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The effect of adding nanoparticles on the tensile strength of a polymeric material

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بِسْمِ اللهِ الرَّحْمنِ الرَّحِيمِ

يَرْفَعِ اللَّهُ الَّذِينَ آمَنُوا مِنكُمْ وَالَّذِينَ أُوتُوا الْعِلْمَ دَرَجَاتٍ

صدق الله ألعلي العظيم

سورة المجادلة، آية (11)

الأهداء

(وَآخِرُ دَعْوَاهُمْ أَنِ الْحَمْدُ لِلَّهِ رَبِّ الْعَالَمِينَ)

حمد لله الذي ما تمّ جُهد ولا ختم سعي إلا بفضله، الحمد لله على البلوغ ثمّ الحمد لله على التمام

الى من أعجز عن وصفه سندي وملجأي بعد الله

أبي (حفظه الله)

الى الجسر الوحيد الذي جعلني أصل لمرحلة التخرج هي

أمي أغلى ما في الكون

الى من ساندني ودعمني في طيلة مسيرتي هذه

الدكتور مشتاق عبد الكريم حسين البحراني

الى من شهدوا معي متاعب الدراسة وسهر الليالي، من كانوا خير عون لي في دربي ..

أخوتي الأعزاء

الى الأصدقاء الأوفياء، الذين ما انفكّوا يومًا عن تقديم العون والمساعدة والدعم لي في أحلك الظروف

الى الذين حصرهم قلمي ولم يحصرهم قلبي

الى اساتذتي و زملائي اهديهم جميعا حروفي ..... حبا و وفاءاً

Acknowledgements

First of all, I would like to thank Allah the great creator of the universe for continuous helping, supporting and blessing.

I would like to thank my supervisor, **Dr. Mushtaq Abdul Kareem Hussein AL- Bahrani** for his very valuable advice, support, friendship, and tuition during the period of this study.

Also, I would like to thank the Dean of College of Engineering **Prof. Dr. Abbas Oda Dawood** and special thank for the head of Department of Mechanical Engineering **Dr. Mahmoud Shaker Mahmoud** for supporting and facilitating the achievement of this work.

I would like to express my sincere gratitude to the staff of the laboratories in Materials Engineering College in the Babylon University.

Finally, I would like to thank my family for their support, encouragement and patience throughout the achievement of this study.

ABSTRACT

PMMA material is used in many engineering industries and applications such as cars, airplanes, traffic lights, light advertisements, lenses, eyeglasses, solar cells, dental industry, many electrical appliances and construction applications such as decorations , commodity displays, sports swimming pools, and golf courses, as well as in many agricultural fields. The PMMA material is characterized by abundance, cheap price, low density, good relative durability, ease of forming and cutting to desire sizes and shapes required, non-toxicity, electrical insulation and chemical resistance, but it's the tensile strength resistance is weak.

Getting high-quality surfaces of PMMA has become a priority for both the manufacturers and the researchers alike for two reasons, one functional and the other aesthetic, because the scratches on the PMMA surface as a result of weak tensile strength reduce their use in the optical industry and many engineering applications, as the presence of scratches leads to increase stress during tensile, impact and fatigue loading that undermines the longevity of PMMA during use. In the current study, silicon oxide nanoparticles (SiO_2) was added in ratios of (1, 2, 3 and 4wt %) in order to study the effect of SiO_2 on the tensile strength resistance of PMMA.

The pure and reinforced PMMA specimens were prepared in dissolving method. Also, all necessary equipment for preparing samples for tensile strength test of PMMA has been designed and manufactured.

The results obtained from the tests conducted an improvement of the tensile strength for PMMA/SiO_2 composites (S1, S2, and S3 specimens) while a decrease in tensile strength was observed for the S4 specimen compared to pure PMMA (S0 specimen). A significant increase in ultimate tensile strength can be observed for PMMA/SiO_2 composite by 1wt% compared with the other ratios of SiO_2. Any other increase in SiO_2 nanoparticles content above 1wt% imposes an adverse effect by reducing the ultimate tensile strength.

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Chapter One
Introduction

Chapter One

Introduction

1.1 Background

The polymer is a term containing two sections: poly which means multi or many, and mer which means part or binary unit. Polymer is a chemical compound that contains large molecules made of many smaller molecules of the same type called monomers; some polymers are naturally present and others are produced in laboratories and factories in a way called the polymerization process. Most polymers are organic (i.e. depended on a carbon chain) however there are also non-organic polymers whose chains are depended on the silicon origin. The polymers are formed as a long chains, and human has discovered the polymeric materials in nature in the past, such as starch, fibers, rubber and gum, and in the 19th century, scientists carried out imitating nature to produce industrial polymers. In the 20th century, when the need for rubber increased, German scientists managed produce artificial rubber with the same composition of polymer which is characterized by the chain length. The polymer industry has grown and developed so far that it has become bigger than the iron, copper and aluminum industries, and even bigger than all industries [1].

The polymer industry has expanded in all applications of human life, surpassing any other type of material, they used in the manufacture of adhesive materials, foam materials, paints, packaging materials, textiles, artificial fibers, composite materials, as well as electronic, biomedical, and optical devices, and many high precision technology products [2].Two important properties determine the most important characteristics of polymer which are the temperature of glass transition and the average of molecular weight.

The temperature of the glass transition is defined as the temperature at which the mechanical properties of the polymer change significantly. If the

temperature of the glass transition is less than the temperature of the room, the polymers act as an elastic matter (thermoplastic). While the temperature of the glass transition is higher than the temperature of the room, the polymers act as a rigid matter (thermoset). A second important characteristic of polymers is the molecular weight since the chemical and mechanical properties of a polymer such as its viscosity, resistance to creep, resistance to abrasion, etc. are determined by the weight of molecular of the polymer.

Polymers can be classified depending on the source origin, structure, forces of molecular and type of polymerization, the chart in figure (1-1) shows this classification. Polymers can be classified depending on structural composition into linear, branched, and crosslink polymers, figure (1-2) shows this classification. When the polymer is composed of one type of repetitive unit (monomer), the polymer is called the homopolymer. When the resulting compound is composed of two types of repetitive units, the polymer is called the copolymer; either when the resulting composite consists of three repetitive units the polymer is called a terpolymer, as shown in figure (1-3). Technological classification of polymers includes thermoplastic and thermoset polymers [3]. Thermoplastic category includes polymers that are affected by heat becomes in the molten- state when the temperature rises and the polymers will return to its solid state when the temperature is lowered. Thermoset polymers undergo chemical changes when they are heated their chains are intertwined and the polymers become poor conductivity of the electric and heat when they are heat-treated [4].

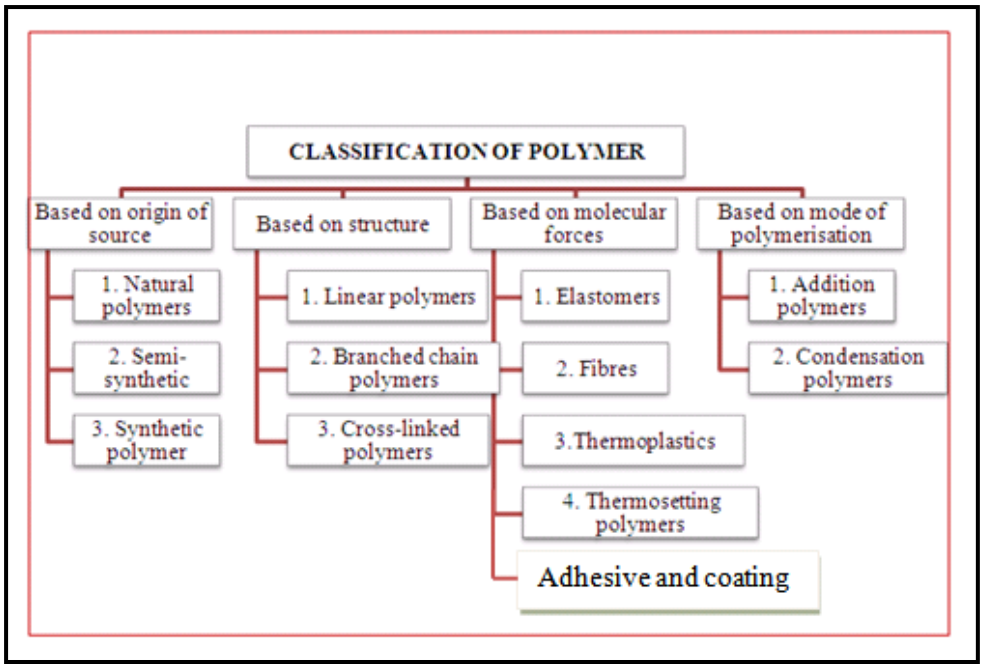

Figure (1-1) General classification of polymers [5].

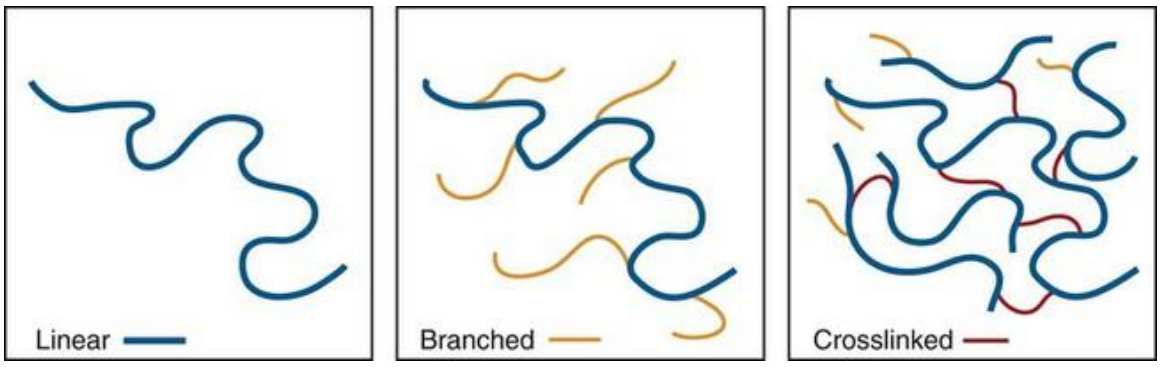

Figure (1-2) Polymers classification depending on structural composition [6].

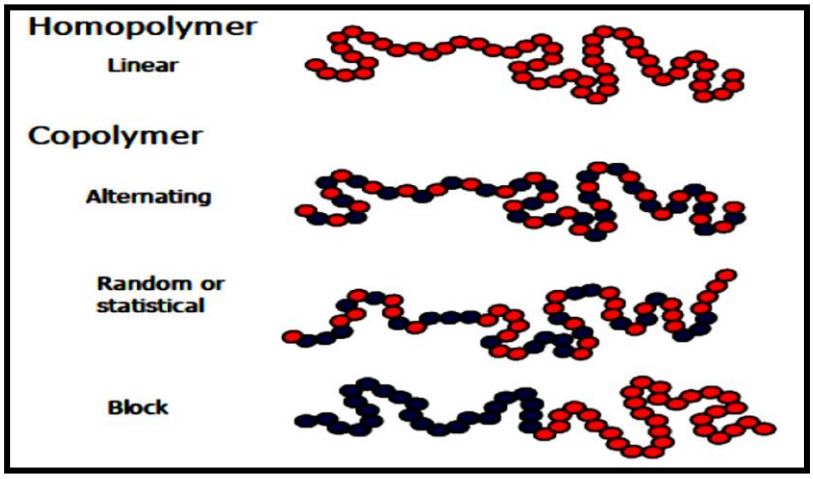

Figure (1-3) Classification of the polymers depending on homogenization [7].

1.2 Polymerization

The simple molecules from which the polymer molecules are built are called monomers. Binding these simple molecules together is called the polymerization process. The monomers are simple chemical compounds that have small molecular weights. The molecules of these compounds are characterized by special structures that enable interaction with other molecules of the same type or molecules of another compound under the appropriate conditions to form the polymer chains. So, the polymerization can be defined as a chemical union for two-part or more of one or more substances with a small molecular composition to form a compound with a great molecular mass and different in its chemical and physical properties from its constituent compounds. Polymerization reactions are classified into two types which are illustrated in the figure (1-4).

1. Polymerization by addition.
2. Polymerization by condensation.

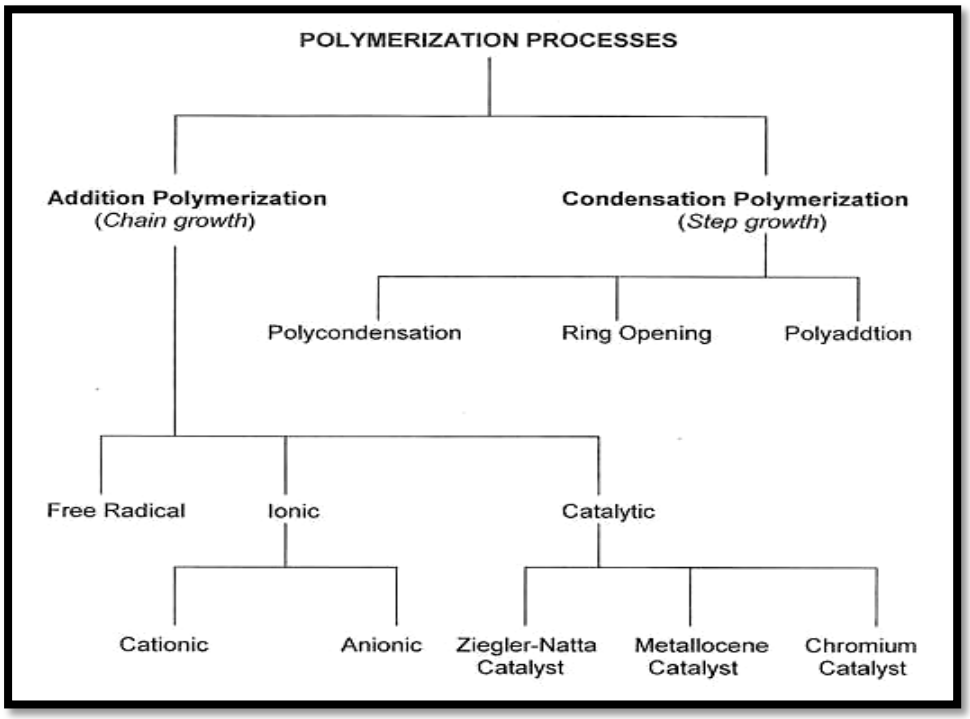

Figure (1-4) Classification diagram of polymerization process [8].

The process in which polymers are produced as a result of adding monomers to each other rapidly and sequentially is called addition polymerization. Passing every moment, a new active center bond is formed that enhances the interaction of additional monomers. The end result of this polymerization is a long polymer chain with a high molecular weight and containing the same number of primary "monomer" atoms. Thus, the molecular formula of the repeated unit in the polymer molecule is similar to that of the monomer. All methods of addition polymerization consist of three stages are:

1. Initiation step: where active stations are formed.
2. Propagation or diffusion step: where the polymers with a high molecular weights are formed.
3. Termination step: where the reaction ends with the disappearance of the active centers by interactions whose nature depends on the kind of active center and the conditions of reaction.

The process in which polymers are produced as a result of the interaction of functional groups in the structure of two molecules of monomers is called condensation polymerization. The final hull is a long polymer chain with a high molecular weight, containing fewer atoms than interacting monomers, with secondary products being made up of small molecules such as water molecules, hydrogen chloride, methanol, etc. The molecular formula of the unit repeated polymer molecules are smaller than the total of two monomer formulations united in the condensation process [8].

The polymerization method was not used in the preparation of PMMA specimens in the current work due to the difficulty of this method, shortage of time and the lack of necessary equipment, but the pure and composite specimens were prepared in casting method, this method is explained in detail in chapter three.

1.3 Organic solvents

Organic solvents are chemical substances with evaporation and volatilization characteristics such as benzene, chloroform, acetone and some cleaning fluids. Organic solvents are organic liquids that have the ability to dissolve other organic and inorganic substances, without changing their chemical properties. This characteristic has led to the use of solvents in many important industries. When selecting the appropriate solvent for organic compounds, follow the rule (like dissolves like) this rule requires knowledge of some characteristics of organic compounds for dissolved in an appropriate solvent chemical reactions. Solvents generally have a low boiling point, evaporate easily or can be isolated by distillation, leaving dissolved solids behind. In order to prevent the dangers of organic solvents, the following points should be considered [1]:

1. The hazardous organic solvent must be replaced with other solvents that are less hazardous to the life of the worker or manufacturer.
2. Ventilation factors should be provided where organic solvents are used.
3. Explain the risks of organic solvents to their users and teach them ways to prevent these risks.
4. Warning signs should be placed in factories that produce hazardous materials.

The solvents used in this work were tetrahydrofuran, acetone, and isopropanol, the specifications of which will be explained in detail in chapter three.

1.4 Polymethyl methacrylate (PMMA)

Polymethyl methacrylate is a thermoplastic transparent plastic notable a lot of unremarkably by the brand "Plexiglass, Lucite, Acrylic, Polycast, Optix, and PMMA". When the large impact resistance is not required of polycarbonate (PC) the acrylic is more suitable to use as alternate to the glass because of the acrylic is analogous to PC in this case. It had been initially made in 1928 and was placed on the market in 1933 by Hass and Rohm. Usually, in the market may be PMMA one amongst more evident plastics types. The primary application of PMMA was in World War II, it had been used for airplanes windows, periscopes of submarine, canopies, and turrets. The eyes of the pilots, who were harmed by acrylic shrapnel, were better than those of the pilots who were harmed by glass shrapnel because the PMMA is breaks into large pieces instead of small shrapnel [9]. Acrylics are used at present for a set of applications that usually benefit from their natural transparency and resistance to the influence of some variables. Common uses include furniture, LCD screens, security barriers, paint, lenses, and medical devices. Because of their clarity, they are oftentimes used for attachments around the exhibits and windows.

PMMA is defined as "thermoplastic" (as against "thermosetting"), and the name depends on the method that the plastic responds to heat. At the melting point the thermoplastic material converts to liquid, the foremost advantage of thermoplastic is maybe liquefying, refrigerated, and reheated without significant disintegration. This feature allows the thermoplastic to be injected into the molds and then recycled instead of burned. On the contrary, thermosetting can only be heated once (usually during casting in molds or injection). The result of the first heating leads to an irreversible change in the chemical properties of the material, if the material is heated again, it will simply be burn. This feature makes thermosetting material a weak candidate for recycling [4].

The PMMA raw material allows for internal light movement with approximately the same glass capacity, making it a great alternative, and this is exactly the same for polycarbonate. PMMA is available in a set of colors (possibly transparent and possibly opaqueness). The major differences include that acrylic does not contain potentially hurtful Bisphenol-A (BPA), while polycarbonate has greater impact resistance also acrylic is cheap and readily available. If impact resistance is an unimportant factor so PMMA can be used as an alternative to polycarbonate. For a dual performance combination of polycarbonate's impact resistance and acrylic's scratch and penetration resistance, a layer of acrylic is added to the surface of polycarbonate. Several bullet-proof glass is made in this manner. The PMMA on the surface used to resist scratching and penetration throughout daily use, the Polycarbonate stops a bullet [9].Table (1-1) represents the PMMA properties; figure (1-5) shows the molecular structure of PMMA.

Table (1-1) Properties of PMMA [1].

Properties	Values
Technical Name	Acrylic (PMMA)
Chemical Formula	$(C_5H_8O_2)_n$
Melt Temperature	130°C (266°F)
Typical Injection Mold Temperature	79-107°C (175-225°F)
Heat Deflection Temperature (HDT)	95°C (203°F) at 0.46 MPa (66 PSI)
Tensile Strength	65 MPa (9400 PSI)
Flexural Strength	90 MPa (13000 PSI)
Specific Gravity	1.18 g cm^{-3}
Shrink Rate	0.2 - 1% (0.002 - 0.01 in/in)

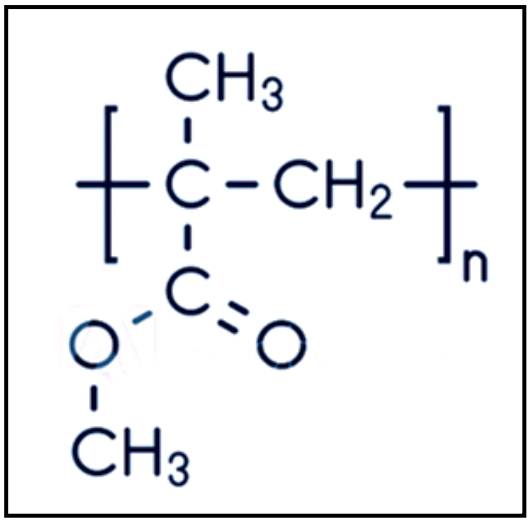

Figure (1-5) Molecular Structure of PMMA [1].

1.5 The importance of the tensile strength resistance of polymers

Obtaining quality surfaces of polymeric materials from an aesthetic and functional point of view has become one of the most important characteristics that determine the choice of these materials in many industries and engineering applications. Producing a desired layer for the polymer surface is a very difficult process, as the difficulty lies in the ability to maintain the surface quality throughout the polymer life cycle during service. One of the main reasons that lead to a decrease in the surface quality of polymeric materials is scratches that are a form of surface deformations. Surface quality characteristics can be categorized based on the applications of polymer into aesthetics of surface, structural integrity of surface, surface durability [10, 11].

Aesthetics of surface, such as the interior and exterior parts of automotive (car dashboards and lighting lamps), covers (housing) for electronic and electrical products and telecommunications devices (cellular phones) , are very important because the scratches of surface may reduce the value of these products although their intended functions are still generally unaffected. In applications of coating, the presence of scratches on the surface of the polymeric

coating causes damage to the wooden substrate or corrosion of the metal. Therefore, the coating layers must maintain their mechanical integrity throughout their expected service life. For applications such as the use of polymeric materials in food packaging, the structural integrity of the packaging membranes is a matter of great concern and important for maintaining food quality and safety. The scratches formed on the food packaging membranes may lead to rupture them and thus spoilage the food inside. In the industry of data storage, surface durability is necessary as scratches can cause permanent data loss from optical storage devices and hard drives. From the point of view of structural integrity, another concern could be added that scratches act as concentration points for stresses. These points lead to a decrease in load bearing capacity that ultimately causes early fracture and structural component failure. Also, scratch damage can extend to nanotechnology devices and precision mechanical devices, as the scratches formation in these devices can lead to completely lose their functions because of the small scale of this type of devices.

From the above it is clear that the tensile strength study is a very important issue for many industries and engineering applications. Attention to the tensile strength and surface quality of polymers has emerged only in the past few decades and has increased in recent years as a result of advances in science and technology of polymers. Research interest in the study of the tensile strength and surface quality of polymers can be represented by the number of research publications related to this topic over previous years as shown in figure (1-6).

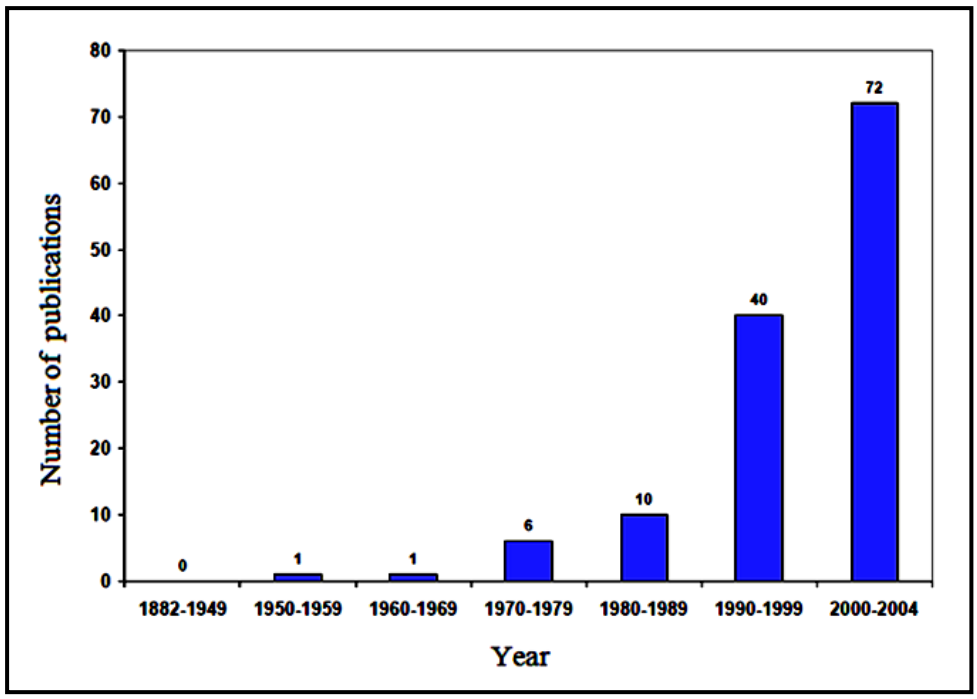

Figure (1-6) Progress of tensile strength and surface quality research over past few decades [11].

1.6 Methods of adding nanofillers to polymers

Polymeric composites (filler/polymer nanocomposites) have received much attention in recent times due to their exciting and beneficial characteristics such as mechanical properties, thermal resistance and chemical resistance. Polymeric composites are manufactured by adding fillers to the polymer. The purpose of adding fillers with different types and ratios is to improve the desired property in the original polymer. The difficulty of this process lies in dispersing the inorganic filler in the organic polymer matrix and finding the appropriate method for dispersion in order to obtain the required nanocomposite. The methods used to produce nanocomposites (for example PMMA/SiO_2 nanocomposites) can be classified into the following methods [12]:

1. Interpolating (intercalation) method
2. Local (in situ) polymerization method
3. In situ formation method for both the nanofillers and polymerization.
4. The direct mechanical mixing method of the polymer and the nanofillers.

Some of these methods require the modification of nano-filler surfaces and/or complex polymerization reactions, which makes them unsuitable for the industrial production of nanocomposites. The procedures for producing SiO_2/PMMA nanocomposites used in the current work were carried out according to the fourth method, which is illustrated as follows:

The fourth method is based on the breaking of the filler agglomerates resulting from the direct mixing process of both the nanofillers and polymer matrix. This method is more suitable for producing filler/polymer composites that contain a nano or micron - scale fillers. In general, two ways are used to mix fillers and polymer. The first is without using any solvents called (powder mixing method) [13, 14]. The second uses solvents to mix the fillers and polymer as a solution called (solution mixing method) [15, 16].

The fourth method was adopted in the current work to produce the SiO_2 / PMMA composites for several reasons, including the simplicity of this method, availability of required equipment, and SiO_2 used is nano-filler.

1.7 The aim of the present work

The current work aims to improve the tensile strength resistance of PMMA by adding SiO_2 nanoparticles and study the factors affecting the tensile strength resistance such as preparation techniques and SiO_2 ratios

The main aim of the present study is achieved by performing the following objectives**:**

1. Preparing new PMMA/SiO_2 composite materials to improve the tensile strength resistance of PMMA.
2. Study the tribological and mechanical properties of composite materials prepared using tribological and mechanical tests.

3. Study the parameters affecting the tensile strength resistance such as preparation techniques of PMMA/SiO_2 composites and SiO_2 ratios.
4. Design and manufacture all the necessary equipment's for the current work such as glassy and metallic molds, glassy containers, horizontal leveling platforms, and nanoparticles dispersing device.
5. Conducting a tensile strength resistance test using a universal test apparatus for pure and reinforced samples and making a comparison between the results and discussing them.

1.8 Layout of the project

The present work is divided into five chapters:

Chapter one describes the introduction and the objectives of the study.

Chapter two presents the most important research on tensile strength resistance .

Chapter three describes the preparation method, tools, and materials used in the preparation of PMMA samples.

The experimental results of the tensile strength test of PMMA samples with different ratio of SiO2 are presented and discussed in the chapter four.

The conclusions and recommendations of the current work are included in chapter Five.

The references are listed at the end of this work.

Chapter Two
Literature Review

Chapter Two

Literature Review

2.1 Introduction

A review of published researches dealing with the development of scratch behavior and the mechanical and tribological properties as a result of adding nanoparticles of PMMA provides a basis for understanding the tensile strength behavior of this substance in the current study. In this chapter, published research papers dealing with mechanical and tribological tests of pure and reinforced PMMA by adding nanoparticles in different ratios and research dealing with PMMA tensile strength behavior and the different devices used to perform the tensile strength test were presented.

In 2014, Saeed Shirkavand and Elnaz Moslehifard [17]studied the effect of titanium dioxide (TiO_2) nanoparticles addition on tensile strength of polymethyl methacrylate (PMMA) resin used in the dental industry. The PMMA resins were widely used in the manufacture of temporary base materials used in the manufacture of dental and prosthetic alternatives due to their optical, aesthetic and biocompatibility properties. According to standard ISO 1567, the 36 samples of the tensile tests were prepared, 9 samples of pure PMMA resin and 9 samples for each ratio of TiO_2-reinforced PMMA resin (0.5, 1, and 2 wt% TiO_2). Using a testing universal machine type Zwick Z100, Germany, the tensile test was obtained of samples under the load of 10 kN and speed of 5mm/min. The results of the tensile test showed a significant increase in tensile strength values of the reinforced PMMA resin specimens by 1wt% TiO_2 nanoparticles comparison with the pure and reinforced by another ratio of TiO_2 for the PMMA resin. Also, any another increase in TiO_2 nanoparticles content above 1wt% imposes an adverse effect by reducing the tensile strength due to the particles agglomeration that act as tense defects and concentration points.

Sura H. Ahmed in 2022[18], studied the tensile strength, modulus of elasticity and elongation at break for acrylic resin (PMMA) when the expansion different proportions (0.1, 0.2 and 0.3 %) from two sorts of nanoparticles (Walnut shell particles (WSP)) and (talc particles TP). The resulted show, the expansion of the nanoparticles (WSP and TP) to the polymer matrix (PMMA) lead to expanded tensile strength and modulus of elasticity while elongation value diminished, Such conduct might be because of the great and uniform dissemination of nanoparticles and decreases diminished, Such conduct might be because of the great and uniform dissemination of nanoparticles and decreases agglomeration (grouping) of the nanoparticles and that may prompt lessen interior stress fixation in Nano composite materials close to the bunching nanoparticles and such little inner stress are insufficient to break the interactions at the area of the interface. It was seen that the polymer Nano composites ((PMMA): X% WSP) get the higher estimations of (tensile strength and modulus of elasticity) separately, however, lower value of the elongation rate as contrasted and their partners of the other kind of nanoparticles (TP). This is because of the strengthening mechanism by this powder, which plays to limiting the slipping of (PMMA) chains, additionally natural of the bond between the matrix, and reinforced materials, which relies upon capacity of PMMA to separate between strong particles as good wettability that led to the invigorating high bond between the matrix and reinforced materials. Just as, the figures show that the most elevated value of (tensile strength & modulus of elasticity) can be obtained of the Nano composite content 0.3% proportion of walnut shell particles was reached to (28 MPa) and (1.28 GPa) respectively, while the most elevated value of (tensile strength & elasticity modulus) can be obtained of the Nano composite content 0.3% proportion of talc particles was reached to (27.5 MPa) and (1.2 GPa) respectively.

Jirun Sun et al. (2011) [19], improved the performance of dental resins by adding a small amount of titanium dioxide nanoparticles (TiO_2 NPs), which have outstanding mechanical properties and unique photo activities. Acrylic acid modified TiO_2 NPs (AP25) were prepared and added to a mixture of bis-phenol-A-methacrylate and triethylene glycol methacrylate (mass ratio 1:1) at seven mass fractions. Disks made of these resins were subjected to FTIR micro spectroscopy, Nano indentation, micro indentation, and 3-point bending to determine the degree of vinyl conversion (DC) modulus and hardness. The shear bond strengths (SBS) of dentin adhesives containing various amount of AP25 were also examined Results. The DC increased as a function of mass fraction of AP25 and reached a plateau at 0.1%. The DC of the resin mixture was improved by ≈7% up to 91.7 ± 0.8%. The elastic modulus and hardness of the composites increased initially as more AP25 were added, and decreased after reached the maximum value at approximately 0.06% mass fraction of AP25. The maximum elastic modulus was ≈48% higher than that of the NP-free resin, and the maximum hardness was more than twice higher than that of the NP-free resin. Using these resin composites as dental adhesives, the mean SBS using resins with 0.1% mass fraction of AP25 was ≈ 30% higher than those using NP-free resin Significance. By adding a small amount of AP25 to the resin, the DC and the mechanical properties of resins were improved dramatically. These findings could lead to better performing dental adhesives.

In 2013,Ahmad Sodagar et al. [20], studied the effects of TiO_2 and SiO_2 nanoparticles on flexural strength (Fs) of (polymethyl methacrylate) acrylic resins. Acrylic specimens (Selecta Plus) in size of 5x10 (-+0.2) x 3.3(-+0.2) mm were prepared and divided into 7 groups: AR containing nanoTiO2, SiO2 and TiO_2 with SiO_2 in two concentration of 1% and 0.5%, in addition to a control group. To prepare Nano AR, nanoparticles were added to the monomer. All specimens were stored in 37 8C-distilled water and underwent Fs test by

universal testing machine (Zwick). The maximum mean flexural strength (43.5 MPa) belongs to the control group and AR containing 0.5% of both TiO_2 and SiO_2 demonstrated the minimum mean Fs (30.1 MPa). Resins contained TiO_2, demonstrated lower values of Fs than those contained SiO_2 with the same concentration, but the differences were not significant ($P > 0.05$). Conclusion: Incorporation of TiO_2 and SiO_2 nanoparticles into acrylic resins can adversely affect the flexural strength of the final products, and this effect is directly correlated with the concentration of nanoparticles.

Yang Xia et al. (2008) [21], improved the mechanical properties of dental resin-based composites(RBCs, Z100, 3 M ESPE) usingTiO_2 nanoparticles treated with the organosilane allyltriethoxysilane (ATES) . TiO_2 nanoparticles were sonically dispersed in an ethanol solution containing ATES. The modified particles were washed in pure ethanol and dried before being used as filler. Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM) were used to analyze the nanoparticles. Five groups of composite resin specimens were prepared: one control group, and four groups using either modified or unmodified nanoparticles comprising 0.5% or 1.0% of the material by weight. The mechanical properties micro hardness and flexural strength) of all five groups of specimens were measured. After modification, the particles' FTIR spectrum shows a new absorption doublet at 1200 and 1020 cm1. TEM images show that the modified particles have better dispersion. and that their clusters are small enough to create a homogeneous surface on dental RBCs. Composite resin specimens including modified nano-TiO_2 have significantly better mechanical properties than the control group ($P < 0.05$). The improvement of adding 1.0% wt modified nano-TiO_2 particles was better than that of 0.5 %wt. modified nano-TiO_2 particles was better than that of 0.5 wt% . Surface modification by the organosilane ATES influences the dispersion and

linkage of TiO_2 nanoparticles within a resin matrix, and the modified particles are found to improve the micro hardness and flexural strength of dental RBCs.

In 2010, Amit Chatterjee. [22], investigated the matrix properties by introducing Nano size TiO_2 (5 nm, 2.0–30% by weight) filler into (PMMA) resin. A twin screw extraction process was developed to disperse the particles into the PMMA. The thermal, mechanical, and viscoelastic properties of the virgin PMMA and nanoTiO_2-PMMA composite were measured. The Nano filler infusion improves the thermal, mechanical and viscoelastic properties of the PMMA. Nano composite shows increase in storage modulus (60%), rubbery modulus (210%), glass transition temperature (27%), crosslink density (213%), initial decomposition temperature (83% at 1% wt. loss), and activation energy (141%). Mechanical performance and thermal stability of the nanoTiO2-PMMA composites are depending on the dispersion state of the TiO2 in the PMMA matrix. Scanning electron microscopic study shows that the particles are well dispersed in the PMMA matrix. They are correlated with loading. Kinetics for thermal degradation analysis was studies. The integral procedural decomposition temperature (IPDT) is enhanced (117%). The Nano composites of high activation energy possess high thermal stability. Interrelation of Tg, crosslink density, IPDT, storage modulus, activation energy, and TiO_2 weight percent are established various reasons for these effects in terms of reinforcing mechanisms have been discussed.

Amit Chatterjee, (2010) [23], studied the effect of TiO_2 Nano fillers (5 nm, 0 –15% weight) have been introduced in the PMMA matrix using a twin-screw extruder to increase the performance of PMMA. The twin-screw extrusion process is optimized to disperse the particles into PMMA. Nano filler infusion improves the thermal, mechanical, and UV absorption properties of PMMA. TiO_2-PMMA Nano composites exhibit the increase in tensile modulus (~90%),

decomposition temperature (~31%), dimension stability (60%) and UV absorption (410%). Properties of the nanoTiO_2-PMMA composites are depending on the dispersion of TiO2 in the PMMA matrix. It is interrelated with loading. Formation and disappearance of the peaks in FTIR confirm the chemical interaction of PMMA.

In 2009, L. Reijnders [24], developed the Nano composites consisting of organic polymers and TiO_2 or amorphous SiO_2 nanoparticles. These nanoparticles may be released from nanocomposites. There is evidence that amorphous SiO_2 and TiO_2 nanoparticles can be hazardous. Thus, in the design of nanocomposites with such nanoparticles, hazard reduction extending to the full Nano composite life cycle would seem a matter to consider. Options for hazard reduction include changes of nanoparticle surface structure or composition, better fixation of nanoparticles in nanocomposites, including persistent suppression of oxidative damage to polymers by nanoparticles, and design changes leading to the release of relatively large particles.

Sushil Kumar Singh (2018) [25], obtained a comparison of the tensile and flexural properties of SiO_2/epoxy polymer nanocomposites. Dispersion of SiO_2 nanoparticles in the epoxy polymer was achieved by ultra-sonication. SiO_2/epoxy nanocomposites contain varying amount of nano size silicon dioxide (SiO_2) up to 8 wt%. The tensile test and three-point bending flexural test were performed to obtain the tensile strength and the flexural properties of nanocomposites respectively. The investigated properties of SiO2/epoxy nanocomposites increases with the increasing nanoparticles dispersion up to 4 wt.% SiO_2 nanoparticles and deterioration in the mechanical properties is realized above 4 wt.%. This may be due to the significant increase in agglomeration and settlement of the SiO_2 nanoparticles during the long curing

time. The tensile strength increases by 30.57 %, flexural strength by 17 % and flexural modulus by 76 % for 4 wt. % dispersion of SiO_2.

Azza Walaaldeen Khairi1, (2022) **[26]**, studied the reinforce high impact acrylic dentures with rice husk-derived silica in various concentrations to improve some of the mechanical qualities of the denture base. Based on the pilot study made in College of Dentistry University of Baghdad, to create the specimens, silica particles from a rice husk source were added to high impact heat cure resin at 1 wt.% and 3 wt.% (by weight). On the basis of the three tests conducted the transverse strength, impact strength and hardness tests, the prepared samples were evaluated using Fourier-transform infrared spectroscopy (FT-IR), particle size analysis, and a field emission scanning electron microscope (FE-SEM). The results revealed that the addition of silica particles yielded a significantly improvement in transverse strength test (P-value= 0.001), and non-significantly increase in both impact strength and hardness values compared with the control group. Silica particles prepared from natural resource waste was successfully added into the high impact acrylic resin. It seemed that adding 1 wt% and 3 wt% silica particles. The most beneficial effects were obtained by improving the mechanical properties of high impact acrylic dentures.

In 2017, Gulfem Ergun, [27] improved the transverse strength, modulus of elasticity, surface roughness, hardness, and water sorption/solubility of nanoparticle zirconium oxide (nano-ZrO_2) added to heat-cured poly (methyl methacrylate)(PMMA) after thermo cycling. The specimens were divided into four groups according to the ratio of nano-ZrO_2 addition to heat-cured PMMA; group 1: 5% nano-ZrO_2; group 2: 10% nano-ZrO_2; group 3: 20% nano-ZrO_2, and group 4 (control): PMMA without nano-ZrO_2. A statistically significant decrease in transverse strength was observed by all additional rates of nano-

ZrO_2 compared with group 4 (control; $P< 0.025$). When hardness results were evaluated, groups 1 and 2 showed statistically higher values compared with groups 3 and 4 ($P< 0.025$). The surface roughness of group 3 was statistically higher than the other groups ($P< 0.025$), but it was within the clinically acceptable limits. As the nano-ZrO_2 addition rate increased, water sorption/solubility values of the specimens also increased. Although nano-ZrO_2 addition had an adverse effect on some mechanical and physical properties of heat-cured PMMA, it increased hardness values, implying that the addition of nano-ZrO_2 would contribute positively to some mechanical properties of PMMA denture base material when nano-ZrO_2 was homogeneously distributed in PMMA.

Chapter Three
Experimental Work

Chapter Three

Experimental Work

3.1 Introduction

This chapter is devoted for describing the techniques and equipment required used for preparing pure and reinforced PMMA samples, and the device used in tensile strength test conducted on the samples to study the evolution of tensile strength resistance of PMMA as well as the types and specifications of the solvents used and the SiO_2 nanoparticle specifications. Figure (3-1) shows the flow chart of experimental work in current study.

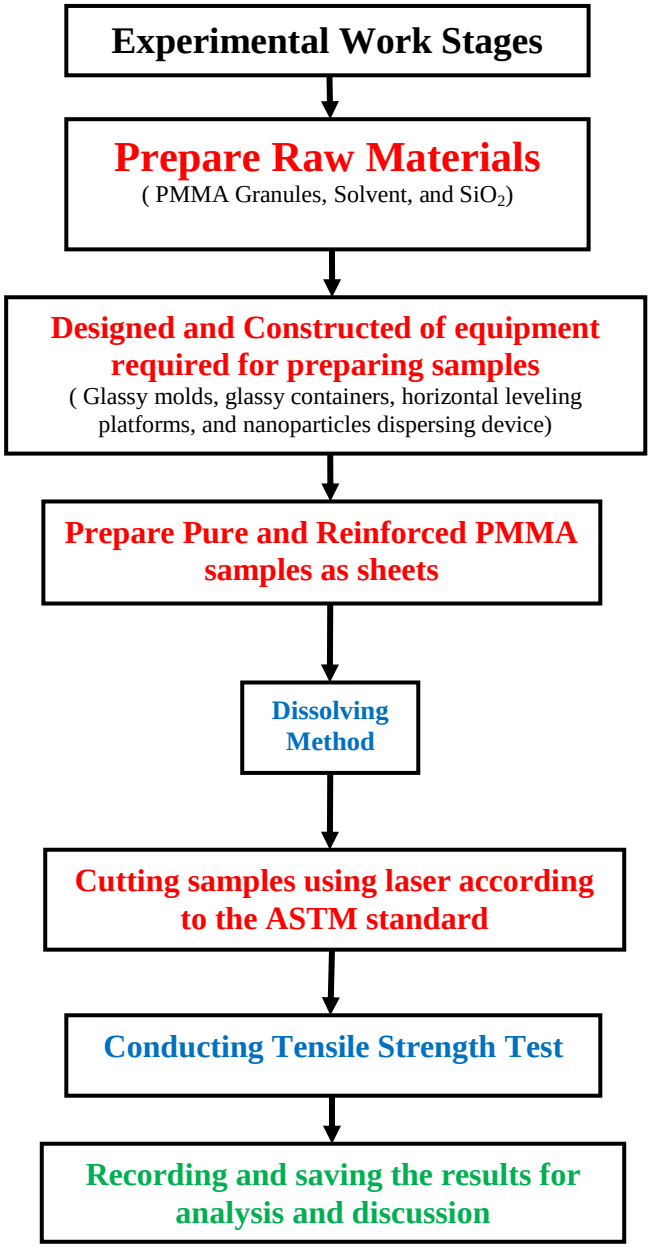

Figure (3-1) Block diagram of experimental work.

3.2 Specimens Preparation

In this section, method for preparing PMMA specimens, types of solvents used, and filler specifications used in the reinforcement will be explained. The polymerization method, whether in addition or condensation, is the general method used in the production and preparation of PMMA industrially. Due to the difficulty of the polymerization process, the lack of necessary equipment, and shortage of time, pure and reinforced PMMA specimens were prepared in dissolving method: by dissolving the PMMA granules in solvents and then casting them into glassy molds.

Dissolving PMMA in organic solvents is an important process for several reasons including the ability of solvent to dissolve the PMMA, the dissolution rate, temperature, the evaporation rate of the solvent, and the formation of bubbles after the evaporation of the solvent. Three solvents were used to dissolve the PMMA granules, which were acetone, tetrahydrofuran (THF) and isopropanol, their specifications, are listed in table (3-1). During experiments in current work, it was established that isopropanol was unable to dissolve PMMA granules, unlike the other two solvents. It was also observed that tetrahydrofuran was faster than acetone in dissolving the PMMA granules, as well as the amount of bubbles present in the sample after the evaporation of tetrahydrofuran was less compared to acetone, this is completely identical to the result obtained by Evchuk et al. [28], as a result for that, tetrahydrofuran was adopted .The perfect amount of solvent used for dissolving the mixture of PMMA granules and filler was four times the weight of the mixture (i.e. for each 100 g of mixture, 400 g of solvent was used). Silicon oxide nanoparticles (SiO_2) have been used as reinforcement filler. The most important reasons that led to the selection of this filler are abundance, cheapness, non-toxicity, high hardness, and high melting point, another important reason is that PMMA transparency is not significantly affected when adding small amounts of SiO_2.

Table (3-1) Specifications of THF, Acetone and Isopropanol [28].

Property	THF	Acetone	Isopropanol
Chemical Formula	C_4H_8O	C_3H_6O	C_3H_8O
Gas Number	109-99-9	67-64-1	67-63-0
Molecular Weight (g/mol)	72.11	58.08	60.1
Appearance	Colorless	Colorless	Colorless
Physical State	Liquid	Clear liquid	Liquid
Boiling Point (^{o} C)	66	56	82
Freezing Point (^{o} C)	-108	-96	-89
Autoignition Temp. (^{o} C)	230	465	235
Density (kg/l)	0.89	0.786	0.785
Vapor Pressure	19300	24700	4400
Purity %	99.7	99.5	99.5

3.3 Dissolving Method

PMMA material used in this method is a spherical granules with a diameter of 3 mm purchased from Xinxiang Chuangmei Technology Co., Ltd in China. The filler is a silicon oxide nanoparticles (SiO_2) treated with silane coupling agent and specifications are purity: 99%, average particle size: 10-30 nm, PH: 5.5-6, and square surface area: 400m^2/gm purchased from SkySpring Nanomaterials, Inc. Houston, TX. 77082. USA. The solvent used is (THF) tetrahydrofuran (C_4H_8O) with minimum assay (GC) 99.7% purchased from Central Drug House (P) Ltd - Company, New Delhi, India, as shown in figure (3-2). Casting molds are glassy molds with dimensions of 210 mm length, 140 mm wide and 20 mm high for first group and with dimensions of 140 mm length, 110 mm wide and 20 mm high for second group. These molds are placed on horizontal leveling platforms to ensure uniform thickness of the cast material. The molds are covered by glassy containers to protect them from unwanted external factors such as dust and wind as well as to ensure a saturated perimeter around the casting mold, as in figure (3-3).

The technique used in this method to prepare the specimens and disperse nanoparticles is based on dissolving PMMA granules using a solvent placed

inside a closed flask with adding filler and mixing the mixture using magnetic stirrer at room temperature for 2 hours and a speed of 1500 rpm. The amount of solvent used to dissolve 100 g of the PMMA and SiO_2 mixture is 400 g. Table (3-2) shows the ratios of mixing (SiO_2) nanoparticles with PMMA granules. The resulting solution is then poured into glassy molds placed on the leveling platform and covered with a glassy container and left to dry under the sun for 5 days to ensure complete evaporation of the solvent, as shown in figure (3-4). The specimens are then removed from the molds by placing them in a cold water tank for 30 minutes. The final stage of preparation is to place the samples in an electric thermal oven at 95 °C for 45 minutes and then compress the samples at a pressure of 0.5 MPa for two minutes using a hydraulic thermal press to ensure a smooth surface and free of bubbles, as shown in figures (3-5) and (3-6). The specimens required for test are cut using a laser cutting device.

Table (3-2) The mixing ratios between PMMA granules and SiO_2

Specimen code	**PMMA** granules (wt %)	**SiO_2** ratio (wt %)
S0	100	0
S1	99	1
S2	98	2
S3	97	3
S4	96	4

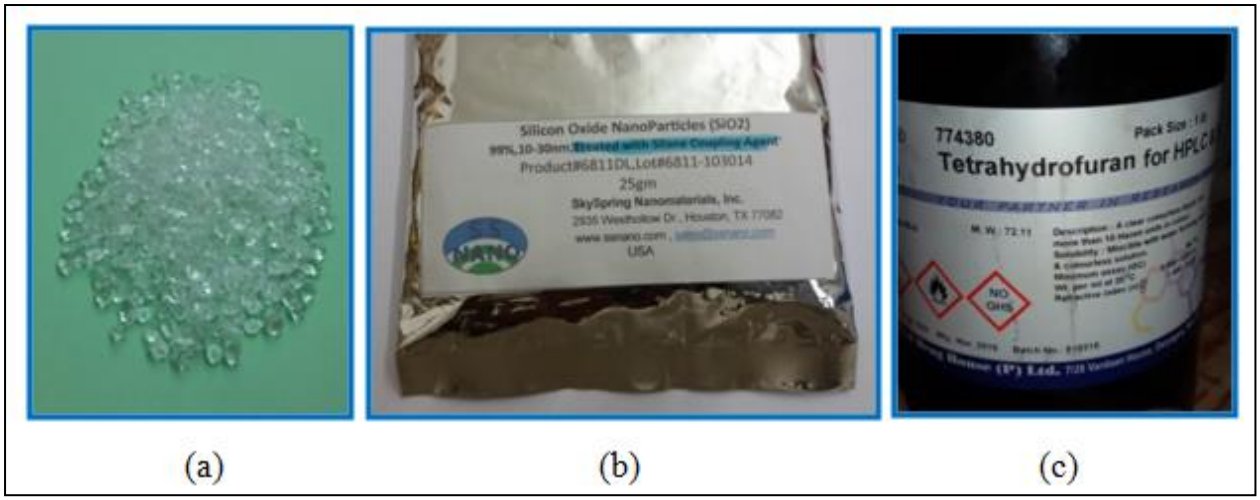

Figure (3-2) (a) PMMA granules, (b) Silicon oxide nanoparticles (SiO_2) and (c) Tetrahydrofuran solvent.

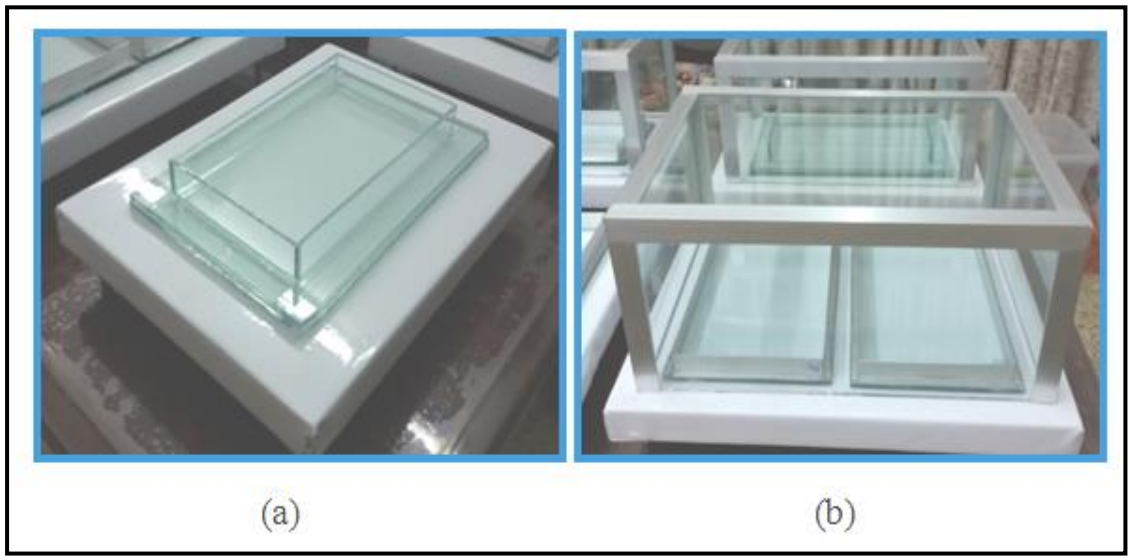

Figure (3-3) (a) Glassy molds and (b) Glassy containers and leveling platforms.

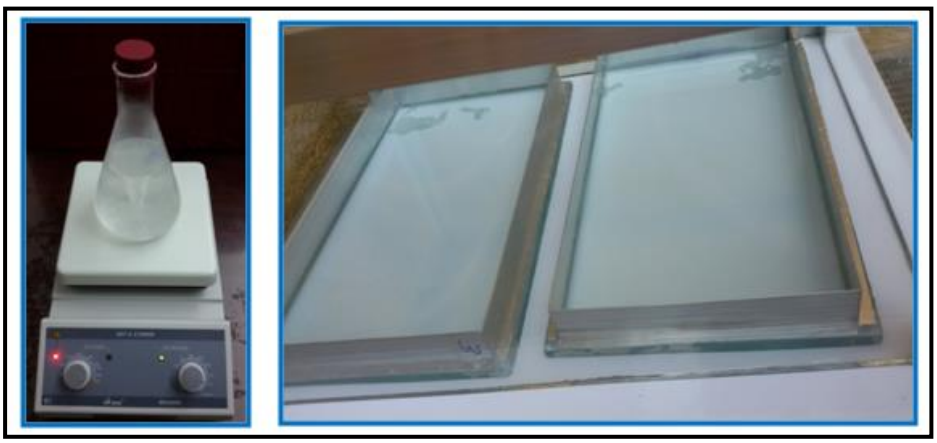

Figure (3-4) Preparation of PMMA/SiO_2 composites

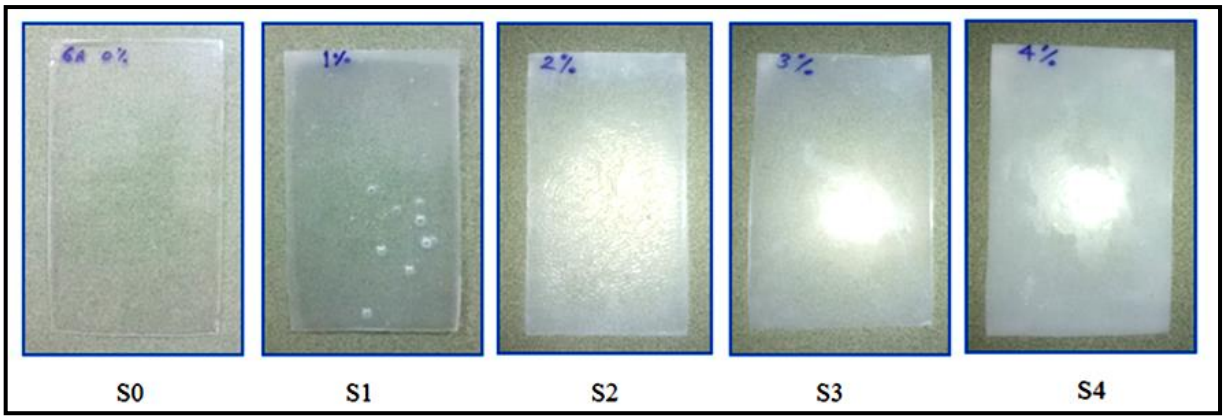

Figure (3-5) Specimens of PMMA/SiO_2 composites (Dissolving Method).

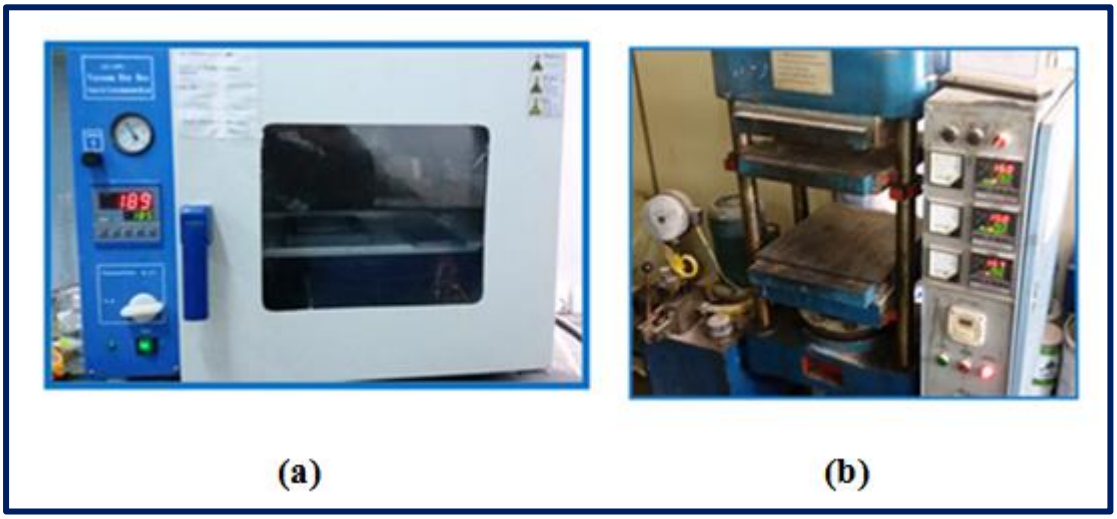

Figure (3-6) Equipment required for pressing specimens (a) Electric thermal oven and (b) Hydraulic thermal press.

3.4 Tensile Test

Samples used in the tensile test were cut according to (ASTM) D-638; the dimensions and shape of test sample are offered in figure (3.7). Tensile test was conducted at the temperature of room (23°C) with rate of displacement of 5 mm/min using a universal test device, as offered in figure (3.8). The tensile tests were conducted in laboratories of Materials Engineering College, Babylon University. The equations used to calculate stress and strain were listed in the appendix. The curve of stress and strain of the samples tested is plotted by the device itself on graph paper. The tensile test samples before and after conducting the tensile tests are shown in figure (3.9).

ASTM D638-10 Type IV	Dimensions (mm)
W – Width of narrow section	6
L – Length of narrow section	33
WO – Width overall, min	19
LO – Length overall, min	115
G – Gage length	25
D – Distance between grips	65
R – Radius of fillet	14
RO – Outer radius	25
T – Thickness	4

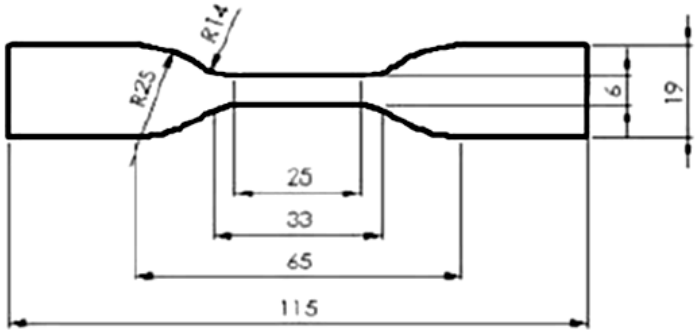

Figure (3.7) The dimensions and shape of the tensile test specimens.

Figure (3.8) Tensile test machine.

Figure (3.9) Specimens of tensile test before and after conducting the tensile tests.

3.5 Specimens Preparation Equipments

The equipment required for preparing the specimens of pure PMMA and SiO_2/PMMA composites in sheet-form are glassy molds, glassy containers, and horizontal leveling platforms will be described in this section.

3.5.1 Glassy Molds

Two sets of glassy molds were made using 6 mm thickness glass sheets, adhesion material, and L-shaped aluminum corners of 20 x 20 mm. The number of molds in the first group is 10, which are used to obtain samples with dimensions of 4 mm thickness, 100 mm width, and 130mm length. The number of molds in the second group is also 10, which are used to obtain samples with dimensions of 4 mm thickness, 130 mm width , and 200 mm length, as shown in figure (3-10). These molds were used in the preparation method which used for preparing pure and reinforced PMMA samples.

3.5.2 Glassy Containers

Ten glassy containers with dimensions of 250 mm length, 160 mm width and 100 mm height were made using 6 mm thickness glass sheets, adhesion material, and L- shaped aluminum corners of 20 x 20 mm, as shown in figure (3-11). These containers were used to cover sample molds prepared . The containers were designed with dimensions that allow placing either one large glassy mold or two small glassy molds within a single container. The glassy containers are used for two objectives, first to protect the samples from dust and impurities during the solvent evaporation process and the solidification of samples.. The second objective is to provide a space saturated with solvent steam that prevents the top layer of the sample surface from solidifying for as long as possible during the evaporation process, which reduces the formation of bubbles.

3.5.3 Horizontal leveling platforms

Ten horizontal leveling platforms were made with dimensions of 270 mm length and 180 mm width using wooden boards with a thickness of 18 mm. Each platform is provided with four legs adjustable up and down in order to level the platform horizontally to ensure that one thickness of PMMA samples is obtained. The surface of the platforms was covered with a layer of chemical-resistant leather, as shown in figure (3-16). The platforms are designed with dimensions that allow one glassy container to be placed on their surface.

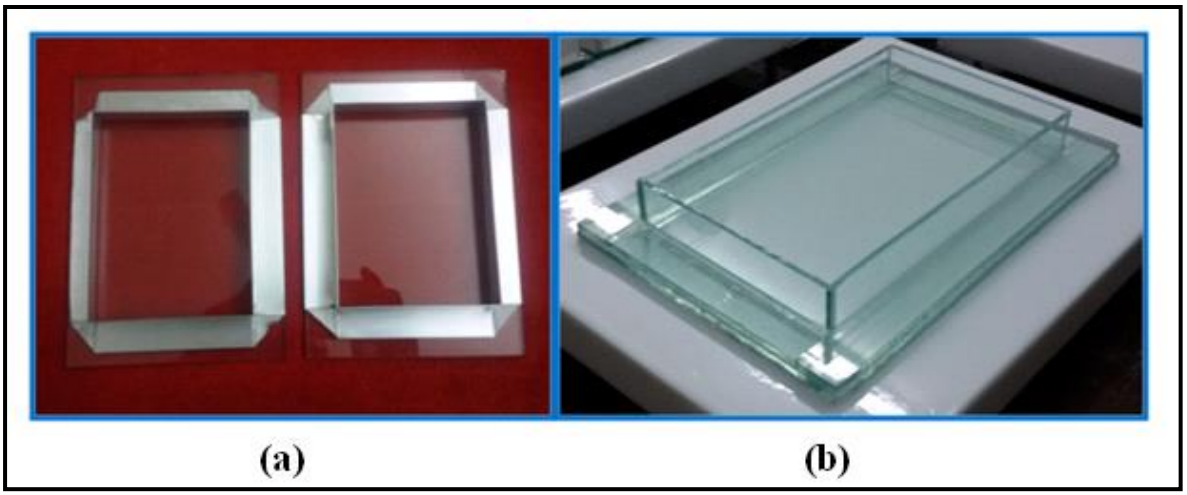

Figure (3-10) (a) Small glassy mold, (b) Large glassy mold.

Figure (3-11) Glassy container.

Figure (3-12) Horizontal leveling platform.

Chapter Four
Results
and
Discussions

Capter Four

Results and Discussion

4.1 Introduction

The experimental tests results for pure PMMA and PMMA/SiO2 composite specimens will be discussed in this chapter. The tested specimens were divided into five groups according to according to the added SiO_2 ratio as illustrated in table (4-1). The properties studied and discussed for all samples in this chapter were tensile strength resistance test.

Table (4-1) Specimens groups

Specimen Code	SiO_2 ratio (wt %)
S0	0
S1	99
S2	98
S3	97
S4	96

4.2 Results and discussion of tensile strength

The samples used in the tensile tests were cut according to (ASTM) D-638; dimensions and shape of the test sample were presented in figure (3.7) in chapter three. Tensile tests were obtained at the room temperature (23°C) and rate of displacement of 5 mm/min using a universal test apparatus, all tests were conducted in laboratories of Materials Engineering College, University of Babylon. The curve of stress and strain of the samples tested was plotted by the device itself on graph paper.

Figures (4.1 to 4.5) represent the tensile test behavior of specimens (S0, S1, S2, S3, and S4). The results showed an improvement of the tensile strength for PMMA/SiO_2 composites (S1, S2, and S3 specimens) while a decrease in tensile strength was observed for the S4 specimen compared to pure PMMA (S0 specimen).This behavior was caused by the effect of nanoparticles scattered

between free spaces within the chain on restricting the movement of molecular chains in the PMMA matrix. A significant increase in ultimate tensile strength can be observed for PMMA/SiO_2 composite by 1wt% compared with the other ratios of SiO_2. Any other increase in SiO_2 nanoparticles content above 1wt% imposes an adverse effect by reducing the ultimate tensile strength as shown in figure (4.6). The reason for this behavior was that the increase in SiO_2 leads to the nanoparticles agglomerate with each other, the same behavior has been observed in the work of Saeed Shirkavand and Elnaz Moslehifard [43]. Aggregated nanoparticles act as centers of stress concentration within PMMA and impose a negative effect by reducing the ultimate tensile strength.

Figure (4.7) represents the elongation of specimens (S0, S1, S2, S3, and S4) as a function of SiO_2 nanoparticles ratio, the results showed a decrease in elongation for PMMA/SiO_2 composites compared to pure PMMA this behavior is due to the distribution of stress on both the base material and the reinforcement filler, while an increase in elongation was observed for PMMA/SiO_2 composites by 2 wt%, 3wt%, and 4wt% ratios compared to 1wt% ratio, the reason is that at a certain stress, the interface between the base material and the reinforcement filler fails, causing its withdrawal and make it act as gaps and areas to focus the stresses.

It was clear from the results of the tensile test of the specimens S1, S2, S3, and S4 that the best ratio for adding SiO_2 nanoparticles was 1wt % compared to other ratios. All the results of the tests conducted on the PMMA specimens were listed in table (4-2) to provide data sheet for the PMMA material where it's mechanical and tribological properties were improved in this study.

Table (4-2) results of tensile strength test

Specimen code	SiO_2 Ratio (wt%)	Tensile Test	
		Ultimate tensile strength (MPa)	Elongation (mm)
S0	0	30.1	2.47
S1	1	68.59	1.41
S2	2	42.57	2.091
S3	3	42.17	1.88
S4	4	23.83	2.164

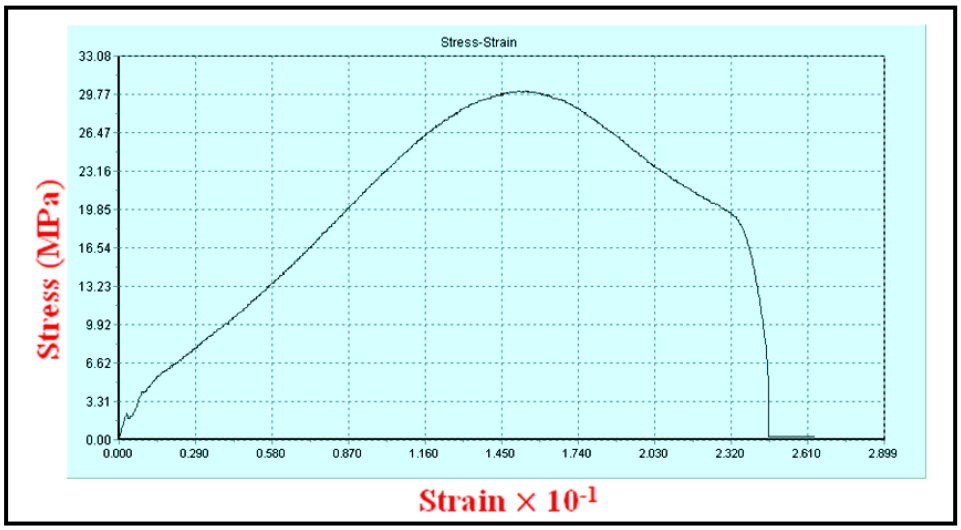

Figure (4.1) Stress – strain curve of S0 specimen (pure PMMA).

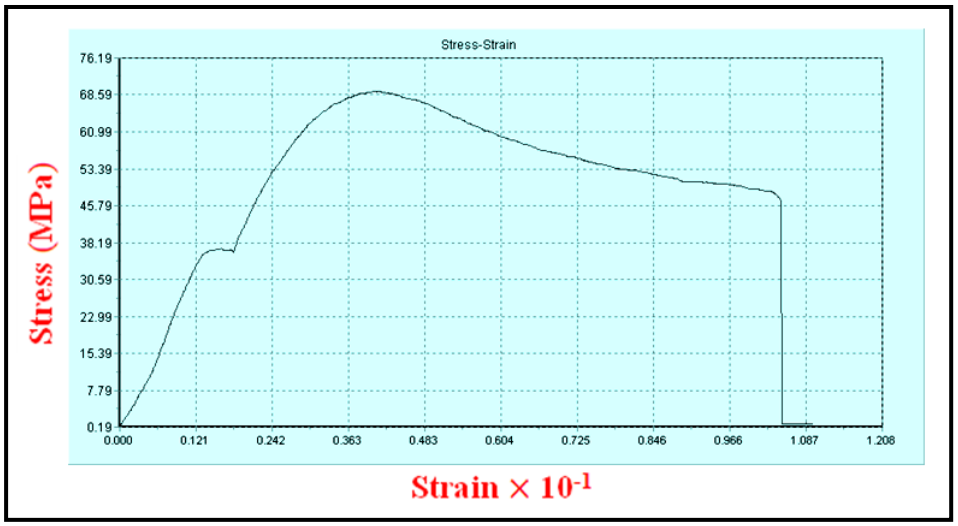

Figure (4.2) Stress - strain curve of S1 specimen (1wt% SiO_2).

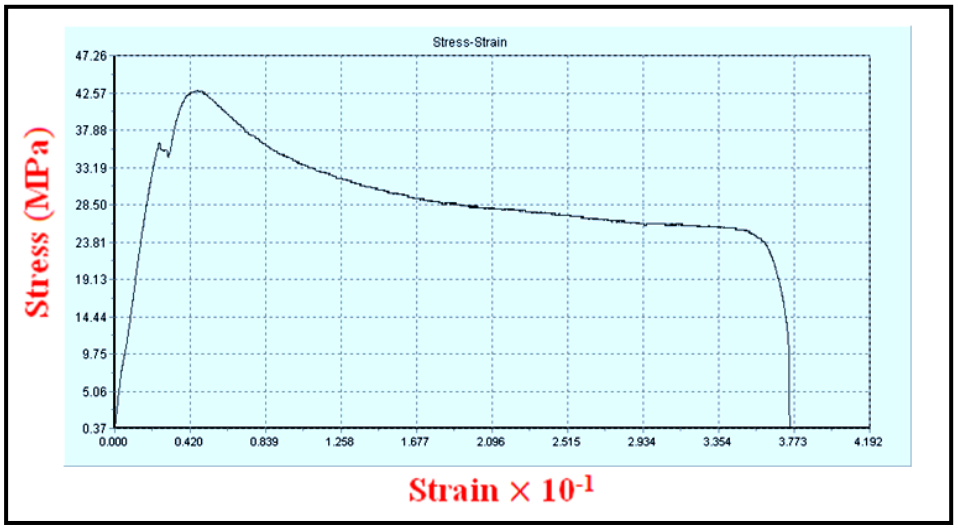

Figure (4.3) Stress - strain curve of S2 specimen (2wt% SiO_2).

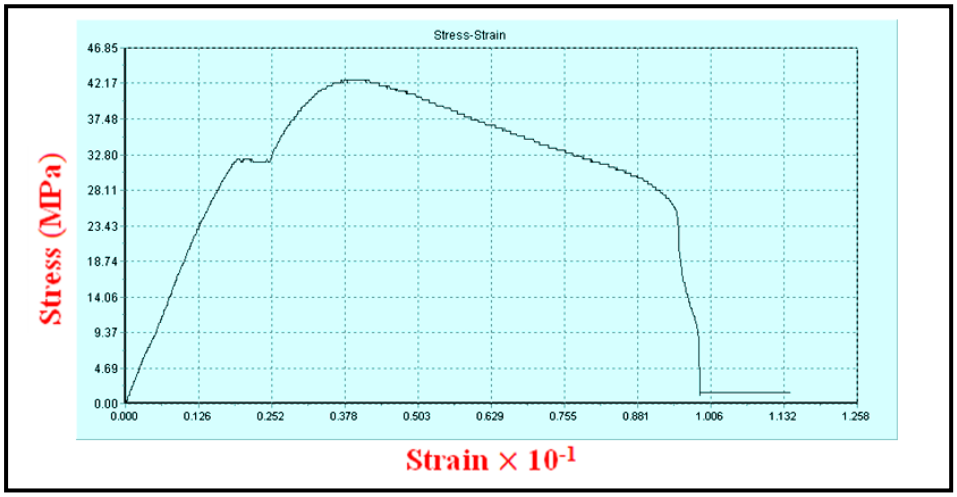

Figure (4.4) Stress - strain curve of S3 specimen (3wt% SiO_2).

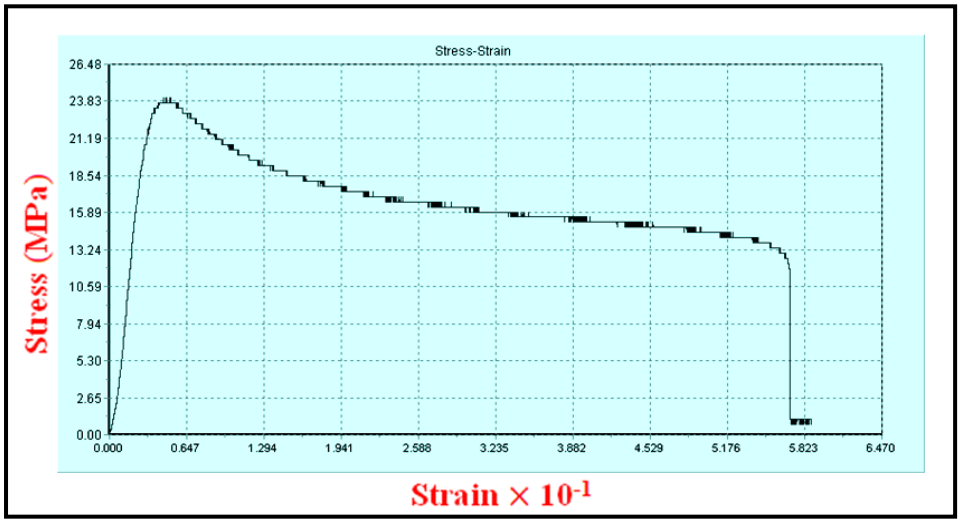

Figure (4.5) Stress - strain curve of S4 specimen (4wt% SiO_2).

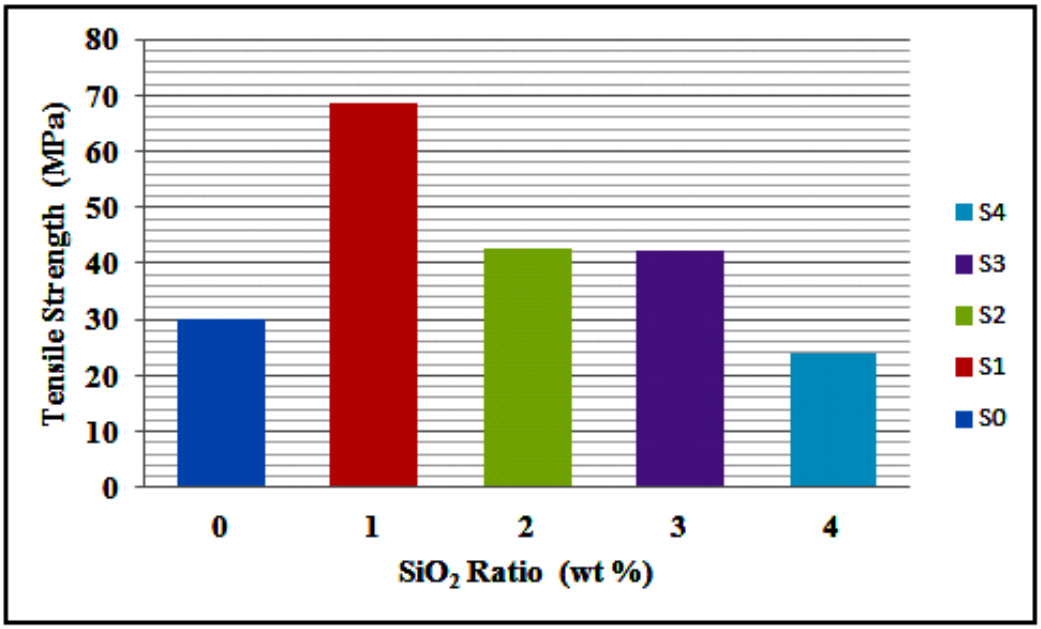

Figure (4.6) Ultimate tensile strength for specimens (S0, S1, S2, S3, and S4) set as a function of SiO_2 nanoparticles ratio.

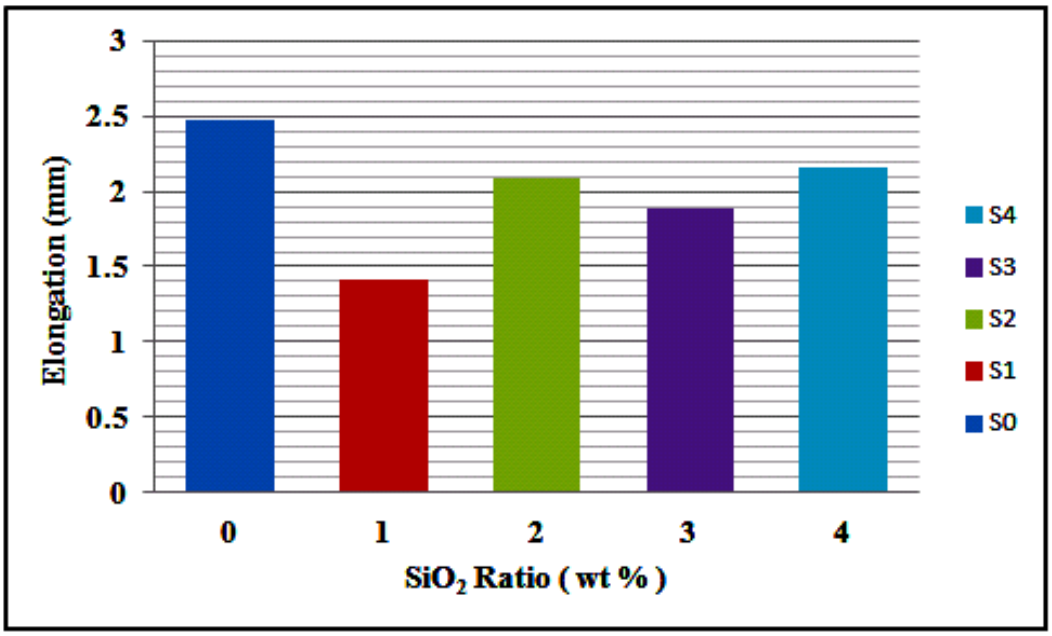

Figure (4.7) Elongation of specimens (S0, S1, S2, S3, and S4) as a function of SiO_2 nanoparticles ratio.

Chapter Five
Conclusions
and
Recommendations

Chapter Five
Conclusions and Recommendations

5.1 Conclusions

The conclusions obtained in the current study can be summarized according to the surface hardness test conducted on the PMMA by the following points:

1. Improvement of the tensile strength for PMMA/SiO_2 composites (S1, S2, and S3 specimens) while a decrease in tensile strength was observed for the S4 specimen compared to pure PMMA (S0 specimen).
2. A significant increase in ultimate tensile strength can be observed for PMMA/SiO_2 composite by 1wt% compared with the other ratios of SiO_2.
3. Any other increase in SiO_2 nanoparticles content above 1wt% imposes an adverse effect by reducing the ultimate tensile strength.
4. A decrease in elongation for PMMA/SiO_2 composites compared to pure PMMA.
5. An increase in elongation for PMMA/SiO_2 composites by 2 wt%, 3wt%, and 4wt% ratios compared to 1wt% ratio.

5.2 Recommendations

The recommendations proposed for future studies can be summarized in the following points:

1. Conducting a theoretical study and using simulation to obtain theoretical results for the purpose of comparing them with the experimental results obtained in the current study.
2. Using other types of nanoparticles separately or mix them with nanoparticles used in the current study.
3. Preparing the pure PMMA and PMMA/SiO_2 composites specimens using polymerization and compare the tests results with the preparing methods used in the present study.

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Appendix

1. The equations used to calculate stress and strain

$$\sigma = F/A$$

$$\varepsilon = L- L_0 / L_0$$

$$\varepsilon = \Delta L / L_0$$

2. The equation used to calculate the strain rate

$$\dot{\varepsilon} = V_{tip} / w$$

الملخص

تستخدم مادة PMMA في العديد من الصناعات والتطبيقات الهندسية مثل السيارات والطائرات وإشارات المرور والإعلانات الضوئية والعدسات والنظارات والخلايا الشمسية وصناعة الأسنان والعديد من الأجهزة الكهربائية وتطبيقات البناء مثل الديكورات وعروض السلع وحمامات السباحة الرياضية، و ملاعب الغولف، وكذلك في العديد من المجالات الزراعية. وتتميز مادة PMMA بالوفرة ورخيصة الثمن والكثافة المنخفضة والمتانة النسبية الجيدة وسهولة التشكيل والقطع حسب المقاسات والأشكال المطلوبة، وعدم السمية والعزل الكهربائي والمقاومة الكيميائية، إلا أن مقاومة الشد ضعيفة.

أصبح الحصول على أسطح PMMA عالية الجودة أولوية لكل من المصنعين والباحثين على حد سواء لسببين أحدهما وظيفي والآخر جمالي، لأن الخدوش التي تظهر على سطح PMMA نتيجة ضعف قوة الشد تقلل من استخدامها في المجال البصري. الصناعة والعديد من التطبيقات الهندسية، حيث أن وجود الخدوش يؤدي إلى زيادة الضغط أثناء الشد والتأثير وتحميل التعب الذي يقوض طول عمر PMMA أثناء الاستخدام. في الدراسة الحالية تم إضافة جزيئات أكسيد السيليكون النانوية (SiO_2) بنسب (1، 2، 3 و 4wt%) من أجل دراسة تأثير SiO_2 على مقاومة قوة الشد لـ PMMA.

تم تحضير عينات PMMA النقية والمعززة بطريقة الذوبان. كما تم تصميم وتصنيع جميع المعدات اللازمة لإعداد العينات لاختبار قوة الشد لـ PMMA.

أظهرت النتائج التي تم الحصول عليها من الاختبارات التي أجريت تحسنا في قوة الشد لمركبات PMMA/SiO2 (عينات S1 وS2 وS3) في حين لوحظ انخفاض في قوة الشد لعينة S4 مقارنة مع عينة PMMA النقية (عينة S0). يمكن ملاحظة زيادة كبيرة في قوة الشد النهائية لمركب $PMMA/SiO_2$ بنسبة 1% بالوزن مقارنة بالنسب الأخرى لـ SiO_2. أي زيادة أخرى في محتوى الجسيمات النانوية SiO_2 أعلى من 1% بالوزن تفرض تأثيرًا سلبيًا عن طريق تقليل قوة الشد النهائية.

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قسم الهندسة الميكانيكية

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تاريخ المناقشة : الاثنين 5 /5/2024