually as the concentration of Zn - ferrite increases above 15 wt% until it becomes 72.8 at 20 wt%. This is because at weight ratio (15 wt %) has a is good diffuse of the Zn Fe₂ O₄ particles in the matrix (PET) polymer. These strangles affect the properties of the mechanical composites and the hardness. In the case of the (20 wt.%) ratio, the shore hardness D decreases the appearance of the clusters which reduces the mechanical properties.

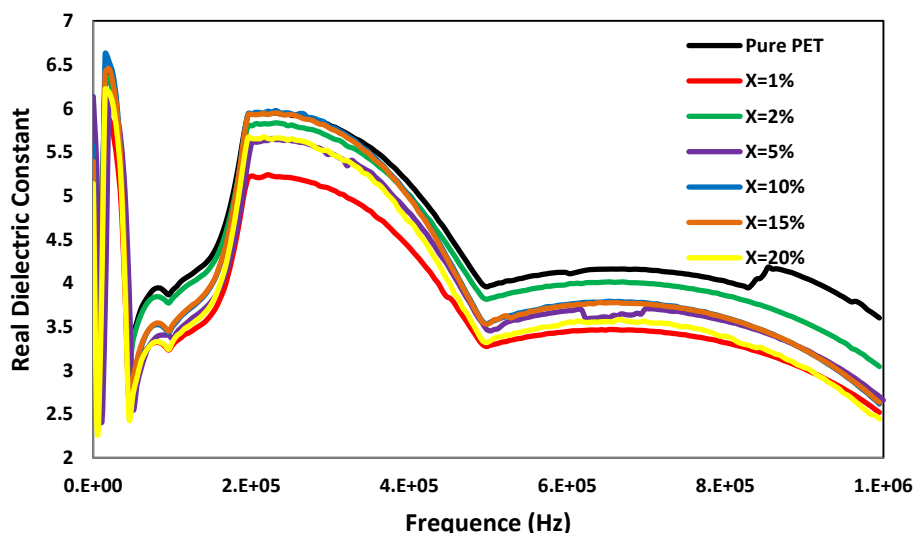


Figure 5. The variations in the real part (ϵ') of dielectric constant for polymer-ceramic (PET/ ZnFe₂O₄) samples as a function of frequency at room temperature

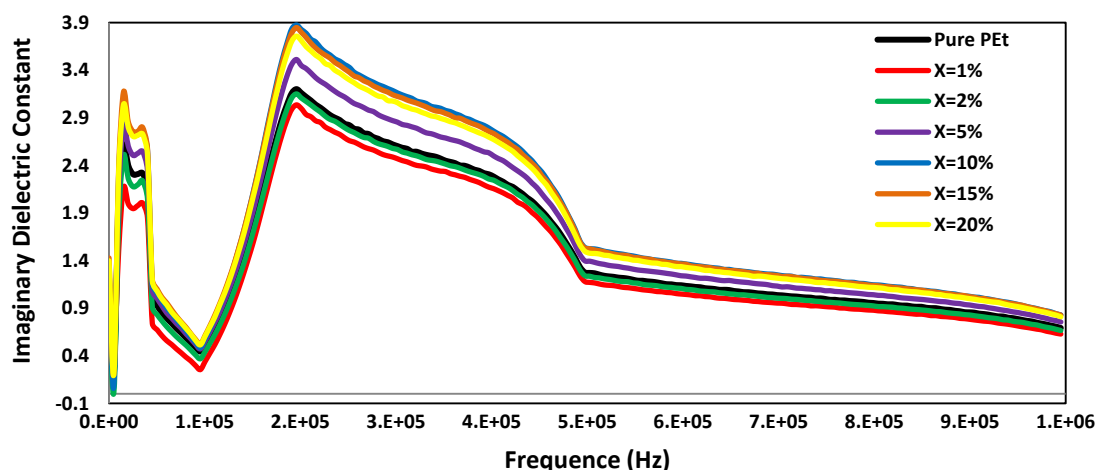


Figure 6. The imaginary part (ϵ'') of dielectric constant for polymer-ceramic (PET/ ZnFe_2O_4) samples as a function of frequency at room temperature

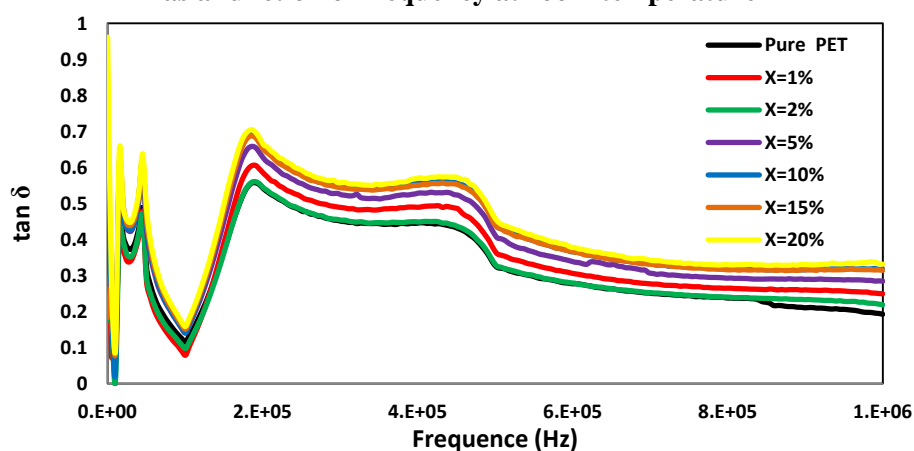


Figure 7. $\text{Tan } \delta$ vs. frequencies for polymer-ceramic (PET/ ZnFe_2O_4) samples as a function of frequency at room temperature

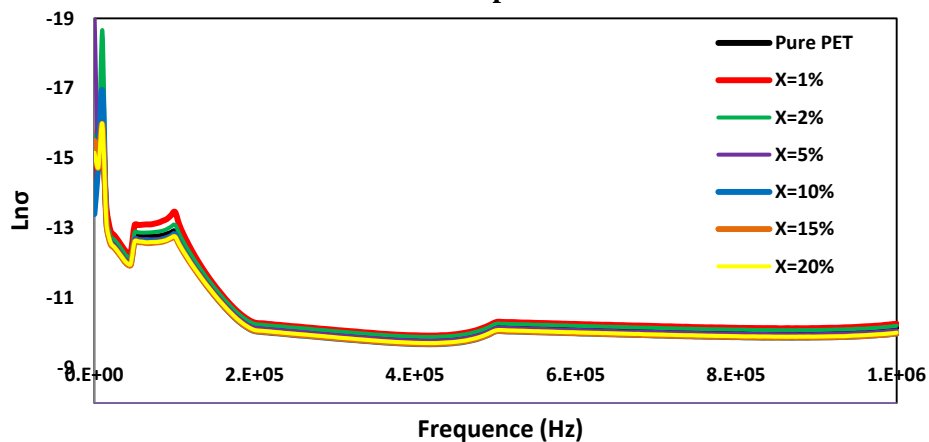


Figure 8. The A.C conductivity as a function of frequency at room temperature for polymer-ceramic (PET/ ZnFe_2O_4) samples

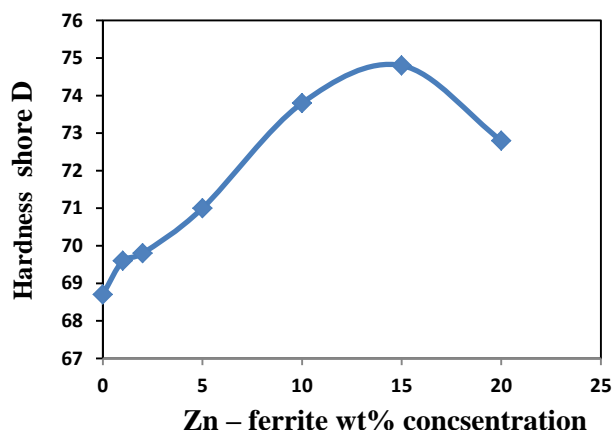


Figure 9. Hardness shore D as a function of Zn-ferrite concentration for PET/Zn- composites

Table 2. Hardness shore D of wt% composite with Zn – Ferrite wt%.

Zn – ferrite wt%	Hardness shore D of composites
0	68.7
1	69.6
2	69.8
5	71
10	73.8
15	74.8
20	72.8

Conclusions:

The effect of adding Zn-ferrite ceramics to the PET polymer to obtain the PET /ZnFe₂O₄ composite utilizing solid-state reaction has been investigated. The samples are mixed and compressed using a cold pressure method and then treated with heat. X-ray diffraction pattern from the Zn ceramics -Ferrite shows single-phase cubic structure. XRD polymer pattern (PET) demonstrates the presence of an Anorthic structure. Dielectric constant, tanδ dielectric loss and dielectric loss, increase with increasing zinc ferrite ceramic content. The AC conductivity of PET / ZnFe₂O₄ polymer ceramic compounds increases as the ceramic content of Zn-ferrite increases. The polymer ceramic composite PET / ZnFe₂O₄ shows that conductivity increases significantly with increasing frequency. Shore D hardness increases with increasing Zn-Ferrite ceramic content in the PET matrix. High dielectric constant and static dielectric properties make PET / ZnFe₂O₄ compounds very useful in industrial applications such as magnetic materials, gas sensors, catalysts, photocatalysts, and absorbent materials.

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Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Author's contributions statement:

The role of authors are:

- (Abbas K. Saadon) Conception, interpretation.
- (Auday H. Shaban) drafting the MS, revision & proofreading.
- (Kareem A. Jasim) design, acquisition of data, analysis.

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تأثير إضافة الفرايت على خواص البولوي إيثيلين تيريفثاليت

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² كلية العلوم، جامعة بغداد، بغداد، العراق.

الخلاصة:

يلعب الحشو دوراً رئيسياً في تحديد خصائص وسلوك المواد المترابكة للمنتج. في هذه الدراسة، تم تحضير ودراسة خصائص مترابكات PET المضاف اليه $ZnFe_2O_4$. العمل يتضمن اعداد عينات من البولوي إيثيلين تيريفثاليت (PET) / الزنك - الفريت ($ZnFe_2O_4$) بنسب إضافات مختلفة مثل الزنك - الفريت (1، 2، 5، 10، 15 و 20) وزن% عن طريق الحصول عليه من خلط المحلول بطريقه الضغط الساخن والمطابقة تحت الظروف المثلى. تم حساب كثافة جميع النماذج. تمت دراسة الخصائص التركيبية باستخدام نمط حيود الأشعة السينية XRD، وأظهرت نتائج تحليل حيود XRD أن هيكل PET النقي هو متعدد التبلور، هناك أربعة قمم عريضة عند $2\theta = 16.46^\circ, 17.45^\circ, 22.72^\circ, 25.98^\circ$ والمركب الخزفي ذات طور بلوري ذو بنية مكعبة مع ثوابت شبكيه وذات مجموعة فضائية $fd- (227) 3m (a=b=c=8.44 \text{ \AA})$ و $(V = 601.45 \text{ \AA}^3)$ أثناء إضافة الزنك-الفريت إلى PET بتركيزات مختلفة للحصول على مادة (PET / $ZnFe_2O_4$) انتج مواد تزداد فيها كثافة الشدة وتغير موقع القمم مع زيادة إضافة الزنك-الفريت، وتجدر الإشارة أنماط الحيود لـ (20 PET / $ZnFe_2O_4$) تشير إلى أنه لم يكن هناك اختلاف واضح في أنماط حيود Zn-ferrite النقي الذي أظهر أن التركيب البلوري يبقى ثابتاً في PET / $ZnFe_2O_4$. بالإضافة إلى ذلك، فإن الخصائص الكهربائية للمترابكات ممثلة بواسطة ثابت العزل الكهربائي، فقد تم قياس خسارة العزل الكهربائي وموصلية التيار المتناوب كدالة لمجموعة من الترددات (50 هرتز - 1 ميجاهرتز)، وأوضحت النتائج أن هذه الخصائص تزداد مع زيادة تركيز السيراميك المضاف. وتقل هذه الخواص بزيادة التردد بسبب التغير في آليات الاستقطاب، وقد ثبت أن هذه المواد المترابكة المحضرة تمتلك خصائص ذات استقرار جيد في نطاق واسع من الترددات، مما يجعل هذه التراكبات البوليمرية ذات استخدام واسع في العديد من التطبيقات. أما بالنسبة لقياسات صلادة Shore D، فقد تبين القياسات صلادة هذا المترابك تزداد مع زيادة تراكيز السيراميك.

الكلمات المفتاحية: التوصيليه المتناوبه، الخصائص العازله، صلادة، PET/ $ZnFe_2O_4$ ، شور د، حيود الاشعه السينيه.