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Studying the Effect of Temperature on the Electrical Properties of Poly (methyl methacrylate) Doped with Lithium chloride

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Abstract

This article explores the electrical properties of a polymer polymethyl methacrylate (PMMA) doped with lithium chloride (LiCl). In general due to their low electrical conductivity, polymers are common for used as insulating materials. PMMA is one of the polymers that have been widely used in electrical and insulating applications due to its distinguished electrical, dielectric, and optical properties, especially after being impregnated with some materials. One of the key objectives of this study is to develop electrical devices using enhanced polymeric materials. The experimental procedure involved preparing thin film samples using the casting method under normal conditions. PMMA polymer was doped with a 0.2% weight ratio of LiCl. Thin film samples were prepared using the casting method under normal laboratory conditions. The samples were thermally treated at different temperatures then treated samples included within an RCL load circuit connected in parallel. Our investigation focused on evaluating the resistance, capacitance, impedance, and dielectric constant of the PMMA/LiCl material using a load circuit operating at low frequencies and comparing these values with values obtained of pure polymer. The obtained results demonstrated significant enhancements in the electrical properties of the polymer, particularly in terms of charge storage at specific temperatures.

Keywords: Casting technique, Dielectric, Doped polymers, Electrical properties, glass transition temperature, PMMA/LiCl..

Introduction

Polymer technology has developed rapidly due to the distinct physical and mechanical properties of these materials and because of the high possibility of modifying and controlling these properties and the urgent need for alternatives with technological properties different from traditional materials, so it has been used in a wide range of fields starting from children's toys to the manufacture of car and aircraft structures [1–4].Thin polymeric films were used after they were developed in the manufacture of capacitors, and these materials entered among the semiconductor materials, where some polymeric insulating materials are converted into conductive materials by adding some impurities, and the polymer can become a superconducting material at not very low temperatures [5–8].

Polymers are characterized by their low electrical conductivity, so it was common for them to be used as insulating materials. Research on the electrical insulating properties of polymers is one of the important studies to know the molecular behavior

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and movement associated with phase change as a result of changing both motion and pressure within certain frequencies [9,10].

Polymers have been used in many electrical applications. They are often used as insulating materials because they have good electrical properties such as volumetric conductivity, volumetric resistance, impedance, and dielectric loss. These achievements in polymers have been achieved through wide and deep study, due to their importance in many industrial applications. The electrical properties of insulating materials used in different sizes in many devices depend on the type of use; and these characteristics are affected by many factors, such as temperature, time, frequency, additives, etc. [11,12].

The wide uses of polymers in technological fields, especially in their use in electronic devices, made them of special importance, as this importance emerged as a clear fact that polymers generally show different changes in their electrical and insulating behavior when doped or mixed. Despite this importance, there are still some engineering applied problems for polymers, such as their lack of connections and lack of strength compared to some metals, so several methods were used to improve the properties of polymers, like fiber reinforcement [13,14].

The methods for measuring insulation properties vary across different frequency ranges. There are two types of technical measurement methods, the first is called the “Distributed circuit method”, and it is usually used at high frequencies, where at these frequencies the positive length of the electromagnetic wave is close to the dimensions of the model, so the model becomes a transparent medium for the wave and then depends on the dielectric constant (έ) and the dielectric loss (ε) of the model based on measuring the optical constants as the absorption coefficient and refractive index. The second technique in which the dielectric properties are measured at low frequencies is called a “Lumped circuit” and it involves representing the polymer with a basic electrical circuit consisting of a capacitor and a resistance connected in series or parallel [15,16].

Experimental and theoretical studies have proven that the representation of the polymer with a resistance and a capacitor connected in parallel is appropriate at low frequencies.

The motivation for the present work is to improve the electric properties of poly-methyl-methacrylate (PMMA) by doping with lithium chloride and thermal annealing processing. These processes can increase the polymer chains' mobility and create more pathways for ion transport. This study uses the casting method under normal conditions. The electrical behavior of the polymer has been calibrated by means of an electrical circuit consisting of a resistance (R_p) and a capacitor (C_p) connected in parallel using the lamped circuit. Parallel capacitor (C_p), resistance (R_p), total impedance (Z), real (ε') and imaginary (ε'') permittivity and loss factor tan(δ) were measured at a fixed frequency of 1KHz. The results were interpreted according to the effect of the molecular behavior of the doped polymer on the electrical properties of the circuit.

Theoretical Considerations

The transfer of charges in insulating materials has a great importance in recent times because it provides general information about the electronic structure of these materials. Many studies have been conducted on electrical properties and many models have been developed for this purpose to understand the mechanism of electrical conduction. There are various mechanisms by which charge carriers can move in an insulator by the influence of an applied field [17–19]. Insulators differ from conductive and semiconducting materials in that the conduction beams are almost devoid of free electrons (or may contain free electrons, but these electrons are located within a conduction beam that separates one from the other insulating regions devoid of electrons). So, the electric current cannot flow when an electric field is applied, because the movement of electrons cannot exceed a few molecules of matter. Despite the limited movement of electrons and the lack of current in the material, this restricted mobility is of great importance in locating the insulating properties of the matter [20,21].

As the voltage applied to the insulating material changes with time, an alternating current arises. Consequently, this insulating material can be represented by a load circuit where the impedance is

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caused by a parallel combination of resistance and capacitance, representing the nature of the material. Insulator molecules can either be polarized particles with a permanent dipole moment or non-polar molecules. In the case of polarized particles, the negative charges within these molecules are attracted to the positive charges at their centers [22]. However, when these molecules are exposed to an external electric field, the positive charges shift toward the field while the negative charges shift in the opposite direction. The orientations of these particles are initially random, but when placed within the influence of an external electric field, they align themselves with the field direction.

The equation for impedance in an AC circuit containing resistance and capacitance is given by [23]:

$$1/Z = 1/R_p + j\omega C_p \cdots\cdots\cdots 1$$

where, Z represents the impedance of the parallel RC circuit. The term $j = \sqrt{-1}$ is the imaginary unit, and ω represents the angular frequency.

The capacity of materials can be described using the following relationship:

$$\varepsilon' = C_p/C_0 \cdots\cdots\cdots\cdots 2$$

where C_o represents the capacitance of a capacitor with air between its poles, and ε' denotes dielectric constant.

The dielectric loss is a measure of the energy dissipated as heat in the electromagnetic field. Its inverse value represents the quality factor (Q) for electrical charge storage. The dielectric loss can be determined by the following relationship [24]:

$$\varepsilon'' = \frac{1}{R_p C_0 \omega} \cdots\cdots\cdots 3$$

In Eq.3, R_p is the parallel resistance, C_o is the capacity of the capacitor with air between its poles, and ω represents the angular frequency.

The dielectric loss tangent ($\tan\delta$) is governed by the following relationship [24]:

$$tan\,\delta = \varepsilon'/\varepsilon'' \cdots\cdots\cdots 4$$

The AC-conductivity in general is a measure of the heat generated as a result of rotating dipoles, and depends on the frequency (ω) [25]: $\sigma = \omega\varepsilon_0\varepsilon \cdots\cdots\cdots\cdots 5$

where ε_o the permittivity of space and ε the permittivity of the medium. And at low frequencies it consists of two components [26]: $\sigma = \sigma_{ac} + \sigma_{dc} \cdots\cdots\cdots 6$

where σ_{dc} is the conductivity of direct current, which does not change with time, and σ_{ac} is the conductivity of alternating current, which expresses the dielectric loss.

The frequency of the electric field affects on the loss factor. At lower frequencies, the dipoles have enough time to orient themselves in the direction of the electric field, and when the frequencies increase, the electric field is reversed very quickly. The diodes cannot be completely oriented with the field and so, the dipole oscillations and the values of the dielectric steadily decrease [27,28].

Materials and Methods

The dielectric properties were measured by measuring the capacitance and resistance of the samples using an RLC motor type PM6036.

i) Materials

1. Poly (methyl methacrylate): It is a flexible and transparent material with a high refractive index, the chemical formula $(C_5H_8O_2)_n$, has a melting point of 213 Celsius, a molecular weight of 40000 gm/mol, and a density of 1.2 gm/cm^3.

2. Dimethylformamide (DMF): Solvent with chemical formula C_3H_7NO, molecular weight 73.10 (gm/mol), boiling point 153 °C, and the density is 0.9445 (gm/cm^3).

3. Lithium chloride: It is a typical ionic compound (Li^+Cl^-), in the form of white crystalline hydrates and has the following properties: molar mass 42.394 g/mol, melting point 605 °C, boiling point 1,382 °C, and the density is 2.07 g/cm³.

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ii) Synthesis and Characterizations

A powder of PMMA was doped with lithium chloride in different weight ratios (0.1% wt. - 0.5%wt.). An amount of 0.5g of the produced mixture was dissolved in 5 ml of DMF solvent. The solution was stirred well using a magnetic stirrer at laboratory temperature for 24 hours, and this mixing continued until it reached a high degree of solubility [29]. To remove some insoluble parts from the polymer solution, the product was filtered using a Bochner funnel. All previous steps were repeated for each weight ratio, respectively.

Aluminium substrates (slides) were washed with acetone and distilled water to ensure they were free of any suspended impurities. The slides were placed on a completely horizontal surface to ensure the homogeneity of the films. The prepared solution was poured onto these slides using the casting method and left to dry for two days. Then, the twenty-one films prepared of PMMA/LiCl were put in an oven at a temperature of 30°C, and then increased by 10 degrees every half an hour, up to 70°C, to remove the moisture and evaporate the solvent residues. Afterward, a gradual cooling process began until it reached laboratory temperature. We found experimentally that the degree of homogeneity of the film depends on the temperature and viscosity of the polymer.

As electrodes were deposited on them using an evaporation device under a low pressure of 10^{-4} Torr, most of the samples (except those with 0.2% wt.) were damaged. Therefore, we limited our study to samples with a doping of 0.2% wt. After the deposition of the electrodes, the prepared films were connected to the device to measure their dielectric properties. The device consisted of an RLC circuit, consisting of a capacitor connected in parallel or series with variable resistance and a coil in a shielded box, operating at a fixed frequency of 1 KHz. The differential scanning calorimeter (DSC) has been used to determine glass transition temperature (T_g). This technique is important for understanding the characteristics of polymer as a function of temperature, because polymers do not behave like conventional solids or liquids when their temperature is raised or lowered.

Results and Discussion

In this work, we present and discuss the results obtained from the experimental investigation of PMMA doped with lithium chloride (LiCl) within a temperature range of 298-333°K. The electrical conductivity of the doped samples was calculated using Eq. 5 and Eq. 6, resulting in average values of $8.14\times10^{-11} S/cm$ for pure samples and $1\times10^{-10} S/cm$ for doped samples.

The relationship between electrical capacitance and temperature is illustrated in Fig. 1. It was observed that as the temperature increased from 303 to 333°K, there was a rapid increase in capacitance values, reaching a peak at 333°K, followed by a subsequent decrease with a further temperature increase up to 343°K. This is consistent with a study by *Pandian Mannu et al* in ternary In–Te–Se nanocomposite thin films; they observed that the capacitance increases as temperature increases within the temperature range of 100 to 300 K [30].

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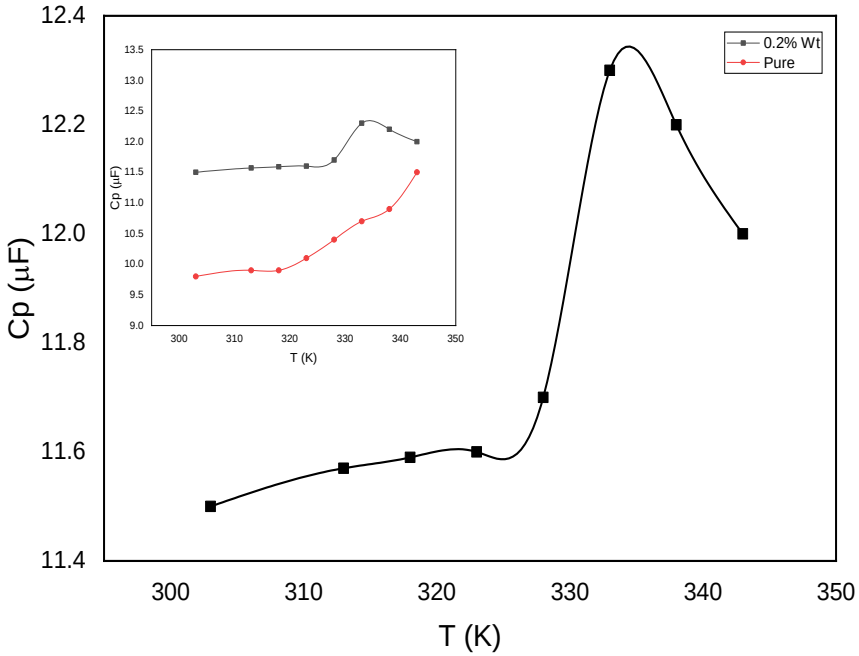

Figure1. Electrical capacity (µF) versus temperature T (K) of /LiCl

This behavior can be attributed to the transition of the doped polymer into an elastic state, allowing greater freedom of movement for dipoles and resulting in the storage of a larger number of electrical charges.

However, temperatures higher than the peak temperature led to the reorientation of dipoles in a manner that countered the effect of thermal excitation, transforming the polymer into an amorphous state and subsequently reducing the capacitor value.

The parallel resistance, depicted in Fig. 2(a), exhibited an inverse relationship with temperature within the same temperature range. Specifically, the series resistance at 333◦K reached a value of 16 MΩ, reflecting a decrease of 52 MΩ compared to the temperature of 303◦K.

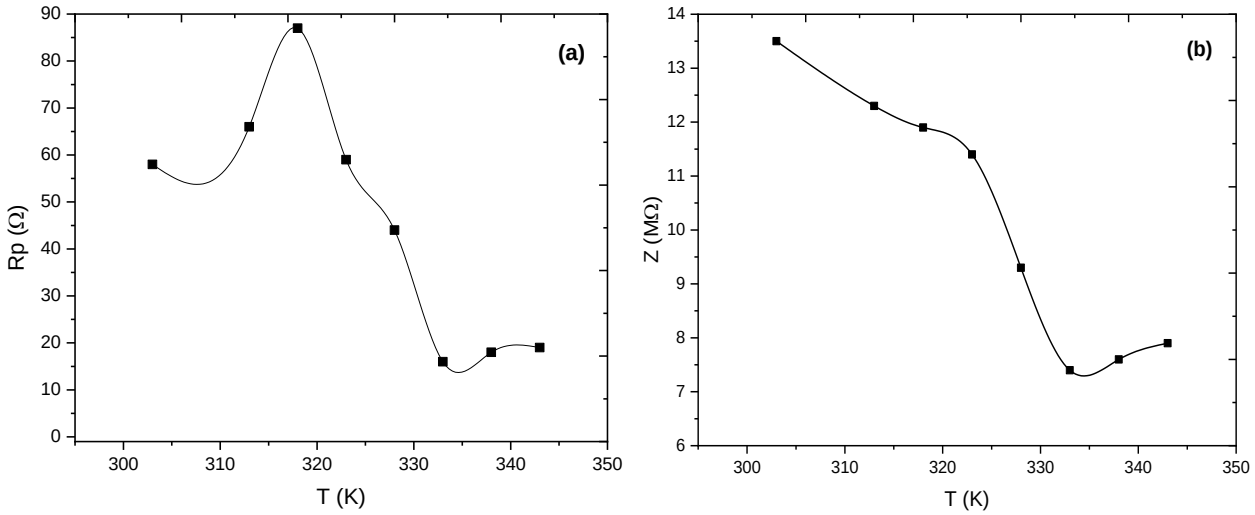

Figure2. (a) Resistance Rp (Ω) and (b) impedance Z(MΩ) versus temperature T (K) of PMMA/LiCl

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This behavior is similar to the observed trend in impedance (Z), as demonstrated in Fig. 2(b). Previous studies by *Basavaraja and Anita* on PMMA films doped with Zirconium dioxide have also shown an increase in impedance with increasing film thickness and a decrease with increasing frequency[28].

The behavior of the dielectric constant, follows a similar pattern to the electrical capacitance with temperature, see Fig. 3(a). The dielectric constant increased with temperature, particularly at low frequencies. This behavior can be attributed to the enhanced movement of dipoles within the molecular chain of the polymer at higher temperatures. The increase in polymer size allows for improved dipole response to the applied electric field, resulting in greater mobility and rotation, and subsequently an increase in polarization and dielectric constant. A study by *Sabbar et al* on polymer blend films (PMMA:PVC:PS) has shown that the real dielectric constant increases with increasing wavelength at certain temperatures but behaves differently at higher temperatures[27].

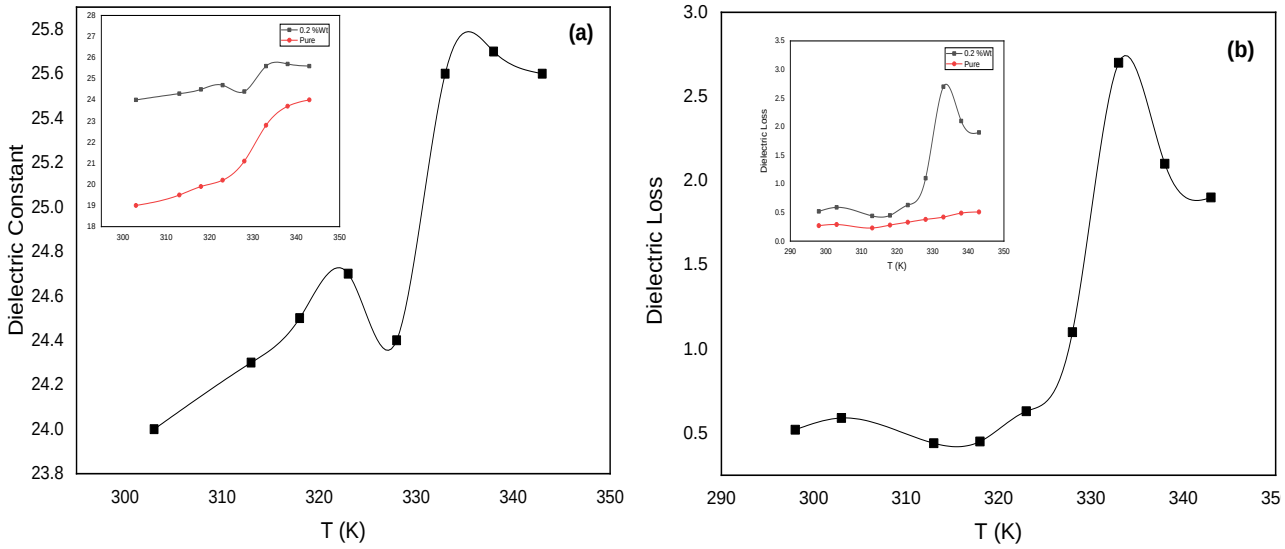

Figure3. (a) Dielectric constant ε' and (b) Dielectric loss ε" as a functions of temperature T(K) for PMMA/LiCl

Moreover, it was observed that the dielectric constant of PMMA increased with increasing film thickness. At lower temperatures, the dipole behavior can be considered as bound in insulating materials, making it challenging for the electric field to induce changes in dipole position[31].

The relationship between dielectric loss (ε") and temperature (see Fig. 3(b)) evident the dielectric loss increases with higher temperatures, which can be attributed to the significant electrical conductivity, especially at elevated temperatures. The increased mobility of charge carriers in polar polymers accounts for this behavior. Notably, two peaks are visible in the figure: the first peak (α-type) at higher temperatures corresponds to the movement of Brownian chains, while the second peak (β-relaxation) in the low-temperature range is likely associated with the rotation of polar groups within the polymer's main chain[32]. Similar observations were reported by *Akram et al.* in their study on polyester resin and polymer composites[33]. It is noteworthy that the dielectric loss in polymeric materials is affected by many factors such as their ionic conductivity and structure, whether they are homogeneous or heterogeneous. In the event that the polymeric structure contains defects or is porous, it will affect the movement of charges in the event of an electric potential, which leads to a dispersion of energy [34,35].

The results of this study indicate that the annealing temperature contributes to raising the value of dielectric loss tangent. The dielectric loss tangent behavior as a function of temperature is illustrated in Fig. 4.

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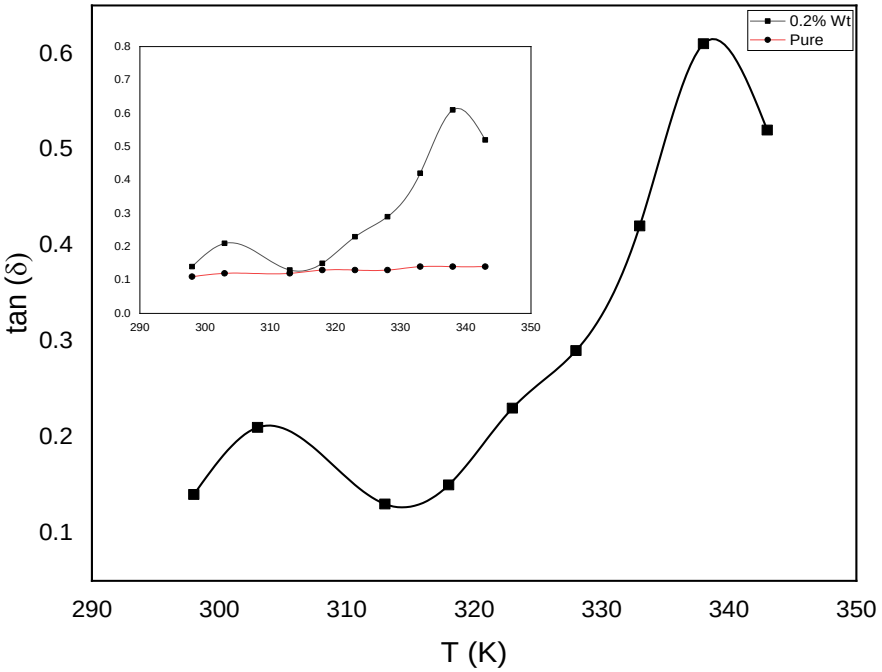

Figure4. Dielectric loss tangent (tan δ) versus temperature T(K) of PMMA doped with lithium chloride 0.2%wt

The highest value of the dielectric loss tangent, which signifies the dissipation of electrical energy, was observed at a temperature of 338°K, reaching 0.61. This represents a significant increase compared to its value of 0.47 at 298°K. This is consistent with the findings of *Al-jammal et al.*, who conducted a study on the electrical properties of PMMA under direct and alternating electrical fields at various temperatures [36]. This behavior can be attributed to the combined effect of ionic jump, loss of the ionic conduction, and loss of electronic polarization during the relaxation process.

Lastly, the glass transition temperature was determined by the differential scanning calorimeter (DSC). For reinforced PMMA with 0.2%wt lithium chloride, the temperature was 109 °C, compared to 105 °C for pure PMMA. This finding indicates the presence of bonding between the polymeric chain and the impurity particles, which hinders the movement of the polymeric chains and thus requires more energy to reach the rubbery state. Our result is consistent with Xiao Yuan Chen et al., who studied the thermal properties of PMMA. They reported an increase in the glass transition temperature from 92.4 °C for pure PMMA to 99.2 °C for PMMA doped with graphene oxide at 0.25 wt%[37].

Conclusion

In conclusion, the experimental results revealed that the PMMA/LiCl material exhibited increased values of dielectric constant, dielectric loss, and tangent loss compared to the pure polymer within the temperature range of 298-333◦K. As the temperature increased, the arrangement of dipoles became more random due to thermal vibration, resulting in a decrease in the dielectric constant. Notably, within the temperature range of 318◦K to 333◦K, the PMMA/LiCl material demonstrated promising potential for efficient electric charge storage. The optimal temperature range for effective performance was observed to be around 323-333◦K. These findings contribute to our understanding of the electrical behavior of PMMA/LiCl and its potential applications. The physical properties of the polymer change with variations in the doping ratio. A different molecular weight can be obtained by adding impurities; for this

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property, the polymer can be used in different industrial applications.

The glass transition temperature value increased for the doped polymer. This increase suggests the existence of bonding between the polymer and the impurity, which hinders the movement of the polymeric chains and thus requires more energy to reach the glass transition temperature.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- The author has signed an animal welfare statement.
- Authors sign on ethical consideration's approval.
- Ethical Clearance: The project was approved by the local ethical committee at University of Al-Muthanna.

Authors' Contribution Statement

A.N. Sabbar contributed to writing the manuscript, drawing figures, and analyzing the results. L.M. Rasheed contributed to preparing and testing samples, writing, and analyzing the results.

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دراسة تأثير درجة الحرارة على الخصائص الكهربائية لمادة بوليمر بولي ميثيل ميثاكريلات المشوب بكلوريد الليثيوم

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الخلاصة

تهدف هذه المقالة الى تحليل الخصائص الكهربائية لمادة ال بولي مثيل ميثا اكريلات (PMMA) المشوبة بكلوريد الليثيوم (LiCl). بسبب توصيليتها الكهربائية الرديئة تستخدم المواد البوليمرية بصورة شائعة كعوازل. يعتبر بولي مثيل ميثا اكرالايت من البوليمرات التي كثر استخدامها في التطبيقات الكهربائية والعازلة وذلك بسب امتلاكها خواص كهربائية وعازليه وبصرية متميزة وخاصة بعد تشوبها ببعض المواد. في هذه الدراسة، تم تشويب البوليمر PMMA بنسبة وزنية 0.2% من كلوريد الليثيوم (LiCl). تم تحضير عينات بشكل اغشية رقيقة باستخدام طريقة الصب في الظروف الاعتيادية للمختبر، ثم معالجة العينات حراريا ولمدى من درجات الحرارة. تم قياس كل من مقاومة التوازي وسعة التوازي والمانعة الكلية بالإضافة الى قياس ثابت العزل بجزيئة الحقيقي والخيالي ومقارنتها بخواص البوليمر النقي. تم استخدام دائرة تحميلية عند ترددات واطئة حيث تم تضمين العينات البوليمرية المعالجة حراريا بدائرة حمل RCL مربوطة على التوازي. اضهرت النتائج تغيراً في هذه البارامترات وخاصة في خزن الشحنات في درجات حرارة معينة وبالتالي زيادة في تحسين خواصها الكهربائية مما يؤهلها في استخدامها في كثير من الأجهزة الإلكترونية ضمن نطاق درجات الحرارة المستخدمة.

الكلمات المفتاحية: تقنية الصب، العزل الكهربائي، البوليمرات المشوبة، الخصائص الكهربائية، درجة حرارة التزجج، PMMA/LiCl.