

Structural, Electrical and Dielectric Properties of Ternary Hybrid PMMA Composite

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Abstract

This study aims to synthesis ternary hybrid composite PMMA polymer matrix with TiO_2 , Al_2O_3 and ZrO_2 as additives using casting technique. Range of weight fractions (84-92 wt %) of Poly (methyl methacrylate), fixed percentage (4%) of TiO_2 and range of weight fractions (4-12 wt %) of Al_2O_3 and ZrO_2 were used as started materials. Structure characterization was examined through XRD and AFM. Obtained micrograph revealed homogeneity structure and XRD pattern display present of crystals for ZrO_2 . Dielectric properties like dielectric constant were carried out for range (10^2 - 10^6) Hz frequency. The addition of Alumina filler exhibit slightly effect on values of dielectric constant for prepared ternary hybrid composite (PMMA- TiO_2 - Al_2O_3) compared with ZrO_2 for ternary hybrid composite (PMMA- TiO_2 - ZrO_2) which dielectric constant increase as concentration. Dissipation factor decrease with increasing frequency in composite of alumina and zirconia but in case the later yield good compatible. D.C electrical conductivity was conducted at temperature range (313-373) K. Markedly increasing of the D.C electrical conductivity for Zirconia filler respect to addition concentration and applied temperature.

Keywords: Ternary hybrid composite, TiO_2 , Al_2O_3 , ZrO_2 , XRD, AFM, Dielectric constant, D.C conductivity.

Introduction:

Electric insulators obviously must have a very low conductivity, or high resistivity, to prevent the flow of current. Insulators must also be able to withstand intense electric fields. For example, the electrons may have kinetic energies sufficient to ionize the atoms of the insulator, thereby creating free electrons and generating a current at high voltages [1]. The importance of polymers is mainly because polymers are still regarded as a cheap alternative material that is manufactured easily. The intensive use of polymer in broad use has led to the development of materials for specific applications namely composites [2]. Composites materials are usually a polymer matrix composite because of its availability and easy to manufacture low cost and good properties [3]. Recently polymer matrix-ceramic filler composites are receiving increased attention due to their interesting electrical and electronic properties. Integrated decoupling capacitors, angular acceleration accelerometers, acoustic emission sensors and electronic packaging are some potential applications [4]. A vigorous development of

polymer composite and extensive utilization of polymer materials in technology has led to the polymer composites [5]. The uses of composites as dielectric are becoming more popular, therefore the electrical properties of polymer matrix composites which reinforced of particles are very important. The dielectric behavior of polymer films is of considerable interest due to their applications for insulation, isolation and microelectronics [6]. There are many different polymers available depending upon the starting raw ingredients [7]. Poly (methyl methacrylate) PMMA swells and dissolves in many organic solvents; it also has poor resistance to many other chemicals on account of its easily hydrolyzed ester groups [8]. Titanium dioxide TiO_2 has been the subject of many studies due to its remarkable optical electronic properties, It is material used in a wide range of common and high-tech applications [9]. Alumina Al_2O_3 is one of the most widely used thermal oxides due to the high resistance to heat, mechanical durability and resistance to thermal shock, Excellent dielectric properties, and low dielectric constant and loss tangent [10]. Zirconia ZrO_2

is an oxide which has a high tensile strength, high hardness and corrosion resistance. Zirconia based ceramics are routinely used in structural applications in engineering, such as manufacture of cutting tools [11]. In this investigation we are prepared thick film ternary hybrid composite PMMA, and study their structure properties (XRD, AFM), electrical properties (D.C conductivity, D.C resistivity, activation energy) and dielectric properties (dielectric constant, loss tangent, A.C conductivity).

Experimental Work:

The composites were synthesis using casting techniques from different concentration solution. The raw materials were dried in a convection oven at 80°C to remove residual moisture. Range of weight fractions (84-92wt %) of PMMA was supplied from (Veracril, Colombia) dissolved in chloroform solution using hand-shaked for an hour, then fixed percentage (4%) of TiO₂ from (Fluka) and range of weight fractions (4-12 wt %) of Al₂O₃ obtained from (riedel-de haen company, German) were added gradually to prepare the solution and continuous stirring using (magnetic stirrer device) at temperature 50°C for about (3-4) hours to insure homogeneity, inhibits particles flocculation in the suspension. This process was repeated another once for started material powder (ZrO₂) supplied from (riedel-de haen company, German) with range of weight fractions (4-12 wt %). After the solution was transferred to clean mold. The dried film was then removed easily. The best concentration, for film production cast without bubbling and can be dismounted easily from the mold was found to be 10% , thickness average of sample was (0.263 - 0.340) mm.

Measurements:

Dielectric constant:

Prepared samples of diameter (25) mm were tested using programal LCR meter model (GW INSTRON, LCR-8105G, Precision LCR-Meter, 20 Hz-5 MHz, GPIB, RS-232,Taiwan). Parallel capacity C_p and loss tangent (tanδ) were carried out in range of frequencies (50Hz-1MHz) and voltage (10mV) at room temperature. For lowest error it was conducted

mask of aluminum electrodes deposited on two sides of sample with circular aperture 15mm as diameter. The evaporation process was started at a pressure of 10⁻⁵ Torr. The best condition for good ohmic contact was satisfied by a layer of 200 nm then was joined with copper wires by silver paste. Edwards's type, E306A unit was used for electrode preparation. The dielectric measurements of dielectric constant (ε'), dissipation factor tan δ and A.C electrical conductivity (σ_{a.c}) of composites were obtained from following equations:

$$\epsilon' = (1/\epsilon_0)(d/A)C \dots\dots\dots (1)$$

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

$$\sigma_{a.c} = \omega \epsilon_0 \epsilon'' \dots\dots\dots (3)$$

where:

- ε': dielectric constant
- ε'': dielectric loss factor
- tanδ: dielectric dissipation factor
- ω: angular frequency
- A: the effective cross- sectional area
- d: thickness of the sample
- ε₀: 8.85*10⁻¹² F/m

Measurement of D.C. Electrical

The D.C volume resistivity is measured in accordance with test methods ASTM D257. Electric conductivity measurements were measured by using Keithley 614 electrometer. The prepared sample of a dimensions (20*20) mm and diameter of electrode effective (15mm), for optimum measurements, it was carried out the electrode method as previously satisfied. Firstly, the measurement applied at room temperature and secondly, at temperature range (300-350)K, sample placed inside the Furnace (carbolite cwf 1200).

$$\rho_{d.c} = \frac{R A}{L} \dots\dots\dots (4)$$

$$\sigma_{d.c} = 1/\rho_v \dots\dots\dots (5)$$

Activation energy (E_a) for sample was calculated from the relationship between ln (σ) versus reciprocal T according to Arrhenius equation [8]

$$\sigma_e = \sigma_o \exp(-E_a / K_b T) \dots\dots\dots (6)$$

Where:

- ρ_{d.c}: volume resistivity

$\sigma_{d.c.}$: volume electrical conductivity

A: electrode effective area.

R: volume resistance (Ohm) .

L: average thickness of sample (m)

$A = D^2\pi/4$, D is diameter of electrode effective =0.015m

T: absolute temperature (K)

σ_0 : minimum electrical conductivity at (0K)

σ_e : electrical conductivity at temperature (T)

K_B : Boltzmann constant.

Atomic Force Microscopy (AFM) from nanosurf flex AFM was used to study morphology and measure a roughness of a sample surface at a high resolution, in addition, to perform a microfabrication of a sample. The phase structure of prepared composites were identified by powder X-ray diffraction technique using the diffractometer (XRD--6000/7000, SHIMADZU CORPORATION).

Results and Discussion:

The XRD pattern of (PMMA-TiO₂-Al₂O₃) composite (12% Al₂O₃) concentration was shown in Fig.(1). It is identifying the amorphous nature at 13.83° which indicated the PMMA corresponding to [Cart No. 13-0835], the other crystalline peaks at 66.9° and 27.4° coincide with (Al₂O₃) and (TiO₂) powders standard [Cart No. (10-0425 Al₂O₃) and (21-1276 TiO₂)] respectively which indicated polycrystalline nature. Furthermore, the crystalline of polymer PMMA has been considerably decreased upon the addition of fillers (TiO₂ and Al₂O₃) and the crystalline peaks of polymer broadens, which indicate to additive fillers changes the chain re-organisation and facilitates for higher ionic conduction in composite matrix. Fig.(2) observed (PMMA-TiO₂- ZrO₂) (12% ZrO₂) XRD pattern, the bands of PMMA together with the peaks of the zirconia 28.4° compared with [Cart No. (37-1484 ZrO₂)]. From the figures (1&2) it can be concluded the addition of fillers to PMMA may induce the significant increase in the amorphicity of composite materials. In case of Al₂O₃ filler the growing of peak polymer broadness more than in type ZrO₂ as additive which may deduce the more decreases in particle size of Al₂O₃ and

homogenously dispersed throughout the polymer matrix (PMMA).

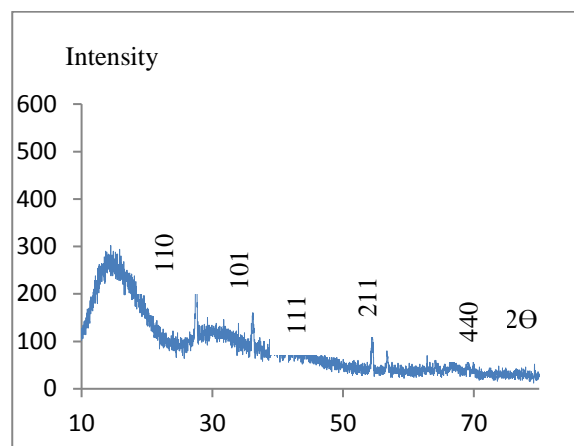


Fig.(1):XRD patterns of (PMMA-TiO₂-Al₂O₃) composite.

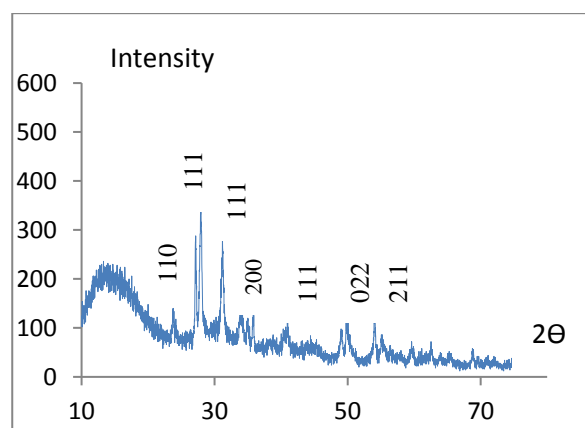
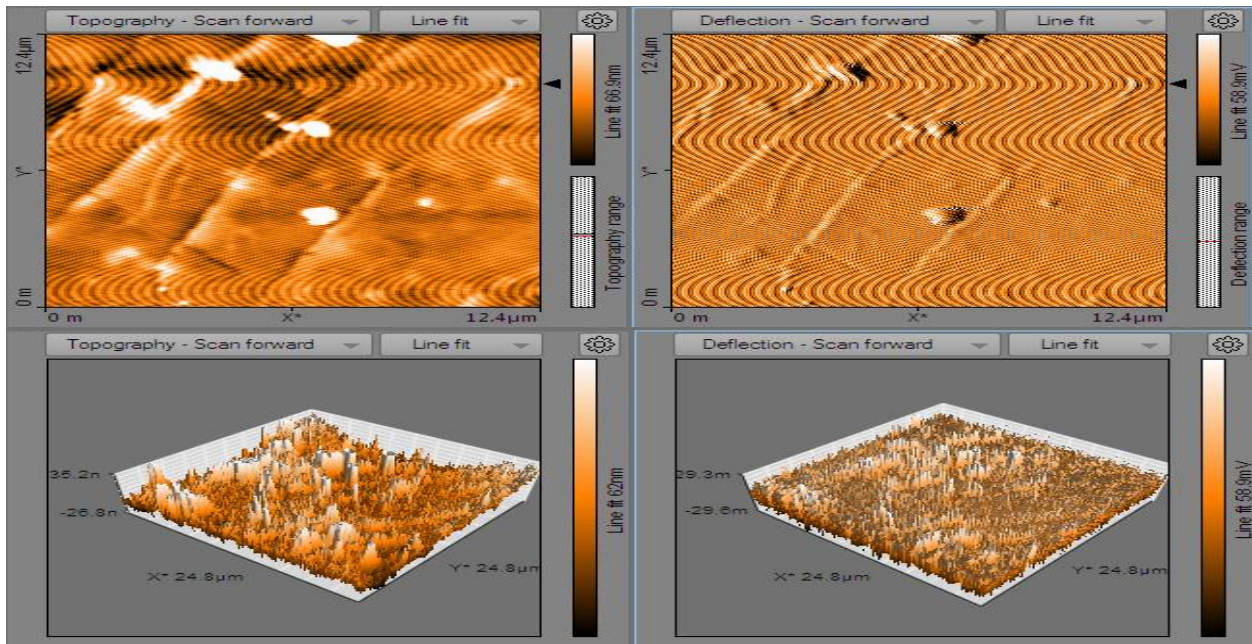


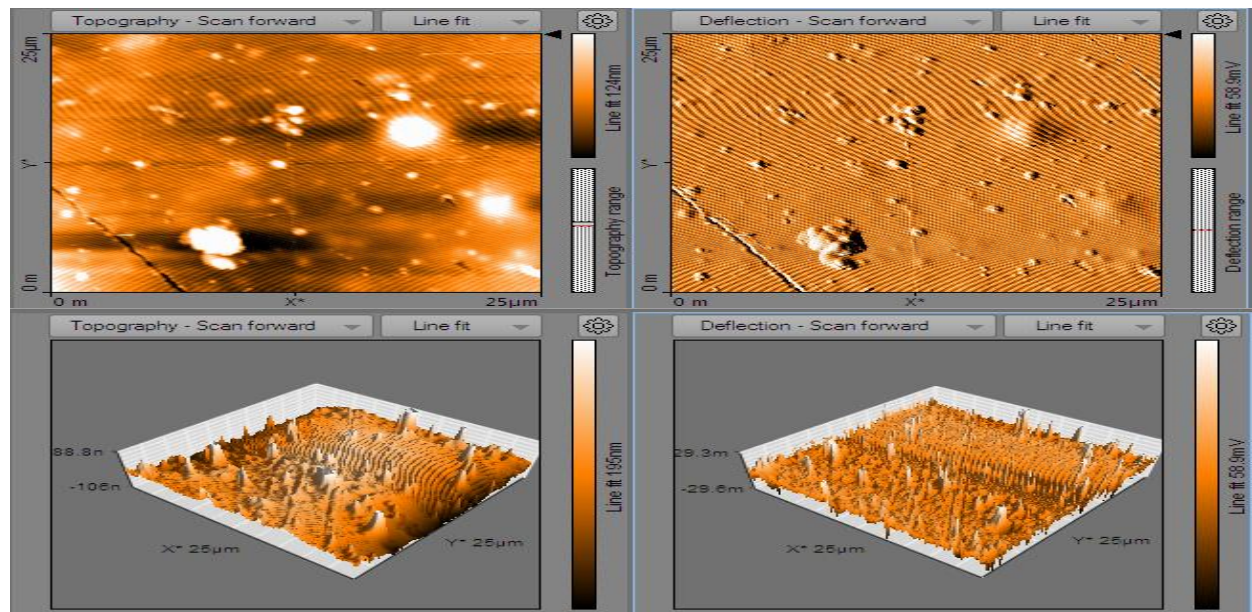
Fig. (2):XRD patterns of (PMMA-TiO₂-ZrO₂) composite.

Atomic force microscopy (AFM) is shown in Fig.(3) for (PMMA-TiO₂-Al₂O₃) (12% Al₂O₃) prepared composite .The root-mean-square (RMS) roughness (15.32 nm) and mean grain size (79nm), revealed the composite possess smooth surface, homogenous phase, dense structure, no cracks formation and fine particles (inorganic fillers) are well dispersed in the PMMA matrix.

In Fig.(4) the micrograph for (PMMA-TiO₂- ZrO₂) (12% ZrO₂) composite. The root-mean-square (RMS) roughness (28.87 nm) and mean grain size (111nm). It can be seen the porous structure which appeared as (dark spots) of polymer composite in corporation of ZrO₂ in to the PMMA matrix. From figures (3&4) we can say the alumina filler give smoother and homogenous compared with zirconium addition in PMMA composite.



**Fig.(3): a:AFM micrographs for (PMMA-TiO₂-Al₂O₃) composite in 2-dimaiton
b : AFM micrographs for (PMMA-TiO₂-Al₂O₃) composite in 3-dimaiton.**



**Fig.(4): a:AFM micrographs for (PMMA-TiO₂-ZrO₂) composite in 2-dimaiton
b : AFM micrographs for (PMMA-TiO₂-ZrO₂) composite in 3-dimaiton.**

Fig.(5) shows the dielectric constant of Ternary hybrid (PMMA, TiO₂, Al₂O₃) Composite as a function frequency for range (10²-10⁶) Hz at room temperature, It had been seen from results of dielectric constant of (PMMA, TiO₂, Al₂O₃) composite was decreased with increasing frequency. This is because of that at lower frequencies of applied voltage, the free dipolar functional groups in the PMMA chain can orient themselves resulting in a higher dielectric constant value at these frequencies and may be attributed to

the electrical relaxation processes. Also the high value of dielectric constant at lower frequency might be due to the electrode effect and interfacial effects of the sample. As the electric field frequency increases, the bigger dipolar groups find it difficult to orient at the same pace as the alternating field, so the contributions of these dipolar groups to the dielectric constant goes on reducing resulting in a continuously decreasing dielectric constant of the PMMA system at higher frequencies [12].

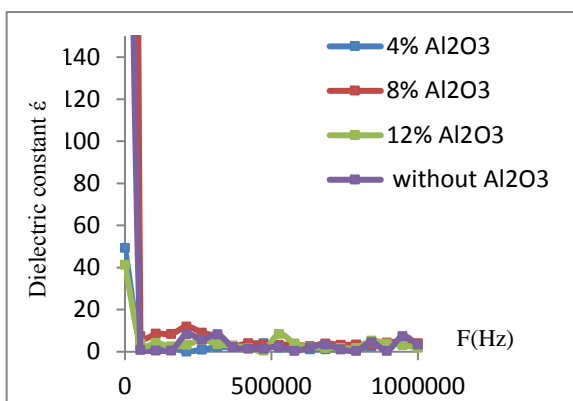


Fig.(5): The Dielectric constant with frequency of (PMMA-TiO₂-Al₂O₃) composite.

From Fig. (5) we can notice at 1MHz the dielectric constant is (3.408) for sample (without Al₂O₃) concentration (PMMA-TiO₂) is higher than that of other concentrations, which ratiocinated the results of dielectric constant give slightly effect with additive filler Al₂O₃, may by attributed to the high value of dielectric constant (85) for TiO₂ compared with dielectric constant (9.6) for alumina [13].

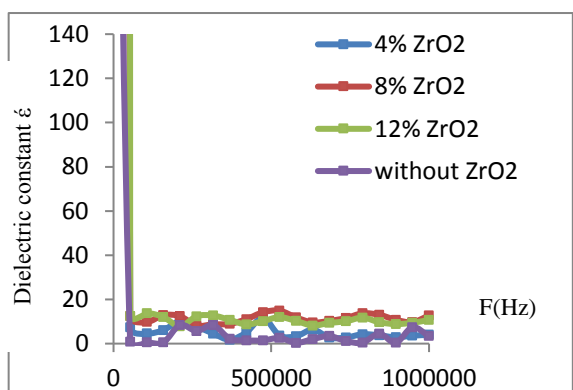


Fig. (6): The Dielectric constant with frequency of (PMMA-TiO₂-ZrO₂) composite.

The dielectric constant of materials is due to the electronic, ionic, dipolar and surface charge polarizations which depend on the frequencies. It is observed from Fig.(6) the dielectric constant of (PMMA - TiO₂ - ZrO₂) prepared composite decreased with increasing frequency at range (10²-10⁶)Hz at room temperature. The large value of dielectric constant at lower frequency might be due space charge polarization arising at interfacial. The decrease of dielectric constant with increasing frequency is the expected behavior in most dielectric materials this is due to dielectric relaxation involves the orientation polarization which in turn depends upon the

molecular arrangement of dielectric to be material. So, at higher frequencies, the rotational motion of the polar molecules of dielectric is not sufficiently rapid for the attainment of equilibrium with the field, hence dielectric constant seems to be decreasing with increasing frequency [14]. Furthermore the dielectric constant values increases with increasing weight fractions of ZrO₂ as additive might be reflected to an increased in number of dipoles per unit volume which mean increase in the number polarized dipoles., i.e, space charge polarization contribution reflects the formation of capacitance network of ZrO₂.

Fig. (7) and Fig. (8) show loss tangent with applied frequency at range (10²-10⁶) Hz at room temperature of (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites. The reason for the decrease in dispersion factor (tanδ) with increasing frequency due to the dipoles absorb energy of the electric field in order to overcome the resistance of the viscous material (matrix) that surrounds them during rotation. This absorbed energy reduces the transition charge carriers between the edges of capacitor with increasing of the frequency and dipoles which need high energy in order to get relaxation and this cause a decreased of dispersion factor (tanδ).

From the figures we notice at high frequency the dispersion factor (tanδ) for sample of (without filler) is high that might be get relaxation for all dipoles by absorb of part of the energy while the other part is used for the transfer of charge carriers[14]. Also may get the case of resonance when consensus applying electric field frequency with the natural frequency of dipoles which gets high loss.

From Figs. (7) and (8), we can observed the decreasing of dissipation factor at 1MHz for prepared hybrid ternary composites (PMMA-TiO₂-Al₂O₃) with (8%Al₂O₃) was (0.821) and for (12%Al₂O₃) was (0.784), and prepared composite (PMMA-TiO₂-ZrO₂) ternary hybrid (8% ZrO₂) have dissipation factor (0.397) and for (12% ZrO₂) was (0.0608) at same frequency. It can be explained to good interfacial adhesion between zirconia, alumina and titania fillers and molecules chains of PMMA polymer matrix, which it is reflect improvement the chemical bond and

interaction between the matrix and fillers as reinforcement.

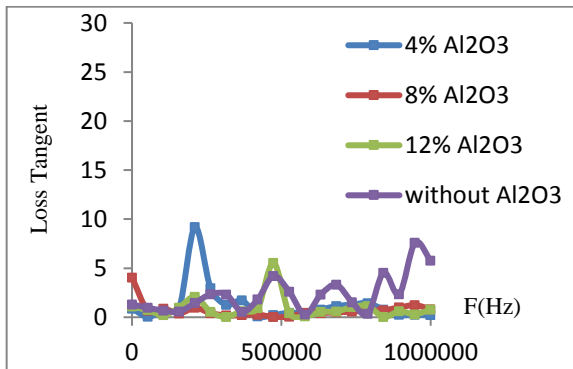


Fig.(7): loss tangent with frequency of (PMMA-TiO₂-Al₂O₃) composite.

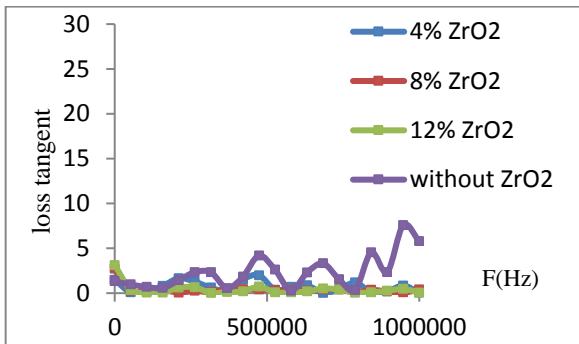


Fig.(8): loss tangent with frequency (PMMA-TiO₂-ZrO₂) composite.

In Fig.(7) at frequency (200000) Hz it noticed “loss peak” for (8% Al₂O₃) explained as the thickness increment may result in more localization of charge carriers along with mobile ions causing higher conductivity. This may be the reason for higher (ϵ') and strong low frequency dispersion. So, we notice at low frequency the dielectric loss of (8%) concentration is high because it have high thickness. For sample (12% Al₂O₃) concentration of (PMMA-TiO₂-Al₂O₃) has loss peak present at (473736) Hz frequency revealed relaxation processes which usually occur in heterogeneous system. The increasing of Al₂O₃ concentration increases the height of the peak and increasing its broadness for these specimens. This is due to the overlapping of relaxation process which is attributed to some structural changes that take place in the composite as result of filler addition. On the other hand, at low frequency, the dielectric loss is considerably higher, due to the interfacial polarization effect.

Fig.(9) and fig.(10) show the electrical conductivity (A.C) with frequency of (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites at room temperature. It is observed from Figures that, the electrical conductivity increase with increase frequency as observed from eq.(3). Also, we noticed the $\sigma_{a.c}$ increasing in high frequency region cab be related to the electronic polarization as well as to the hopping of charge carrier over a small barrier height. Furthermore the electrical $\sigma_{a.c}$ increasing as concentration increase ,the reason behind this lead to addition the filler is the appearance of charges between the interfaces surfaces (between filling material and incubator material PMMA) which increases with increasing the filler content to a maximum value with increasing frequency.

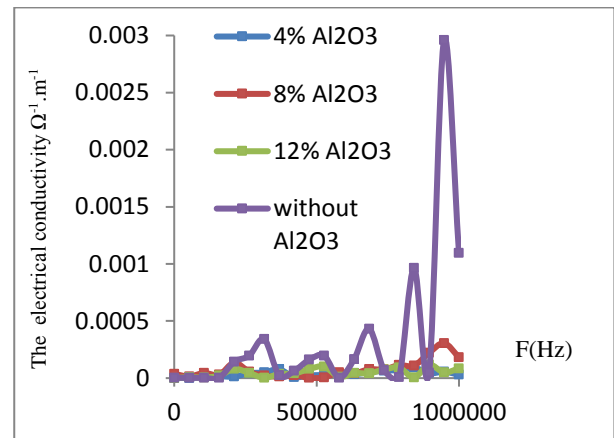


Fig.(9): The electrical conductivity (A.C) with frequency of (PMMA-TiO₂-Al₂O₃) composite.

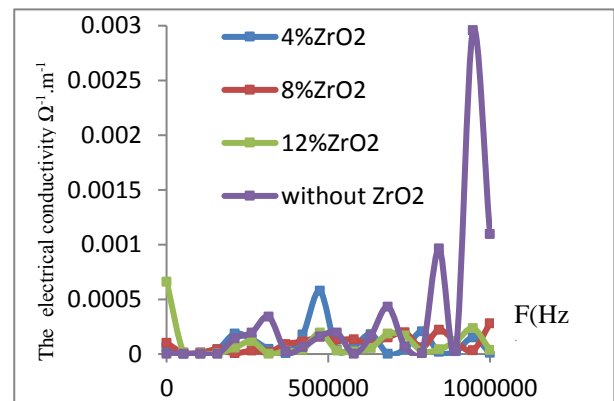


Fig.(10): The electrical conductivity (A.C) with frequency (PMMA-TiO₂-ZrO₂) composite.

From eq.(3) it can see there are direct relation between the A.C conductivity $\sigma_{a.c}$ and

loss tangent ($\tan\delta$). So, it can be noticed that for prepared composite ((PMMA-TiO₂) have high $\sigma_{a.c}$, this leads to smaller resonance as previously mentioned.

Figs. (11) and (12) show the values of D.C electrical conductivity in temperature range (313- 373)K of (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites.

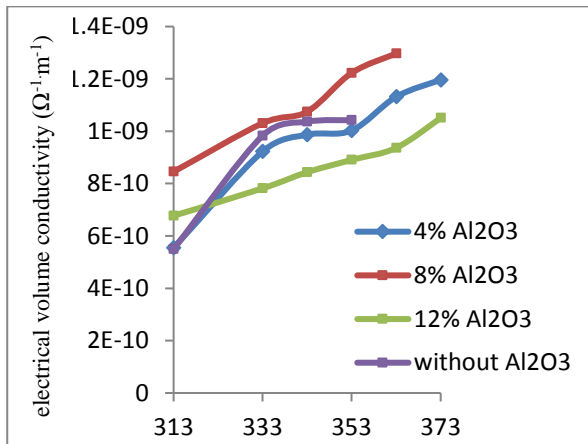


Fig.(11): electrical volume conductivity (DC) with temperature of (PMMA-TiO₂-Al₂O₃) composite.

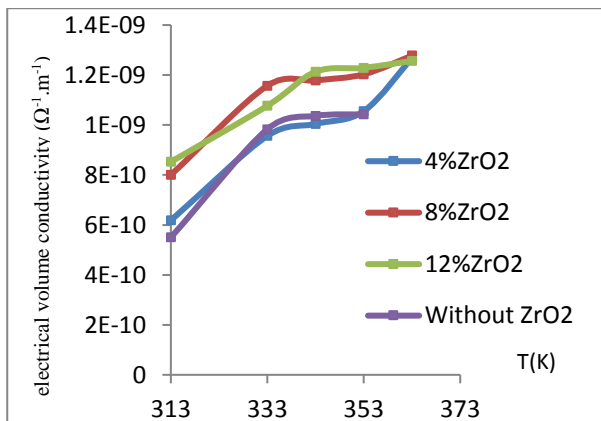


Fig.(12): electrical volume conductivity (DC) with temperature of (PMMA-TiO₂-ZrO₂) composite.

Note that the electrical conductivity increases with increasing temperature that any of this material has a negative thermal coefficient of resistance. The interpretation of this is that the polymeric chains and alumina particles act as traps for the charge carriers which transit by hopping process. On increasing the temperature, segments of the polymer begin to move, releasing the trapped charges. The release of trapped charges is intimately associated with molecular motion. The increase of current with temperature is

attributed to two main parameters: charge carriers and mobility of these charges. The increase of temperature will increase the number of charge carriers exponentially. The mobility depends on the structure and the temperature. We notice in Fig.(11) the electrical conductivity of concentration (12%) is low, that is due to the low thickness. While for (8% Al₂O₃) there may be impurity which represents (defects), thus $\sigma_{d.c}$ increases rapidly.

Figs. (13) and (14) show the values of D.C electrical volume resistivity in temperature range (313- 373)K of (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites, it is observed that the volume resistivity decreases with increasing the concentration of filler content and heat. The electrical resistivity is inverse to the electrical conductivity.

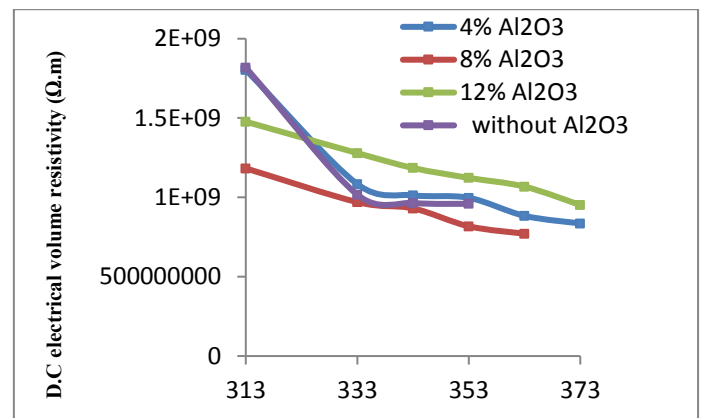


Fig.(13): D.C electrical volume resistivity with temperature of (PMMA-TiO₂-Al₂O₃) composite.

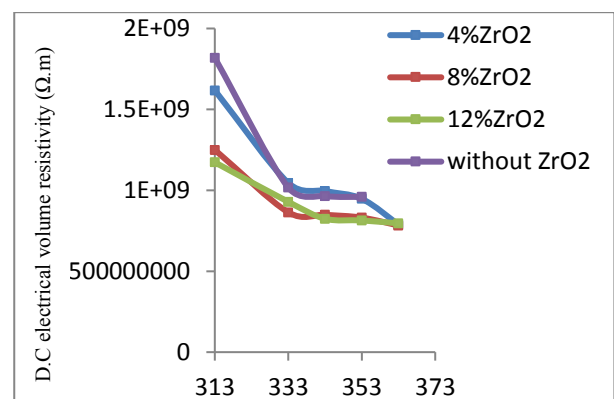


Fig.(14): D.C electrical volume resistivity with temperature of (PMMA-TiO₂-ZrO₂) composite.

The high increase of $\sigma_{d.c}$ for about (353)K reflects the glass transition temperature (T_g) for PMMA, this is because after T_g free volume increases and main chain of polymer starts moving, which makes the movement of

molecular chains easy and then, they take place in conduction process with increase temperature.

Figs. (15) and (16) show D.C electrical volume conductivity with concentration wt% for (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites. The increase of conductivity with increasing of concentration of filler due to the increase of ionic charge carries created from the filler [15]. This mechanism is called “quantum mechanical tunneling” effect [16]. Where the electron can move through the insulator between conductive elements (filler) with a certain probability. i.e., electrons can hop from conductor to conductor by “tunneling” through the insulating barrier.

Markedly increasing in electrical conductivity for prepared hybrid composite (PMMA-TiO₂- ZrO₂) ,this may be attributed to open structure which is shown in Fig.(4),in contrast the dense structure lead to decrease the electrical conductivity specially at (12%) as seen in Fig.(15) and Fig.(3) for Al₂O₃ filler.

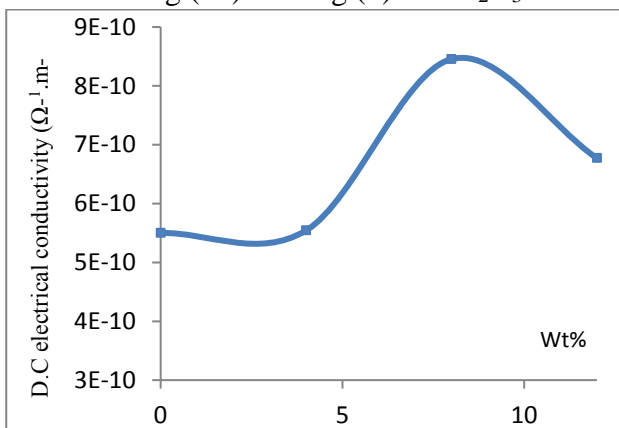


Fig.(15): D.C electrical conductivity with Al₂O₃ wt% concentration for (PMMA-TiO₂-Al₂O₃) composite.

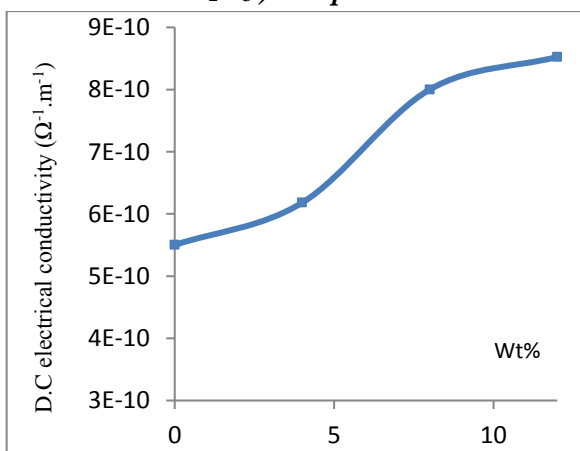


Fig.(16): D.C electrical conductivity with ZrO₂wt% concentration for (PMMA-TiO₂-ZrO₂) composite.

The Figs. (17) and (18) show The relationship between $\ln(\sigma)$ and $(1/T)$ for (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) composites. The activation energies were calculated from eq (6), the results which observed from figures are caused by hopping and the band conduction phenomena. The conduction mechanism can be attributed to hopping between traps in localized state close to the Fermi level.

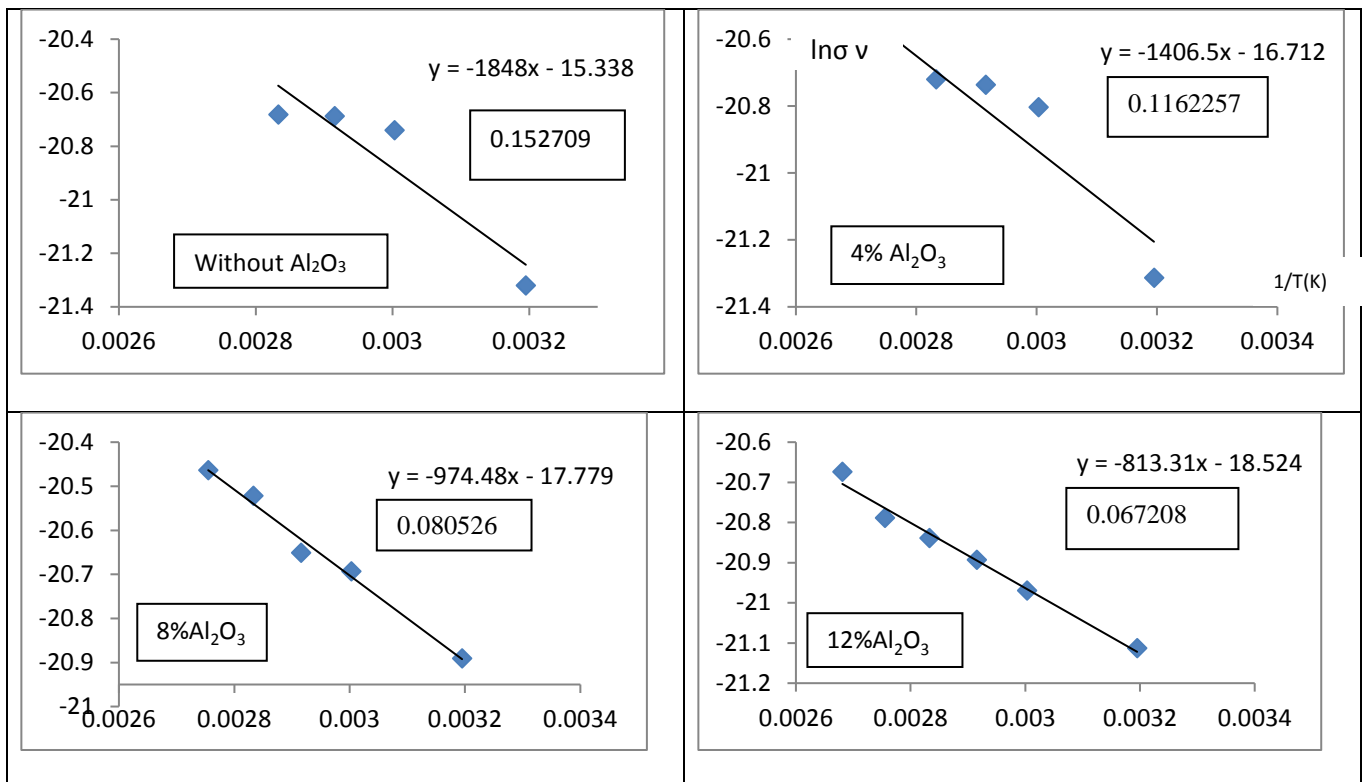


Fig.(17): The relation between $\ln \sigma$ and the inverse absolute temperature for (PMMA-TiO₂-Al₂O₃) Composites.

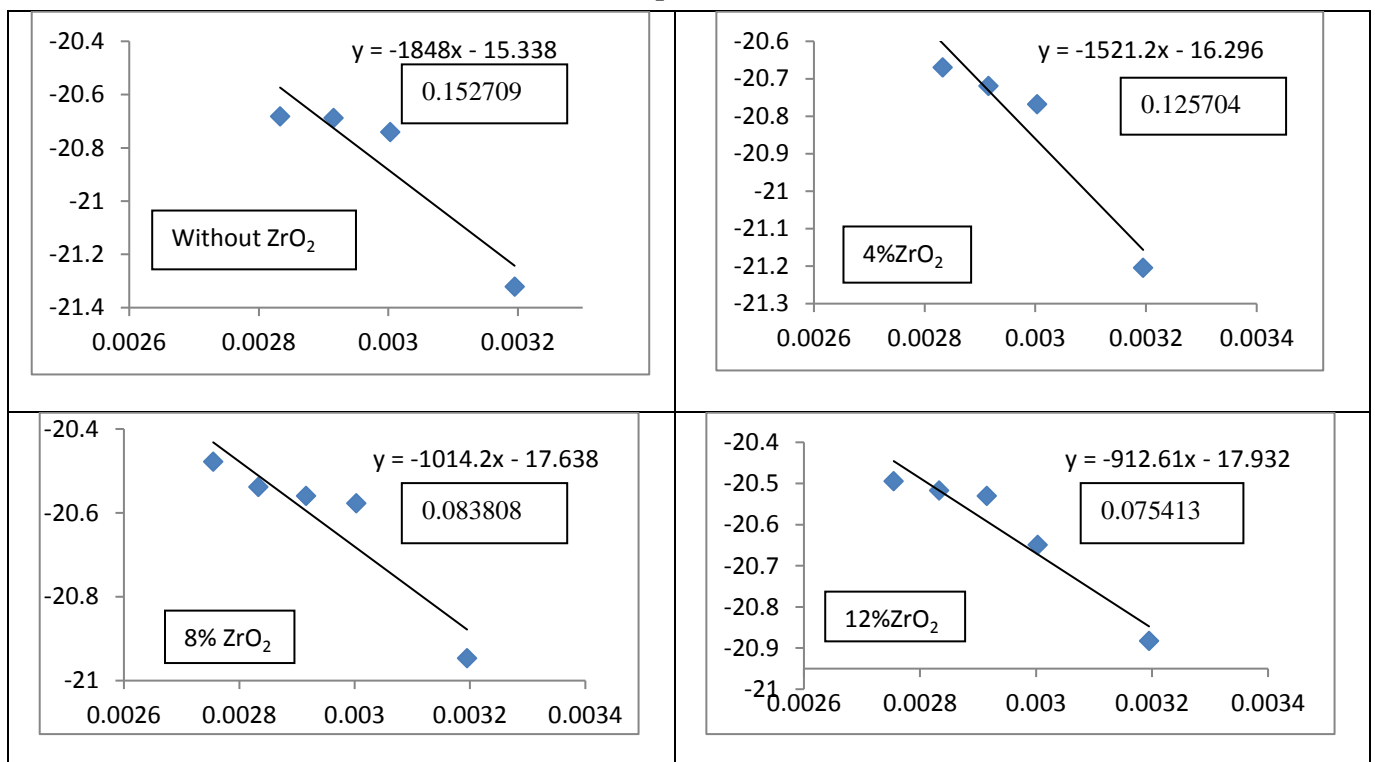


Fig.(18): The relation between $\ln \sigma$ and the inverse absolute temperature for (PMMA-TiO₂-ZrO₂) Composites.

Conclusion:

Ternary hybrid composites (PMMA-TiO₂-Al₂O₃) and (PMMA-TiO₂-ZrO₂) were prepared by casting method. The addition of Alumina filler exhibit slightly effect on prepared ternary hybrid composite (PMMA-

TiO₂-Al₂O₃) for it is values of dielectric constant compared with ZrO₂ for ternary hybrid composite (PMMA-TiO₂-ZrO₂), where ϵ increase as concentration. Dissipation factor decrease with frequency in both composites of Alumina and Zirconia but in case the later

there are good compability. Markedly increasing of the D.C electrical conductivity for Zirconia filler respect to addition concentration and applied temperature. Micrograph AFM revealed homogeneity structure and present of crystals as shown in XRD pattern for ZrO₂.

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الخلاصة:

يهدف هذا البحث الى تحضير متراكبات ثلاثية هجينة من PMMA مقواة بجسيمات TiO₂-Al₂O₃- ZrO₂ بنقنية الصب. كسور وزنية (84-92 wt %) من بولي ميثيل ميثا اكريلك ونسبة ثابتة (4%) من تيتانيا مع نسب وزنية تتراوح بين (4-12 wt %) للالومينا والزركونيوم. تم دراسة المواصفات التركيبية من خلال حيود الاشعة السينية ومجهر القوة الذرية. تبين نتائج الصور المجهرية تجانس في البناء البلوري كما تظهر قيم الحيود وجود البلورات الزركونيوم في المتراكب المحضر الهجيني. الخواص العزلية فحصت من خلال قياس ثابت العزل عند مدى تردد (10²-10⁶) Hz ان النتائج تظهر تاثير طفيف عند اضافة الالومينا للمتراكب الهجيني (PMMA-TiO₂-Al₂O₃) مقارنة بمضاف الزركونيوم للمتراكب الهجيني (PMMA-TiO₂- ZrO₂) حيث قيم ثابت العزل تزداد مع التركيز. في حين يتناقص عامل الفقد بزيادة التردد. قيست التوصيلية الكهربائية المستمرة لمدى درجات حرارية تتراوح بين (313 - 373)K هناك زيادة ملحوظة في القيم التوصيلية بزيادة التركيز ودرجة الحرارة في حالة الزركونيوم المضاف للمتراكب الهجيني.