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Study Effect of Heat Treatment and Pressure on Structural and Physical Properties for Nano polycarbonate polymer

A Thesis

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By

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1440 A.H.

بسم الله الرَّحمن الرَّحيم

" وَيَسْأَلُونَكَ عَنِ الرُّوحِ قُلِ الرُّوحُ مِنْ أمرتي وَمَا أُوتِيْتُمْ مِنَ الْعِلْمِ إِلَّا قَلِيلًا "

صَدَق الله الْعَظِيْم

سورة الإسراء الايه (85)

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Committee Certification

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إلى من رضا الله من رضاهها إلى من ترافقني دعواتهها الى من فرشوا ايام عمرهها طريقا لأيصالي .. الى من سهروا فصبروا طلبا لامالي .. الى من اضائوا لي عمرهما لينيرو لي كل الأزمان .. الى من اطوقاني بالحب والحنان ليمحوا كلمة الحرمان .. الى من لولاهما لما كنت هذا الانسان .. الى من منحتني الأمل الما كنت هذا الانسان .. الى من منحتني الأمل الذي ينير لي الطريق ...زوجتي الى من علمني الأصرار والنجاح إلى من أحبني فأحببته ... (اخوتي) الى من ساندي وشد ازري (مشرفتي) إلى الذين أحببتهم وأحبوني صدقاً ... أصدقائي

والى كل قلب ينبض بالحب من أجلى أهدي ثهره جهدي الهتواضع

محهد

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Abstract:

In this study, we studied the structural properties, which included Xray diffraction and electrical properties, which included: ac-conductivity (σ_{ac}), real dielectric constant (ϵ') and loss tangent of dielectric (tan δ), thermal properties, including thermal conductivity (K) for the nano polycarbonate polymer as a function of temperature and pressure in the same time.

The pressure values in this study were (0, 61, 65, 69, 73, 78, 82) MPa and the temperature values were (25, 50, 100, 150, 200, 250) °C.

The structural parameters calculated from x-ray diffraction are grain size, degree of crystallinity, d-spacing and inter-chain distance. And it has been shown that all previous parameters increase with increasing temperatures and decrease with increasing pressure.

The results of electrical properties showed that the values of electrical conductivity increased with increasing pressure, temperature and frequency. However, the values of the real dielectric constant and the loss tangent of dielectric have been shown to be inversely proportional to frequency while directly proportional to pressure and temperature.

As for thermal properties, thermal conductivity is inversely proportional to pressure and directly with temperature.

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List of abbreviations and Latin symbols

Abbreviation/Symbol	Meaning
РС	Polycarbonate
BPA	Bisphenol A
Tg	Glass transition temperature
PET	Polyethylene terephthalate
atm	Atmospheric pressure
SAN	Styrene-Acrylonitrile
ABS	Acrylonitrile-butadiene-styrene
PANI	Polycarbonate –polyaniline
СВ	Carbon black
PTFE	Polytetrafluoroethylene
GNP	Granular graphite nanoparticles
PS	Polystyrene
PVC	Polyvinyl chloride
Ра	Pascal's
n	Positive integer
d_{hkl}	The spacing of the crystal layers
X _t	Angle 20
A _t	Peak height
Pt	Angular location of top peak

Abbreviation/Symbol	Meaning
I _t (2Θ)	Totally scattering for separated peaks in the spectrum
I _{nor} (2 Θ)	Totally scattering for spectrum
В	Number of separated peaks in spectrum
WAXD	Wide angle X-ray diffraction
X _C	The degree of the crystallinity
I _C	Intensity of crystalline region
I _a	Intensity of amorphous region
D	Grain size
β	The line broadening at half the maximum intensity
θ	Bragg angle
r	Inter-chain distance
k	Thermally Conductivity
Q	The local heat flux density
K _t	Coefficient of thermal conductivity
$\frac{d\mathrm{T}}{dx}$	Temperature gradient
$T_{\rm C}, T_{\rm B}, T_{\rm A}$	The temperature of the disks C, B, A respectively
ds	Disc thickness
С	Capacitance of the sample
d	The thickness of the sample
ε _o	The permittivity of vacuum
f	Frequency of the applied field
ω	Angular frequency
σ_{ac}	AC-conductivity

Abbreviation/Symbol	Meaning
tan δ	The loss factor
ε′	Real dielectric constant
ε″	Imaginary dielectric constant
А	The cross section area of the sample



Chapter One Introduction &Literature review

1-1 Introduction

Plastic materials are preferred for designers and engineers, because of the advantages of these materials since they are lightweight, corrosionresistant, flexible, easy to process, transparent and a number of other properties. Polymers have distinct properties that are otherwise unavailable. The most important is the ease of production and resistance to oxidation and acidic and basic solutions, as well as the speed of colouring [1, 2].

The scientific and industrial importance of polymer science has grown significantly in the late 20th century, where statistics show that (40%) of scientists and (20%) of engineers work in the fields of plastic technology and industries [3].

Polymers are long-chain molecules of very high molecular weight. For this reason, the term "macromolecules" is frequently applied when referring to polymeric materials [4].

Polymers are large particles comprised of chemical repeating units that are associated with one another as a series of beads. They usually contain more than five monomers and others may contain hundreds or thousands of monomers in each series [5].

One of the most commonly used polymers in the world is called polycarbonate (PC), which is one of the polymers of thermoplastics. It contains in its chemical structure a group of carbonates. There are many applications of polycarbonate used in engineering because it is tough, strong and optically transparent in some grades, it works easily in terms of thermoformed and moulded [6]. There are trace amounts of monomer bisphenol A (BPA) in polycarbonate products, Bisphenol A is produced by the condensation of phenol with propanone [7].

1-2 Application of PC:

Polycarbonate is a very useful plastic material in applications requiring high resistance and transparency. It is used as a natural filter for ultraviolet radiation and a lighter alternative to glass. It is therefore used for eyewear and many other industries as in the following examples:

***** Electronic Ingredients:

Polycarbonate is a material that is used mainly in electronic applications which require heat resistance characteristics and good electrical insulation and flame retardant. It is used in telecommunications hardware, in electrical appliances and in mobile phones. It was used in (2011) for the first time in mobile phones by (Nokia) company in the (N9), and then used in (2012) as a cover for batteries in the third version of the phone of (Samsung's Galaxy), and then began Apple Company in (2013) to use it as a body in (iPhone 5C's) [8].

***** Construction material:

The construction industry is the second biggest consumer of polycarbonate, where it's used in the curved or flat glass and in the sound walls, which require multiwall or flat solid sheet, or corrugated sheets.

Data storage:

The production of discs of all kinds (Compact, Blu-ray and DVDs) is one of the basic applications of polycarbonate, which is manufactured by a mould cavity that is injected by the moulding of polycarbonate into its, these mould has a side of the mirror surface and the other side a negative image of the disk data.

Security, aircraft, automotive and railway components:

Very smooth surfaces can be obtained in the automotive industry when using injection moulding of polycarbonate, which makes these surfaces suitable for evaporation deposition of aluminum without the requirement for a basic layer. Optical reflectors and decorative bezels are usually made of polycarbonate. Polycarbonate is considered to be one of the most widely used materials in the lens industry for automotive headlamps, due to its high impact resistance and low weight. These lamps require an external coating to protect against scratches and ultraviolet radiation, which makes them deteriorate (yellowing). The use of polycarbonate is limited to low-stress applications for cars, where the stress caused by plastic welding or moulding render or fasteners effects on the polycarbonate and make it susceptible to stress corrosion cracking such as plastisol and salt water. Polycarbonate can be used in the manufacture of bullet-proof windows, as well as in the manufacture of thick plastic transparent barriers to bank windows and teller's windows by making it in the form of laminated layers [9].

* Specialty applications:

In many small applications, polycarbonate is a versatile material with physical properties and attractive processing, such as the industry of glasses, food containers and inverted syrup bottles. However, the use of (BPA) in polycarbonate industries has increased potential hazards in food contact applications, and thus led to the development of polycarbonate and to be used free of (BPA) in various formulations. Other applications in which polycarbonate is used are swimming glasses, sunglasses, safety glasses, sporting helmets, police riot gear and windshields for many motorcycles, golf carts and helicopters [10].

<u>1-3 Literature Review:</u>

- In (1970), Siegmann A and Geil PH studied the possibility of crystallization of the polycarbonate glass state and found that the thermal treatment under T_g affected the amorphous regions, causing it to be arranged and increased the degree of crystallization of polycarbonate, and the crystallization rate of the PC increased when the annealing under the T_g and after that the seed nuclei is formed that affects other processes [11].
- In (1984), Murff SR, Barlow JW, and Paul DR studied the mechanical and thermal attitude of PC- PE and where the results showed that the PET-PC blends rich in polycarbonate exhibit two glass transitions of the PE-PC blends point ou the existence of two amorphous phases of this blend. Interestingly, there is only one T_g of PET-rich blends and thus one mixed amorphous phase. Given the crystallization behaviour of polycarbonate, it is observed that the interaction between PET and PC does not lead to a common randomized copolymer and it is not very large during the practical processing conditions and shows that there are no large significant negative of the PET-PC when studying the mechanical properties of the typical additions [12].
- > In(1987), **Beckman E and Porter RS** studied the Crystallization of bisphenol a polycarbonate induced by supercritical carbon dioxide, and they found that when the previous compound is exposed to carbon dioxide for one hour and at low pressures and temperatures up to (100atm) and (75°C) respectively, the crystallization will begin and then the degree of crystallinity will increase rapidly when the pressure increases from (100 to 300)atm for the carbon dioxide, This is probably due to the minimum T_g of the PC-CO₂ mixture resulting from the opposite pressure on the glass temperature of the polymer [13].

- In (1987), Marshall J, Skochdopole RE and Finch CR studied the mechanical, thermal and impact properties of the polycarbonate mixture with two types of Styrene-Acrylonitrile (SAN), obtained by injection moulding. Where the results showed linear dependence and for many parameters, such as the heat distortion temperature, modulus and strength with blend composition and also showed that the solubility is limited among the components when analyzed by T_g, where the soluble of SAN more than the PC [14].
- > In (1988), **Kim and Burns** Concerning the thermal behaviour found that the T_g of PC in PCiABS blends reduces linearly with rising the acrylonitrile-butadiene-styrene (ABS) content. The T_g of ABS, on the other hand, increased linearly with rising polycarbonate amount. The T_g decrease of polycarbonate in PC/ABS blends was attributed to the polybutadiene chains acting as a plasticizer for PC [15].
- In (1991), Amin M and others studied the Current-voltage characteristics of polycarbonate samples (PC) have been studied in the voltage range 50–1000 V and thermal annealing on the electrical conductivity (σ) of PC. They have seen that the conductivity decreases with increasing time of annealing (2 h to 24 h). This may be due to the decreasing of free ions due to recombination [16].
- In (1999), Jeon BH, Kim S et al have studied characterization and synthesis of polycarbonate –polyaniline (PANI) composites that produced in emulsion polymerization manner, they found that increasing the temperature of the glass transition increases electrical conductivity This is due to the facility of transmission of the electron through the hydrogen bond between the (PC) and the (PANI), that resulted from the frequent contact between the PANI chains. Furthermore, the mechanical properties have shown that low (PANI) content results in a decrease in the tensile strength of the compound, because of the (PANI) functioned as a defect in

polycarbonate matrix, and also led to increasing the tensile modulus continuously due to the higher rigidity of the (PANI) than molecules [17].

- In (2000), Beyer J, Morshuis PH and Smit JJ studied the conduction current measurements on Polycarbonate samples which underwent simultaneous electrical and thermal aging. Where By recording the conduction current for different values of the electric field strength a transition from ohmic to high field conduction has been observed [18].
- In (2000), Lee WJ, Kim YJ and Kaang S studied the electrical properties of polyaniline (PANI)/sulfonated polycarbonate blends, where they used the conductor polymer polyaniline (PANI), and the matrix was polycarbonate, which was prepared by using the blending method. Using thermal and mechanical properties, the researchers were able to monitor the effect of ionic groups of polycarbonate. Morphology and electrical conductivity was also measured using the amount of (PANI) complex with the presence of protonating agents, it was observed that the amount of (PANI) complex protonated increases with increasing electrical conductivity up to (7.5 S/cm) [19].
- In (2007), Onbattuvelli VP studied the synthesis and characterization of palladium/polycarbonate nanocomposites, where the electrical, optical and thermal properties of nanocomposites have been studied in terms of the effect of varied morphology on them. Thermal stability and better optical transmission in ex situ nanocomposites were observed by the thermal stability of this composites on the heating rates, while higher electrical conductivity was detected in situ nanocomposites. By varying the shape, size, distribution and concentration metal nanoclusters, synthetic strategies can be developed to design new materials [20].

- > In order to study the properties of the dielectric (permittivity ε', resistivity ρ, dielectric relaxation time τ, dielectric relaxation process, dielectric loss ε) in terms of the influence of (MnCl2) salt on them, **Ayesh AS** prepared in (2008) the (PC/MnCl2) composites, at room temperature and at the frequency range (10 Hz to 306 kHz), studies of dielectric were performed as a function of salt concentration and frequency and the results showed that with increased frequency value and salt concentration, the resistivity of the composite would decrease. There was a change in the dielectric properties of the (PC) when the (MnCl2) salt was added to the (PC) host. They also found an increase in the dielectric loss and permittivity and reducing the thermal stability and relaxation time when broadening the dielectric spectra [21].
- In (2009), Ya. Lebedev et al studied specimens of Teflon by the use of x-ray diffraction. At first, three polymer components were used for the carried out quantitative analysis of X-ray powder. One crystalline phase and two amorphous phases were figured out for each specimen of Teflon. By the low molecular weight outputs, one of the amorphous phases was formed. Through XRD methods and computer simulation, the structure of this phase was confirmed [22].
- In (2011), Michael D. Via, Julia A. King, and Michelle E. Kinget studied the Electrical and thermal conductivity of carbon black/polycarbonate composites, where added varying amounts (2 to 10 wt %) of Carbon black (CB) to polycarbonate (PC), and the results showed that the carbon black/polycarbonate composites have electrical and thermal conductivity more than pure polycarbonate because of the Carbon black (CB) is very effective at increasing electrical and thermal conductivity for the composite [23].

- Some thermal analyses were applying by Rodrigo Canto et al in (2011) on the samples of pure or filled (PTFE) powders. These samples were obtained by uniaxial compaction or by isostatic pressing. Significant deformations were observed for the samples, which depended mainly on the pressure mode and level, and when applying the uniaxial compaction on these specimens, these distortions will be anisotropic strongly. As the various mechanisms, such as void closure and thermal expansion and recovery are the main cause of these deformations, which were observed during sintering [24].
- ➤ Julia A King et al studied in (2012) the possibility of adding granular graphite nanoparticles (GNP) to polymers for making an electrically conductive compounds, where varying quantities of graphite nanoparticles were added to PC, the electrical conductivity of these resulting composites was measured, and the addition of (GNP) to polycarbonate increased the composite electrical and thermal conductivity. The (8wt %) (5.0vol %) (GNP) in polycarbonate composite had a good combination of properties for electrostatic dissipative applications. The electrical resistivity and thermal conductivity were (4.0 × 107) ohm-cm and (0.37) W/m · K, respectively [25].
- The electrochemical characteristic and the electrode structure of poly (tetrafluoroethylene) are influenced by heat treatment time, as a conclusion by **MinJoong Kim** et al in (2013). The results showed that the large pore volume in the electrode decreases with increasing heat treatment time and concentration overpotential in the high-density region [26].
- In 2018, Sumadiyasa and Manuaba studied the possibility of using XRD technology to determine the size of the crystallite by applying the Williamson-Hull equation and Scherrer equation and apply them on Image-J software, then they found that the results are almost identical in both previous equations [27].

Gupta A and Goyal RK in (2019) studied the electrical properties of polymer matrix nanocomposites based on polycarbonate as matrix and expanded graphite (EG) as reinforcement, were fabricated using a simple solution method followed by hot pressing, and they found that the dc and ac electrical conductivities of the nanocomposites increased with increasing EG content in the matrix. They found that the increase in electrical conductivity, dielectric constant, and dissipation factor for the nanocomposites might be good for the applications in antistatic/electromagnetic interference shielding applications [28].

1-4 The aim of the work:

Due to the numerous and important uses of polymeric and plastic materials in previous years and in many advanced industrial applications, the purpose of the survey is as follows:

- 1- Investigation of the thermal properties of Nano PC at different temperatures and pressures.
- 2- Calculate the degree of crystallinity, Grain Size, d-spacing and the inter chain distance of Nano PC at different temperatures and pressures.
- 3- Investigation of the electrical properties of Nano PC at different pressures and temperatures.



Chapter Two Theoretical Part

2.1 Introduction

The word "polymer" is derived from the Greek (poly) and (meros), meaning many and parts, respectively. Some scientists prefer to use the word "macromolecule," or large molecule, instead of polymer. [29] The polymer is formed by a combination of quite small molecules called monomers by the interaction between them that called polymerization [30].

There are two types of polymerization, namely polymerization of condensation and addition, which were explained in (1929) by Carothers, Where the interaction of conversion from monomers to long-chain polymers by the elimination of small molecule such as water called polymerization condensation, While the interaction of conversion from small chain monomers to long-chain polymers without the elimination of any small molecules or atoms, is called addition polymerization [31].

2-2 Classification of Polymers: [32-35].

There are several ways of classification of polymers based on some special considerations. The following are some of the common classifications of polymers:



Figure (2-1) ways of classification of polymers [33].

(2-2-1) Classification Based on Source:

There are three subcategories under this sort of classification:

a- Natural polymers:

Plants and animals are natural sources of the polymer, which are found in animals in the form of proteins, or in plants in the form of cellulose and starch, as well as rubber that is harvested from the latex of the tropical plant considered as a natural polymer.

b- Semi synthetic polymers:

When the natural polymers are modified in the laboratory, the resulting polymers will be called Semi-synthetic polymers. These polymers have commercial significance such as Cellulose acetate (rayon), as well as Vulcanized Rubber, which uses sulfur as a bonding material in the natural polymer(Which uses sulfuric as a bonding material in natural rubber chains). These Semi-synthetic polymers are formed in a controlled environment and by some chemical reactions.

c- Synthetic polymers:

Polyethylene, which is used in packing, as well as Nylon Fiber, which is used in fishing nets and clothing, is a common example of Synthetic Polymers. These polymers are synthesized in laboratories and are used commercially for human necessities

(2-2-2) Classification Based on Structure of Polymers:

As shown in figure (2-2), there are three different types based on the structure of the polymers:

a- Linear polymers

PVC (Poly-vinyl chloride) is a common example of these polymers, which have a high density and melting point, where the structures of these polymers are similar and their straight long chains are bound by identical links connected to each other.

b-Branched chain polymers

Low-density polyethene (LDPE) used in containers and plastic bags is one of the Branched-chain polymers. The structure of these polymers is similar to the branches originating at random points from a single linear chain, and their low densities and melting points indicate that the polymers not closely packed together because of the different branches lengths and associated with straight and long chains that created by the monomers

c- Cross linked or Network polymers

A three-dimensional network is formed by monomers linked together in this type of polymer, these polymers are hard and brittle because they contain monomers with strong covalent bonds. Examples of these polymers are Melamine and Bakelite used as electrical insulators.



Figure (2-2) some patterns of polymers based on the chain forms

(2-2-3) Classification Based on the Mode of Polymerization:

Polymerization is the process by which monomer molecules are reacted together in a chemical reaction to form a polymer chain (or threedimensional networks). Polymers based on this type can be classified into:

a- Addition Polymers

Additional polymers are formed by the frequent addition of monomer molecules such as the formation of polypropene from propane and polythene from ethene. There are two types of polymers: copolymers polymers such as Buna N, Buna S which formed by the addition of polymerization of two different monomers, the other type is homopolymers such as polythene, which made by polymerization of the single monomeric gender.

b- Condensation Polymers

Nylon 6 is a common example of this type of polymer. In a frequent reaction between two monomer unit (tri-functional or bi-functional) condensation polymers will be formed, small molecules such as water, alcohol and hydrogen chloride are eliminated during this reaction.

(2-2-4) Classification Based on Molecular Forces:

The strength of the forces between molecules in solids such as polymers affect the properties of these materials. Polymers can be classified into four species depending on the molecular forces:

a- Thermoplastic Polymers

These polymers are softened when heated and hardened when cooled, it can be easily shaped by heating and using moulds, as it does not contain any cross-links. The long chains of these polymers are linked by inter-molecules forces (Van der Waal's forces), such as (PVC), which is used in the manufacture of pipes and polystyrene.

b- Thermosetting Polymers

These polymers have a low molecular mass and are semi-fluid in nature, but when heated, they will become infusible and hard and start to cross-link between polymer chains, and formation a three-dimensional structure, which is irreversible in nature and examples of that Bakelite that used in the insulators.

c- Elastomers

These solid materials are elastic, rubber-like, and stretchable because the intermolecular forces that bind polymer chains are weak. These polymers can return to their original place after releasing the forces, due to the presence of 'cross-links' amongst polymer chains, such as neoprene and vulcanized rubber.

d- Fibers:

These polymers are a thread-like in nature where they can be easily woven. These fibers have a high melting point, high tensile strength and low elasticity, because of the strong inter-molecules forces between the chains. These intermolecular forces are dipole-dipole interaction or hydrogen bonds. These fibers are used in apparels and carpets such as Nylon-66

2.3 Polycarbonate:

Polycarbonates are polyesters of carbonic acid and diols or diphenols that feature a carbonate group (-O-C (O)-O-) in their repeating unit and which are each of the aromatic and the aliphatic type [36-38].



Figure (2-3) structure of polycarbonate.

Polycarbonates are a principal engineering plastics which find uses in a wide range of fields, in particular in appliances and consumer goods, packaging and building materials, and electrical, automotive, aircraft, and electronic components, safety helmets, optical lenses, load-bearing electrical components, shields, medical apparatus, electrical insulators, business machine components. Certainly, they feature excellent physical properties, a fair chemical resistance, very good heat resistance, flame retardancy, excellent toughness, electrical insulation, optical characteristics, and fair processing, A variety of materials are produced and are useful by polycarbonate, which has the characteristics of transparency and shock resistance such as bulletproof glass [36-38].

The Polycarbonate is for the time being most commonly synthesized by the ring-opening polymerization of cyclic carbonate monomers or the copolymerization of epoxides with carbon dioxide, while the past approach allows in some status the preparation of telechelic polymers, that oftentimes impeded by the formulation of the five-membered cyclic carbonate side product and by the concomitant decarboxylation reaction leading to poly (ether-carbonate) instead of nano Polycarbonates. [36-38]. Through the 1980s, polycarbonate turned into the principle thing of business and research was done on its properties, including optical qualities, electrical and mechanical properties. In view of its capacity to be changed and custom fitted to explicit applications, the polycarbonate business is very broad [39].

2-4 Principle of Powder Diffraction:

X-rays can be defined as wavelength-specific electromagnetic waves, located between ultraviolet rays and gamma rays, whose wavelengths range from (0.1-100) Å, so they are best used in most crystal diffraction experiments. In general, X-ray diffraction depends on: the crystalline structure and the wavelength of the radiation used, that is, the wavelength should be equal to or near the fixed lattice.

The scientific technique used to characterize the structure of the material is called powder diffraction which use x-ray, neutron or electron in the detection of micro-crystalline or powder samples.

Even if the size of the crystal is very small, the powder diffraction technique can provide accurate structural information about that crystal.

The applications of powder diffraction have increased dramatically in recent years. This technology has been used in the manufacturing, research and data processing, and has given laboratories the ability to quickly characterize and analyze unknown materials. This technique allows a rapid and non-destructive analysis of multi-component mixtures without the need for preparation of these samples, This technique has been applied in many fields such as materials, chemistry, geology, forensics, mineralogy, biological, pharmaceutical and archaeology sciences. This technique is characterized by:

- Simplicity of specimen preparation: many materials are readily available for this technique.
- Rapidity of measurement: collection times can be very short, since all possible crystal orientations are measured simultaneously. It is one of the most powerful methods to characterize and identify new materials [40-43].

Bragg reached to deduce his law, which is based on the difference of the path between the incident and reflected radiation, which is equal to the length of one wave or the correct multiples of the wavelength (n λ). Bragg's law is described in the following relation: [44]

 $2d_{hkl}\sin\theta = n\lambda\dots\dots\dots(2-1)$

Where:

n is a positive integer

 $\boldsymbol{\lambda}$ is the wavelength of the incident wave

 d_{hkl} is the spacing of the crystal layers (path difference)

 θ is the incident angle (the angle between incident ray and the scatter plane)



Figure (2-4) Bragg diffraction.

In order to achieve the reflection of Bragg, this condition, which is called the Bragg condition, must be met [44].

 $\lambda \ \leq \ 2 \ d_{hkl} \qquad \dots \dots \dots \dots \dots \dots (2-2)$

The information that can be obtained by studying X-ray diffraction is:

(2-4-1) Degree of Crystallinity:

It is the degree of structural arrangement in the solid materials where atoms or molecules are organized in a uniform and periodic manner in crystal. This property plays an important role in influencing the density, hardness, transparency and diffusion of materials and can be extracted by differential scanning or X-ray diffraction [44].

There are several methods to determine the degree of crystallinity by X-ray diffraction:

a- Separation of overlapping peaks: This method was applied in (1989) by Sh.M.Abdo and Hineleh on synthetic polymers. The degree of

crystallization was determined in the cases of preferred orientation or texture polymers.

It requires quantization crystalline in this method to separate the distribution of intensity of peak of the amorphous regions from crystalline regions, it also requires correction of the intensity from the effect of the Lorenz factor and polarization [45].

The intensity I_{cal} can be calculated based on the Gauss function and according to the following equation:

Where:

A_t: Peak height;

X_t: Angle 2 Θ ;

n: Number of height peaks calculated;

Pt: Angular location of top peak.

The ratio between the scatterings of the totally separated peaks to total scattering under the form side of the spectrum at a specified angular range is known as crystallinity X_C and as shown in the equation below: [46].

$$X_{\rm C} = \frac{\sum_{t=1}^{B} \int_{2\Theta_1}^{2\Theta_2} I_t(2\theta) d(2\theta)}{\int_{2\Theta_1}^{2\Theta_2} I_{nor}(2\theta) d(2\theta)} \dots \dots (2-4)$$

Where:

 $I_{nor}(2\Theta)$: totally scattering for spectrum;

 I_t (2 Θ): totally scattering for separated peaks in the spectrum;

B: number of separated peaks in spectrum;

 $2\Theta_2$ - $2\Theta_1$: angular range chosen.

b- Wide-angle x-ray diffraction (WAXD): is one of the most common methods to calculate the degree of crystallinity. The entire area under the curve represents the intensity of the X-ray scattered. This area is divided into two regions, called the crystalline region and the amorphous region, where the intensity of the beam is measured in each region, the intensity of X-ray in the part (I_c) will be under the sharp peaks, while the intensity of x-ray in the amorphous part (I_a) is the remaining area under the curve [47].



Figure (2-5) shows the amorphous and crystalline regions.

For this method, the degree of crystallinity is calculated by the following equation:

$$X_{c} \% = \frac{Ic}{Ic + Ia} \dots (2-5)$$

$$X_c \% = \left(\frac{Ic + Ia}{Ic}\right)^{-1}$$
..... (2-6)

$$X_c \% = (1 + \frac{la}{lc})^{-1} \dots (2-7)$$

Where:

Ia: Intensity of amorphous region;

X_c: Is the degree of the crystallinity of the polymer;

I_c: Intensity of crystalline region.

In this method, it is preferred a slow speed of angular scanning and long count times to get high-quality data, where this method is superior to that of conventional intensity minimum line structure method [48].

(2-4-2) Grain size:

The grain size (D) is calculated by Debye-Scherrer equation: [49]

Where:

D is the grain size (Å);

 β is the line broadening at half the maximum intensity (FWHM)(Å);

 λ is the X-ray wavelength(1.5418)(Å);

 θ is the Bragg angle(deg);

K is a dimensionless shape factor, with a value close to unity (0.89-0.9).

(2-4-3) Inter-chain distance:

The inter-chain distance (r) is calculated by flowing equation: [50]

 $r = \frac{5}{8} * \frac{\lambda}{\sin\theta} \quad \dots \quad \dots \quad \dots \quad \dots \quad \dots \quad (2-9)$ Where:

 θ is the Bragg angle(deg);

r is the inter-chain distance (Å);

 λ is the X-ray wavelength(1.5418) (Å);
(2-4-4) d-spacing:

The d-spacing can be described as the distance between planes of atoms that give rise to diffraction peaks, Calculation of the interstitial distance of crystalline materials according to Bragg's law [51].



Figure (2-6) showing the d-spacing between the planes of atoms.

2-5 Thermally Conductivity:

Polymers are poorly thermal conductivity materials and this fact leads to the identification of one of the most important thermal properties that affect most industrial applications known as thermally conductivity [52].

Thermal conductivity is one of the methods of heat transfer in the material. It is referred to as the ability of the material to connect the heat and transfer it. Its denoted by κ ,k, or λ . There are other methods of heat transfer known as radiation and convection. Fourier's laws are the basis for the heat transfer equation [53].

Molecular agitation is responsible for the occurrence of thermal conductivity. Along the temperature of the gradient, the heat will move from the high molecular energy (high temperature) regions to the low molecular energy (low temperature) regions. This transfer will then stop when the thermal equilibrium is reached. The specific thermal properties of the material and the magnitude of the temperature gradient are responsible for the heat transfer rate [54].

The reciprocal of thermal resistivity (W/mK) is thermal conductivity measurement units that calculated by the equation: [55]

 $Q = -K_t \frac{dT}{dX}$ (2 - 10)

Where:

Q is the local heat flux density $(W \cdot m^{-2})$; K_t is coefficient of thermal conductivity $(W \cdot m^{-1} \cdot K^{-1})$; $\frac{dT}{dx}$ is the temperature gradient $(K \cdot m^{-1})$.

A negative signal means that the heat moves from the hightemperature zone to the less hot zone. There are two basic methods for calculation of thermal conductivity (K): [54]

• Searle's Bar Method:

This method is used to measure the thermal conductivity (K) of a material with good thermal conduction. The principle of this system is the application of Fourier's law described in Relationship (2-10).

• Lee Disc Method:

This method is simple and effective to calculate the thermal conductivity of materials with low thermal conductivities, as shown in figure (2-6).



Figure (2-7) shows Lee disk device [54].

The sample to be examined (S) is placed between the two copper plates (B, A) and The heater connected to the power supply source is placed between the two disks (C, B), When a constant voltage difference is placed on both ends of the heater, the temperature will increase and the heat energy will then be transferred to the two disks (C, B) evenly or in very close quantities, This is due to the full contact of the two disk with the heater. Thermal energy is transferred from disk (B) to disk (A) by the test sample (S), from the following equation, we can derive the value of thermal conductivity: [56]

$$k\left[\frac{T_B - T_A}{ds}\right] = E\left[T_A + \frac{2}{r}\left[d_A + \frac{1}{4}ds\right]T_A + \frac{1}{2r}ds T_B\right]\dots\dots(2-11)$$

Where:

E is the amount of heat passing through the unit of area per second.

The value of (E) can be calculated by knowing that the energy entering the heater is equal to the energy coming out of it. In other words, the energy entering the heater is equal to the energy dissipated to the outer environment via the disks (C, B, A) and thus: [56]

$$IV = \pi r^2 E(T_A + T_C) + 2\pi re \left[d_A T_A + ds \left(\frac{T_A + T_B}{2} \right) + d_B T_B + d_C T_C \right] \dots (2 - 12)$$
Where:

where:

I: Current in the heated coil (I = 0.37 Ampere);

V: The voltage difference on both ends of the heater coil (V = 6 Volt);

T_C, T_B, T_A: The temperature of the disks C, B, A respectively;

ds: Disc thickness (2.6)(cm).

2-6 Electrical Conductivity:

The electrical conductivity depends in general on the presence of free ions or electrons and their movement, These ions or electrons in the

insulating materials are constrained, because of the strong correlation between the atoms, which is formed by strong covalent bonds, In other words, electrons are highly localised in atoms and are not free to roam in the crystal [57].

Because of the strong bonding of electrons in the case of insulation materials, including polymers, due to an energy gap, which causes low electrical conductivity. This conductivity varies from polymer to polymer. In branched polymers, the value of electrical conductivity is increased by a certain amount due to the interconnectivity that makes the electrons move more easily [58].

Electrical conductivity is the measure of the materials susceptibility to transport electrical charges, and its unit is siemens per meter (S/m) and its symbol is σ [59-61].

The dielectric constant of polymers is of great importance and has become increasingly important when polymers have been used as insulating materials, because they are important for engineering applications. The insulation constant is measured from the capacitance of the electrolytic capacitor which includes the polymer material as an insulating medium to the capacitor capacitance when the air only includes an insulating medium [62,63].

Since the Polycarbonate behaves as an insulator (dielectric material) at pre-breakdown region (low current region) so it has dielectric properties which include the real dielectric constant (ϵ'), the imaginary dielectric constant (ϵ'') and the loss tangent (tan δ).

The real dielectric constant (ϵ') or the relative permittivity is the ratio of the material's permittivity relative to the permittivity of vacuum, [64] and it can be calculated from the equation:

 $\epsilon' = Cd/A \epsilon o \dots \dots \dots \dots \dots \dots \dots \dots \dots (2-13)$

 ε_{o} : the permittivity of vacuum;

C: the capacitance of the sample;

A: the cross section area of the sample;

d: the thickness of the sample.

While the imaginary dielectric constant (ϵ'') (dielectric loss) can be calculated from the equation:

 $\epsilon'' = \tan \delta * \epsilon' \dots \dots \dots \dots \dots \dots \dots \dots \dots (2 - 14)$

 $tan\delta$: is the loss factor which refers to the ratio between the amount of real and imaginary dielectric constants. [65]

Electrical conductivity is measured from the following equations:

 $\omega = 2\pi f \dots \dots \dots \dots \dots \dots \dots \dots \dots (2-15)$

 $\sigma ac = \omega \varepsilon \varepsilon \varepsilon' * \tan \delta \dots (2-16)$

Where:

 ω is angular frequency;

 σ_{ac} is ac-conductivity;

f is the frequency of the applied field.



Chapter Three Experimental Part

3-1 introduction:

This chapter includes the materials and devices used in the preparation process, and includes a method Preparation and the shape of prepared samples, as well as a general description of the devices used in this study.

The experimental work is explained in the flowchart of the figure (3-1).



Figure (3-1) A flowchart explains the experimental work

3-2 Samples used

Nano polycarbonate has been used in our current research in the form of powder and product from Japanese company (Teijin).

Glass transition temperature (Tg)	297°F (147°C)
Compressive strength	80 MPa
Density (ρ)	1.20–1.22 g/cm3
Thermal conductivity (k) at 23 °C	0.22–0.19 W/(m·K)
Dielectric constant (ϵ_r) at 1 MHz	2.9
Dissipation factor at 1 MHz	0.01

 Table (3-1) Some properties of Nano PC.

3-3 Samples preparation

The Nano polycarbonate samples are prepared by the following steps:

(3-3-1) Weighting:

To measured proper weights, we used a sensitive electronic balance. 42 samples were weighed, where each sample weight was (3g). When using the sensitive electronic balance in the laboratory, it should be clean from the inside and outside. It is preferable to use the same balance throughout the duration of preparation of the samples to avoid the error. This type of scales has the ability to determine the weights at high accuracy 10^{-4} g (as shown in Fig (3-2).



Figure (3-2) Sensitive electronic balance.

(3-3-2) Forming (Compacting):

The powder was pressed for 1 min, into disc-shaped pellets 10 mm in thickness and 19 mm in diameter, using hydraulic uniaxial press on 36 samples, every six samples were compressed in one of the following ranges (61, 65,69,73,78,82) MPa , as shown in figure (3-3),(3-4).



Figure (3-3) hydraulic uniaxial press with the model.



Figure (3-4) One of the samples after pressing it's by hydraulic uniaxial press.

(3-3-3) Heat treatment:

Heating each Seven of the different pressured samples at one of the ranges (25, 50,100,150,200,250) °C for 20min by using a non-vacuum electric furnace (Carbolite), at the heating rate 5 °C/min. The furnace is an electrical device with thermal insulation temperature controlled by a thermal regulator and is used for the process of drying, heating, sintering and calcification, temperature range ranging from (25-1100) °C, as shown in figure (3-5).



Figure (3-5) Electric Furnace.

<u>3-4 X-Ray Diffraction Measurements</u>

In order to measure the Bragg's angle and obtaining a high accuracy of diffraction spectra for the samples, as well as the intensities of the sites associated with the angles, the X-ray diffraction system, was used. The reflections were recorded from the crystalline levels by the counter, which appears on the computer by a special program

Through the use of an X-ray diffraction device (SHIMADZU Japan) (XRD600), this technique was worked as shown in the figure (3-6), with the following features:

Radiation	CuKa1
Voltage	40 kv
Wavelength	1.5418 A
Current	30 mA
Speed	8deg /min
Range	(5-80) deg

 Table (3-2) Specifications of XRD device.



Figure (3-6) Show XRD device.

42 samples were tested in this device at different temperatures and pressures for each sample in the same time and according to the following ranges of temperature and pressure:

- 1- P=(0,61,65,69,73,78,82) MPa
- 2- T= (RT, 50,100,150,200,250) °C.



Figure (3-7) shows all samples processed for the XRD analysis.

3-5 Thermal Conductivity Measurements

George and Griffin Company manufactured Lee's Disc device which was used in our current study, which enables us to calculate the thermal conductivity of all samples, in which heat is transferred from the heater to the disc that follows it until it reaches the final disk. The temperature of the three disks can be determined (TA, TB, TC) using the thermometers respectively, shown in figure (3-8).



Figure (3-8) shows thermal conductivity test device.

36 samples were tested in this device at different temperatures and pressures for each sample in the same time and according to the following ranges of temperature and pressure:

- 1- P=(61,65,69,73,78,82) MPa
- 2- T= (RT, 50,100,150,200,250) °C.



Figure (3-9) shows all samples processed for thermal conductivity measurements.

3-6 Electrical Conductivity Measurements

The ac-conductivity and the dielectric characteristic such as the loss tangent of dielectric $(\tan \delta)$, the real (ϵ') and imaginary (ϵ'') dielectric constant were measured in the range (50 Hz,1 MHz,3 MHz) using the (LCR METER) model (8000G Series, GW instek company, Japan, frequency range (50Hz-10MHz). as shown in figure (3-10).



Figure (3-10) shows electrical conductivity test device.

36 samples were tested in this device at different temperatures and pressures for each sample in the same time and according to the following ranges of temperature and pressure:

- 1- P=(61,65,69,73,78,82) MPa
- 2- T= (RT, 50,100,150,200,250) °C.



Figure (3-11) shows all samples processed for electrical conductivity measurements.



Chapter Four Results & Discussion

(4-1) introduction :

We will discuss the results of the experimental work in this chapter, which include the effect of pressure and temperature on some properties of Nano polycarbonate by using various analyzes and tests, such as electrical and thermal properties tests, X-ray diffraction analysis.

(4-2) X-ray Diffraction :

(4-2-1) the Degree of Crystallinity from X-ray Diffraction (XRD)

The spectrum of refractive radiation shown in Figure (4-1)(A-B-C-D-E-F) that scanned in the angular extent of $(5^{\circ}-80^{\circ})$ for the samples treated at different temperatures between (25 - 250) °C and different pressures between (61 - 82) MPa.







Figure (4-1) (A) XRD of polycarbonate at 50 ° C with different pressures.





Figure (4-1) (B) XRD of polycarbonate at 100 ° C with different pressures.







Figure (4-1) (C) XRD of polycarbonate at 150 ° C with different pressures.





Figure (4-1) (D) XRD of polycarbonate at 200 ° C with different pressures.







Figure (4-1) (E) XRD of polycarbonate at 250 ° C with different pressures.





Figure (4-1) (F) XRD of polycarbonate at 25 ° C with different pressures.

The degree of crystallinity of the polycarbonate samples was calculated from the intensity measurements of the diffraction spectra at the different temperatures and pressures by using the equation (2-5) and applying it's on the Microsoft Office Excel program, where the results of the degree of crystallinity (X_C) were summarized in Table (4-1). Where it is show that values of the degree of crystallinity increase with increasing temperature but decreases with increasing pressure.

Degree of crystallinity								
T/P	T=25 °C	T=200°C	T=250°C					
P=0 MPa	60.3579	61.98763	62.7041	64.8136	65.8136	70.11049		
P=61 MPa	56.6916	59.84739	60.1264	60.4756	62.6525	67.25158		
P=65 MPa	55.8074	57.44095	57.9607	58.9487	60.2601	64.26014		
P=69 MPa	55.7727	56.36842	57.7571	58.5863	59.3099	61.08975		
P=73 MPa	53.4975	54.09508	55.3854	55.6538	56.5331	58.16458		
P=78 MPa	52.0335	52.16867	53.6847	53.6329	54.3428	55.15921		
P=82 MPa	50.5695	50.24227	51.984	51.612	52.1524	52.15384		

 Table (4-1): Degree of crystallinity as a function of pressure and temperature



Figure (4-2) X_C of polycarbonate at different pressure and temperature.

We observe from Figure (4-2) that the temperature increasing will increase the degree of crystallinity (X_C) until the temperature reaches (250 ° C). This is due to the fact that the polycarbonate samples are a shrinkagefree during heat treatment. This causes freedom in the movement of the molecules inside the crystal lattice. This is because the way of manufacturing the samples leads to the highest preferred orientation of these samples, and this is contrary to semi-crystalline polymers such as (PP, PET, PE...) in which the degree of crystallinity decreases during the temperature increase [66].

The degree of crystallinity decreases with increasing pressure Due to the break of covalent bonds as a result of pressure and thus decreases the degree of structural order [67].

(4-2-2) Grain Size:

The Bragg angle (2θ) and FWHM were calculated through (Origin Lab) program. Where the (XRD) values were included in this program and extraction the data that needed to calculate the grain size. The grain size was calculated from (Debye-Scherer) equation (2-8) and applying it's on the Microsoft Office Excel program, as shown in Figure (4-3).

The results obtained in Table (4-2) show that values of grain size increase with increasing temperature but decreases with increasing pressure.

Grain size (Å)							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=200°C	T=250°C	
P=0 MPa	1.65907	1.678535	1.83272	1.98914	2.02519	2.244628	
P=61 MPa	1.60017	1.686895	1.72923	1.7974	1.96817	2.185022	
P=65 MPa	1.59946	1.639458	1.67853	1.73308	1.73933	2.144628	
P=69 MPa	1.45235	1.497089	1.57591	1.64994	1.96817	2.06519	
P=73 MPa	1.44995	1.437757	1.55433	1.57546	1.66371	1.668165	
P=78 MPa	1.3084	1.416594	1.64482	1.60017	1.68002	1.785022	
P=82 MPa	1.26012	1.245125	1.54591	1.58152	1.63182	1.717225	

Table (4-2): Grain size of polycarbonate at different pressure and temperature.



Figure (4-3) Grain size of PC as a function of pressure and temperature.

The dependence of grain size on temperature is shown in Fig (4.3). Where the grain size increased slowly at first whilst the grain size increased more rapidly after that. Grain growth rate increases more rapidly at the higher annealing temperature (135 °C for PC). These kinetics of grain growth trend was influenced by the grain size which is consistent with previous studies [68].

(4-2-3) d_{hkl} –spacing:

The Bragg's equation (2-1) was used to calculated the d-spacing of the polycarbonate samples, taking into account that the wavelength is (1.5418) Å and (n = 1) as shown in Figure (4-3).

The results obtained in Table (4-4) show that values of grain size increase with increasing temperature but decreases with increasing pressure.

d–spacing(Å)								
T/P	T=25 °C	T=50°C	T=150°C	T=200°C	T=250°C			
P=0 MPa	0.51375	0.517321	0.51953	0.52155	0.53148	0.54045		
P=61 MPa	0.51322	0.514613	0.51794	0.52009	0.52394	0.534009		
P=65 MPa	0.51293	0.513729	0.51585	0.51834	0.52123	0.531998		
P=69 MPa	0.51247	0.513121	0.51409	0.51678	0.5193	0.524367		
P=73 MPa	0.51202	0.512514	0.51334	0.51522	0.51737	0.520735		
P=78 MPa	0.51157	0.515906	0.51458	0.51467	0.51744	0.518104		
P=82 MPa	0.51112	0.512298	0.51283	0.51311	0.51551	0.517473		

 Table (4-4): d_{hkl} –spacing of polycarbonate at different pressure and temperature.



Figure (4-3) d_{hkl} –spacing of PC as a function of pressure and temperature.

Figure (4-3) show that when the temperature increases, the energy distribution between the atoms of the molecules in (PC) changes, thermal vibration of the atoms increases. The unit cell expands and causes changes in d-spacing and therefore in (2 θ) positions of the diffraction lines.

The (d-spacing) increases with decreasing pressure due to the number of broken bonds per unit volume at a distance from the surface at the time. We follow previous theories in assuming that broken bonds occur whenever this bond breaking density exceeds the threshold value for force bonding [67].

(4-2-4) inter-chain distance:

The inter-chain distance of the polycarbonate samples was calculated using the equation (2-5) and applying it's on the Microsoft Office Excel program, where the results of the inter-chain distance (r) were summarized in Table (4-5).

The results obtained in Table (4-5) show that values of inter-chain distance (r) increase with increasing temperature but decreasing with pressure, and for all samples and the same reasons of the d-spacing.

Inter-Chain distance (Å)								
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=200°C	T=250°C		
P=0 MPa	0.64219	0.646652	0.65118	0.65577	0.66435	0.680563		
P=61 MPa	0.64665	0.655767	0.65577	0.65886	0.66674	0.67155		
P=65 MPa	0.64491	0.649662	0.65156	0.65342	0.66654	0.667511		
P=69 MPa	0.64272	0.646652	0.64966	0.65577	0.66435	0.664354		
P=73 MPa	0.64166	0.642776	0.64618	0.64994	0.65042	0.660564		
P=78 MPa	0.64132	0.641938	0.64674	0.64886	0.65153	0.659642		
P=82 MPa	0.64072	0.642642	0.64377	0.65155	0.65423	0.654938		

 Table (4-5): inter-chain distance of polycarbonate at different pressure and temperature.



Figure (4-4) inter-chain distance (r) of PC as a function of pressure and temperature.

(4-3) Thermal Conductivity:

Phonons are the only carrier of thermal energy, through which the thermal conductivity of insulation solids material is made because the phonons play a large role in the thermal conductivity of all solid materials [69].

The thermal conductivity of polycarbonate specimens was measured by using equations (2-10), (2-11) and applying it's on the Microsoft Office Excel program, as shown in Fig (4-5). The results obtained in Table (4-6) show that values of thermal conductivity increase with increasing temperature but decreases with increasing pressure.

Table (4-6): Thermal conductivity as a function of pressure and temperature.

Thermal conductivity (W/M.K)								
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=200°C	T=250°C		
P=61 MPa	0.0035	0.003985	0.0051	0.00599	0.00675	0.006857		
P=65 MPa	0.00244	0.002641	0.00315	0.00376	0.00464	0.005131		
P=69 MPa	0.00236	0.002405	0.00269	0.00271	0.00313	0.003278		
P=73 MPa	0.00223	0.002401	0.00248	0.00263	0.0029	0.003131		
P=78 MPa	0.00216	0.002355	0.00258	0.00278	0.00308	0.003281		
P=82 MPa	0.00212	0.00231	0.00268	0.00293	0.00316	0.003222		



Figure (4-5) Thermal conductivity as a function of pressure and temperature

We note from figure (4-5) that the thermal conductivity increases with increasing temperature due to increased segmental mobility in the polymer chains [70].

Figure (4-5) shows also that the thermal conductivity decrease with increasing pressure , here when the pressure increase, the contrast in the bonding strength between no bonded and covalent interactions becomes less pronounced; these effects may reduce the density of localized vibrations at high pressures and suppress contributions to thermal transport from localized modes and anharmonic effects [71].

(4-4) Electrical Conductivity:

(4-4-1) AC-Conductivity (σ_{ac}):

The alternating electrical conductivity of polycarbonate was calculated at different pressures, temperatures and frequencies by equations (2-13) (2-14) (2-15) (2-16).

Table (4-7): Electrical conductivity as a function of pressure and temperature at 50 Hz.

Electrical conductivity $(\Omega. m)^{-1}$ at 50 Hz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	2.4*10-6	2.49*10-6	2.55*10-6	2.61*10-6	1.05*10-5		
P=65 MPa	3.61*10-6	3.80*10-6	3.82*10-6	3.94*10-6	2.50*10-5		
P=69 MPa	5.44*10 ⁻⁶	5.61*10 ⁻⁶	5.68*10-6	6.38*10 ⁻⁶	2.50*10-5		
P=73 MPa	5.72*10-6	5.85*10 ⁻⁶	6.22*10-6	6.23*10-6	2.50*10-5		
P=78 MPa	5.82*10-6	5.86*10 ⁻⁶	6.46*10-6	6.29*10 ⁻⁶	3.71*10 ⁻⁵		
P=82 MPa	5.84*10-6	5.86*10-6	6.33*10-6	6.30*10-6	3.86*10-5		

Table (4-8): Electrical conductivity as a function of pressure and temperature at 1MHz.

Electrical conductivity $(\Omega, m)^{-1}$ at 1MHz								
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C			
P=61 MPa	5.20*10-5	5.36*10-5	5.58*10-5	6.02*10 ⁻⁵	7.02*10 ⁻⁵			
P=65 MPa	5.25*10-5	5.37*10 ⁻⁵	5.69*10-5	6.05*10 ⁻⁵	7.21*10 ⁻⁵			
P=69 MPa	5.33*10 ⁻⁵	5.47*10 ⁻⁵	5.79*10-5	6.09*10 ⁻⁵	7.92*10 ⁻⁵			
P=73 MPa	5.76*10-5	5.83*10-5	5.92*10-5	6.09*10-5	9.14*10 ⁻⁵			
P=78 MPa	5.79*10 ⁻⁵	5.46*10-5	5.76*10-5	6.17*10 ⁻⁵	0.00011			
P=82 MPa	5.78*10 ⁻⁵	5.44*10 ⁻⁵	5.64*10-5	6.21*10-5	0.00015			

Table (4-9): Electrical conductivity as a function of pressure and temperature at 3MHz.

Electrical conductivity (Ω . m) ⁻¹ at 3MHz								
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C			
P=61 MPa	1.02*10-4	1.04*10-4	1.12*10-4	1.40*10-4	2.08*10-4			
P=65 MPa	1.03*10-4	1.04*10-4	1.31*10-4	1.42*10-4	2.77*10-4			
P=69 MPa	1.04*10-4	1.07*10-4	1.33*10-4	1.46*10-4	2.79*10-4			
P=73 MPa	1.08*10-4	1.10*10-4	1.34*10-4	1.52*10-4	4.36*10-4			
P=78 MPa	1.1*10-4	1.11*10-4	1.39*10-4	1.58*10-4	4.45*10-4			
P=82 MPa	1.12*10-4	1.13*10-4	1.4*10-4	2.12*10-4	5.94*10-4			



Figure (4-6) Electrical conductivity as a function of pressure and temperature at 50 Hz



Figure (4-7) Electrical conductivity as a function of pressure and temperature at 1MHz


Figure (4-8) Electrical conductivity as a function of pressure and temperature 3MHz

Table (4-7), (4-8) and (4-9) show electrical conductivity at (50Hz, 1MHz, 3MHz), it has been shown that the values of electrical conductivity increase with increasing temperature, pressure and frequency, and for all samples.

From figures (4-6), (4-7) and (4-8) we note the response of the σ_{ac} for (PC) at various temperatures. Generally, the noted improvement in σ_{ac} is attached to the electronic interactions, where the samples contain definite quantities of impurities and metallic ions which are activated at a special temperature, yet samples that have a poorer degree of conducting components must show a less reaction and, in this way, giving weak electrical conductivities. [72, 73].

We conclude from figures (4-6), (4-7) and (4-8) above that the σ_{ac} improved by increasing of the frequency, where at the high-frequencies the contributions of the polarization effects reduce [74-76].

When we study the figures (4-6), (4-7) and (4-8) that the utilisation of pressure caused an increasing conductivity, where pressure caused movement of the polymer chains, which affect the arrangement structure of the polycarbonate. The variance in pressure with σ_{ac} can be explained by looking at two phenomena that occur in the structure of the material, namely the formation of additional conductive networks, and the disruption of existing connection networks. The formation of this continuous pathway occurs not only through direct contact between the electrically conductive molecules that distributed in the polycarbonates, but also when the distance between the molecules is very small, so the electrons can easily jump across the gap [77]. Thus, there is a threshold value for the molecular gap, which is electrically equivalent to the molecular contact. The formation of a continuous pathway is facilitated by high pressure by decreasing the molecular gap in the non-continuous zone.

(4-4-2) Dissipation Factor:

The Dissipation Factor or so – called the loss tangent of dielectric (tan δ) of polycarbonate was calculated at different pressures and temperatures by equations (2-13) (2-14).

It is can be define as the ratio of the loss of energy in the electrically insulating materials to the total moving transmitted energy during the dielectric.

Dissipation factor at 50 Hz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	4.42 *10 ⁻¹	4.58 *10 ⁻¹	4.65 *10 ⁻¹	6.93 *10 ⁻¹	1.62		
P=65 MPa	4.57*10 ⁻¹	4.56 *10 ⁻¹	4.67 *10 ⁻¹	7.01 *10 ⁻¹	1.79		
P=69 MPa	5.02*10 ⁻¹	4.79 *10 ⁻¹	5.29*10 ⁻¹	6.92*10 ⁻¹	2.34		
P=73 MPa	4.89 *10 ⁻¹	4.90 *10 ⁻¹	5.46*10 ⁻¹	6.75*10 ⁻¹	2.67		
P=78 MPa	5.20*10 ⁻¹	5.36*10 ⁻¹	5.97*10 ⁻¹	7.29 *10 ⁻¹	3.24		
P=82 MPa	5.52*10 ⁻¹	5.75*10 ⁻¹	6.24*10 ⁻¹	6.97*10 ⁻¹	3.17		

Table (4-10): Dissipation factor as a function of pressure and temperature at 50 Hz.

Dissipation factor at 1MHz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	2.05 *10 ⁻¹	2.42 *10 ⁻¹	2.73 *10 ⁻¹	3.28 *10 ⁻¹	3.69*10 ⁻¹		
P=65 MPa	2.22 *10 ⁻¹	2.24 *10 ⁻¹	2.82 *10 ⁻¹	3.01*10-1	3.71*10 ⁻¹		
P=69 MPa	2.39 *10 ⁻¹	2.44 *10 ⁻¹	2.78 *10 ⁻¹	3.13 *10 ⁻¹	3.90 *10 ⁻¹		
P=73 MPa	2.50 *10 ⁻¹	2.85 *10 ⁻¹	3.01*10-1	2.97 *10 ⁻¹	4 *10 ⁻¹		
P=78 MPa	2.49 *10 ⁻¹	2.57 *10 ⁻¹	3.10*10-1	3.28 *10 ⁻¹	4.80*10-1		
P=82 MPa	2.48 *10 ⁻¹	2.46 *10 ⁻¹	2.70 *10 ⁻¹	3.29 *10 ⁻¹	5.80*10 ⁻¹		

Table (4-11): Dissipation factor as a function of pressure and temperature at 1MHz.

 Table (4-12): Dissipation factor as a function of pressure and temperature at 3MHz.

Dissipation factor at 3MHz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	9.10 *10 ⁻²	9.80 *10 ⁻²	1.02 *10 ⁻¹	1.15*10-1	1.93 *10 ⁻¹		
P=65 MPa	9.34 *10 ⁻²	9.62 *10 ⁻²	1.16 *10 ⁻¹	1.42 *10 ⁻¹	2.10 *10 ⁻¹		
P=69 MPa	9.65*10 ⁻²	1.02 *10 ⁻¹	$1.14*10^{-1}$	1.45 *10 ⁻¹	2.19 *10 ⁻¹		
P=73 MPa	9.92 *10 ⁻²	1.03 *10 ⁻¹	1.23 *10 ⁻¹	1.65 *10 ⁻¹	2.34 *10 ⁻¹		
P=78 MPa	1.02 *10 ⁻¹	1.05 *10 ⁻¹	1.29 *10 ⁻¹	1.80 *10 ⁻¹	2.47 *10 ⁻¹		
P=82 MPa	1.05 *10 ⁻¹	1.07 *10 ⁻¹	1.34*10 ⁻¹	1.95*10 ⁻¹	2.60 *10 ⁻¹		



Figure (4-9) Dissipation factor as a function of pressure and temperature at 50Hz



Figure (4-10) Dissipation factor as a function of pressure and temperature at 1MHz



Figure (4-11) Dissipation factor as a function of pressure and temperature at 3MHz

Table (4-10), (4-11) and (4-12) show dissipation Factor at (50Hz,1MHz,3MHz), It has been shown that the values of dissipation Factor increase with increasing temperature, pressure but decreases with increasing frequency.

We note from figures (4-9), (4-10) and (4-11) that the value of the dissipation factor is high in the low frequencies due to the mobility of dipoles and therefore not to keep up with the poles of the electrode field and the dissipation factor stays fairly stable at high frequencies [78].

The relationship between dissipation factor and temperature was studied in the last three forms. This relationship is typical of polar insulators, thereby the increasing the temperature lead to facilitated of the direction of the dipole momentum. The increase in the dissipation factor may also result from an increasing in crystallization [79], but not in noncrystalline polycarbonates.

The pressure increases with increasing dissipation Factor. Where the pressure leading to a rearranging in the conductive molecules and hence the dissipation factor of the PC increase [80].

(4-4-3) Dielectric constant (ε):

The real dielectric constant of polycarbonate was measured at different pressures and temperatures by equation (2-13) (2-14).

Real dielectric constant at 50Hz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	6.56*10	4.56 *10 ²	1.03 *10 ³	4.42 *10 ³	4.52 *10 ⁴		
P=65 MPa	9.08*10	7.72 *10 ²	1.08 *10 ³	5.09*10 ³	4.98 *10 ⁴		
P=69 MPa	1.16*10	1.09 *10 ³	1.12 *10 ³	5.75*10 ³	9.47 *10 ⁴		
P=73 MPa	$1.41*10^{2}$	1.40 *10 ³	1.17 *10 ³	6.42 *10 ³	1.50 *10 ⁵		
P=78 MPa	1.67 *10 ²	1.72 *10 ³	1.21 *10 ³	7.09 *10 ³	1.85 *10 ⁵		
P=82 MPa	1.92*10 ²	2.04 *10 ³	1.26 *10 ³	7.76 *10 ³	2.29*10 ⁵		

Table (4-13): Real dielectric constant as a function of pressure and temperature at 50Hz.

Real dielectric constant at 1MHz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	3.44	4.39	4.67	6.24	7.01		
P=65 MPa	3.92	4.5	4.99	6.27	7.08		
P=69 MPa	4.41	4.62	5.3	6.3	7.16		
P=73 MPa	4.54	4.7	5.86	6.79	7.23		
P=78 MPa	4.67	4.79	6.41	7.28	7.31		
P=82 MPa	4.81E	4.88	6.97	7.3	7.38		

Table (4-14): Real dielectric constant as a function of pressure and temperature at 1MHz.

Table (4-15): Real dielectric constant as a function of pressure and temperature at 3MHz.

Real dielectric constant at 3MHz							
T/P	T=25 °C	T=50°C	T=100°C	T=150°C	T=250°C		
P=61 MPa	1.55	1.92	2.62	2.92	3.29		
P=65 MPa	1.64	2.03	2.64	2.99	3.37		
P=69 MPa	1.67	2.36	2.83	3.28	3.38		
P=73 MPa	1.74	2.54	2.9	3.42	3.44		
P=78 MPa	1.8	2.76	3	3.6	3.49		
P=82 MPa	1.86	2.98	3.11	3.78	3.53		







Figure (4-13) Real dielectric constant as a function of pressure and temperature at 1MHz





Table (4-13), (4-14) and (4-15) and show real dielectric constant at (50Hz,1MHz,3MHz), It has been shown that the values of real dielectric constant increase with increasing temperature and pressure but decreases with increasing frequency.

It can be observed in figures (4-12), (4-13) and (4-14) that the ε' increasing as the temperature increases and thus facilitates the routing of the dipoles. Thus we conclude that the segmental movement is the dominant mechanism [81, 82].

The inverse relationship between the ε' and frequency can be explained by the fact that at low frequencies the time period is sufficient for the dipoles to arrange the molecules and align them to the current between the poles. At high frequencies, however, the time period is short and less than the time required to align the molecules so that they can align themselves with the direction of the electric field external [78].



Chapter Five

Conclusion & Suggestions for Future Works

5-1 Conclusions:

- 1- The heat treatment of polycarbonate improve the degree of crystallinity of it, which will but decreases when the pressure on the sample increases.
- 2- The best method of measuring crystallinity is to construct the complete (XRD) diagram and compare it with reliable theoretical values, such as what can be obtained from the (ATHAS) database.
- 3- When the samples are heated, the thermal energy of the molecules will be increased and thus the cell of the unit will expand causing changes in the thermal conductivity-spacing, interchain distance and the grain size. Conversely, when the pressure increases, the covalent bonds between the molecules will be broken.
- 4- All the electrical properties that are included loss tangent of dielectric, real dielectric constant and ac-conductivity were found to increase with increasing temperature and pressure.
- 5- The ac-conductivity of (PC) as a function of frequency was calculated and it was observed to increase as frequency increased.
- 6- The real dielectric constant and loss tangent of the dielectric for PC as a function of frequency was calculated, and it was noted to reduce as frequency increased.

5-2 Suggestion:

1- Study the effect of rainwater and saline water on Nano polycarbonate with different tests.

2- Study the effect of irradiation on the optical properties of polycarbonate.

3- Study the effect of climatic conditions on the mechanical and structural properties of polycarbonate.

4- We can employ other polymeric materials and make the same measurements like:

-Polyethylene (PE)

-Polyethylene terephthalate (PET)

-Polybutylene terephthalate (PBT)

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الخلاصة :

تم في هذا البحث دراسة الخواص التركيبية (Structural Properties) والتي تضمنت حيود الاشعة السينية (XRD) ,الخواص الكهربائية (Electrical Properties)) والتي تضمنت كل من :التوصيلية الكهربائية (σ_{ac})و ثابت العزل الحقيقي (ε') و عامل الفقد (tan δ) ,الخواص الحرارية (κ اليوليي الحرارية (K اليوليمر البولي الحرارية (Nano polycarbonate)) والمتضمنة التوصيل الحرارة الحرارة والضغط في فن الوقت, حيث كانت قيم الضغوط في هذا البحث (RT, 50,100,150,200,250) و قيم درجات الحرارة (RT, 50,100,150,200,250) .

ان المعلمات التركيبية التي تم حسابها من حيود الأشعة السينية هي كل من الحجم الحبيبي (Grain)، درجة التبلور (d-spacing) , المسافة البينية (d-spacing) و المسافة بين الذرات (Inter-chain distance) وقد وجد ان جميع المعلمات السابقة تزداد بارتفاع درجات الحرارة وتقل مع زيادة الضغط.

اظهرت نتائج الخواص الكهربائية ان قيم التوصيلية الكهربائية تزداد مع زيادة الضغط ودرجة الحرارة والتردد الا ان قيم عامل الفقد وثابت العزل الكهربائي قد اظهرت تناسبا عكسيا مع التردد و وتناسبا طرديا مع الضغط ودرجة الحرارة.

اما بالنسبة الى نتائج الخواص الحرارية فقد اظهرت ان التوصيلية الحرارية تتناسب عكسيا مع الضغط وطرديا مع درجة الحرارة.



جمهورية العراق وزارة التعليم العالي والبحث العلمي جامعة بغداد كلية التربية للعلوم الصرفة /ابن الهيتم قسم الفيزياء

دراسة تأثير المعاملة الحرارية والضغط على الخواص التركيبية و الفيزيائية للبوليمير بولي كاربونيت النانوي

رسالة مقدمة الى مجلس كلية التربية للعلوم الصرفة / ابن الهيثم – جامعة بغداد وهي جزء من متطلبات نيل درجة ماجستير علوم في الفيزياء

من قبل

محمد مثنى غازي

بكالوريوس علوم فيزياء / ٢٠١٦

باشراف

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