

**Republic of Iraq
Ministry of Higher Education
and Scientific Research
University of Baghdad
College of Dentistry**



The effect of silicon carbide nanoparticles addition on some physical and mechanical properties of heat cured acrylic resin denture base material

A Thesis

**Submitted to the council of the College of Dentistry at the
University of Baghdad in partial fulfillment of the
requirements for the degree of Master of Science in prosthetic
dentistry**

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2019 A.D.

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Dedication

*To whom that paradise under her feet, to whom that she
exhausted herself for me...*

“My Mother”

*To whom that I'm proud of carrying his name and honored to be
related with him...*

“My Father”

To the hearts that being with me for all my life...

“My Sisters”

Acknowledgment

All praise and thanks to **Allah** the exalted for his never-ending blessings and mercy upon me. This work cannot be accomplished without the help, encouragement, and guidance of family, colleagues and friends throughout my course of study.

I like to say thank you to **Prof. Dr. Hussain F. Al-Huwaizi** Dean of College of Dentistry, University of Baghdad for his support to postgraduate students.

I like to express my thanks to **Prof. Dr. Nidhal H. Ghaib**, Assistant Dean for Scientific Affairs and Students for her help and support.

I want to acknowledge **Prof. Dr. Raghdaa Kareem Jassim**, Head of Prosthodontic department for her kind help and support.

My all respect and appreciation to my supervisor **Assist. Prof. Dr. Hikmat Jameel Abdulbaqi** for his valuable help, great wise advice and continuous support all the way through finishing this thesis.

Special thanks to family especially my dear father for continuous support and providing me the appropriate environment to have the degree.

I am very grateful to **Prof. Dr. Abdul Jabbar Attiah** and members of laboratory of college of chemistry Al-Mustansiriyah University for their great help in my thesis.

I would like to thank my colleagues, **Dr. Mohammed Abdulaziz**, **Dr. Alhakam jaleel** and **Dr. Mustafa Saadi** for their friendship and kind help throughout my Master degree course.

Finally, I would like express my gratitude to everyone participated to accomplish this piece of work, all efforts are so valuable.

Abstract

Statement of the problem

Polymethyl methacrylate (PMMA) is the most commonly used material in denture fabrication. The material is far from ideal in fulfilling the physical requirements such as poor thermal conductivity, insufficient surface hardness, increased surface roughness and poor mechanical properties.

Aim of the study

The purpose of this study was to investigate the effect silicon carbide nanoparticles addition in percentages of 0.125% weight and 0.25% weight on following properties of heat cured acrylic resin denture base material : thermal conductivity, impact strength, transverse strength, surface hardness and surface roughness.

Materials and methods

One hundred fifty (150) specimens were prepared from heat cured acrylic resin and these specimens were divided into five groups according to the tests, each group consisted of 30 specimens and these were subdivided into three groups: (control group), reinforced polymethyl- methacrylate (0.125% weight silicon carbide nanoparticles) group and reinforced polymethylmethacrylate (0.25% weight silicon carbide nanoparticles) group. The thermal conductivity, impact strength, transverse strength, surface hardness and surface roughness were investigated.

Results

The results of this study showed that 0.125% weight silicon carbide nanoparticles, was a highly significant increase in both thermal conductivity and surface hardness while there was non-significant decrease in transverse strength ,impact strength and surface roughness when added to acrylic denture base material.

At the concentration of 0.25% weight silicon carbide nanoparticles the results showed a highly significant increase in both thermal conductivity and surface hardness ,and a non-significant decrease in transverse strength , impact strength and surface roughness when added to acrylic denture base material.

Conclusion

The addition of silicon carbide nanoparticles into heat cured acrylic resin denture base material improves thermal conductivity, surface hardness and surface roughness, with a non-significant decrease (not improved) in both transverse strength and impact strength.

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List of abbreviations

μm	Micrometer
3PB	Three Point Bending
ADA	American Dental Association
ANOVA	Analysis of variance
COTE	Coefficient of thermal expansion
EDS	Energy Dispersive X-ray Spectroscopy
FTIR	Fourier Transform Infrared Spectrophotometer
HSD	Honestly significant difference
ISO	International Organization for Standardization
IU	Indentation unit
Kcal	Kilocalorie
MMA	Methyl methacrylate
Mpa	Migapascal
MPS	Tri –methacryloxypropyl trimethoxysilane
PMMA	Polymethyl methacrylate
Psi	Pound per square inch
SD	Standard Deviation
SE	Standard Error
SEM	Scanning Electron Microscope
SiC	Silicon Carbide
SPSS	Statistical package for social science
W/m.K	Watts per meter –Kelvin
Wt. %	Weight Percentage

Introduction

Introduction

Since decades, loss of teeth and their replacement by dentures is a common procedure. The denture base had been made from different materials like plastics, metals and different materials (like cellulose and epoxy resin). Acrylic resin Polymethylmethacrylate (PMMA) is frequently used not only as prosthetic-base material, but also in orthodontic removable appliances such as retainers and functional appliances like bite raising appliance (**Price, 1994**). PMMA is generally used because of its optical properties, being biocompatible and good appearance. In spite of that, the mechanical properties against impact, bending, and fatigue are low and it is important to be addressed to improve PMMA as a denture base material (**Bettencourt *et al.*, 2010**).

Only a small number of studies have been made on improvement the thermal conductivity of acrylic resin, which is one of its disadvantages, and new studies are required. In elder patients, high thermal conductivity of denture base material influence patient satisfaction, tissue preservation, better taste sensation and reduce the feeling of denture as a foreign body (**Kul *et al.*, 2016**).

Various techniques have been made to improve the mechanical properties such as chemical correction of polymeric structure by addition of certain materials like rubber (**Bettencourt *et al.*, 2010**). To improve acrylic resin, there is another way, by addition of fibers and particles. Manufacturers traditionally improve polymers with micrometer-size and nanometer size fillers to improve strength and stiffness, to increase resistance to solvent or to reduce the price (**Nejatian *et al.*, 2006**). Due to these drawbacks and to increase the thermal conductivity of acrylic resin, particles of conductive materials can be added to the liquid or powder of acrylic resin before polymerization (**Kul *et al.*, 2016**).

Silicon carbide with many excellent properties such as good thermal and electrical conductivity, chemically stable, high mechanical strength and low

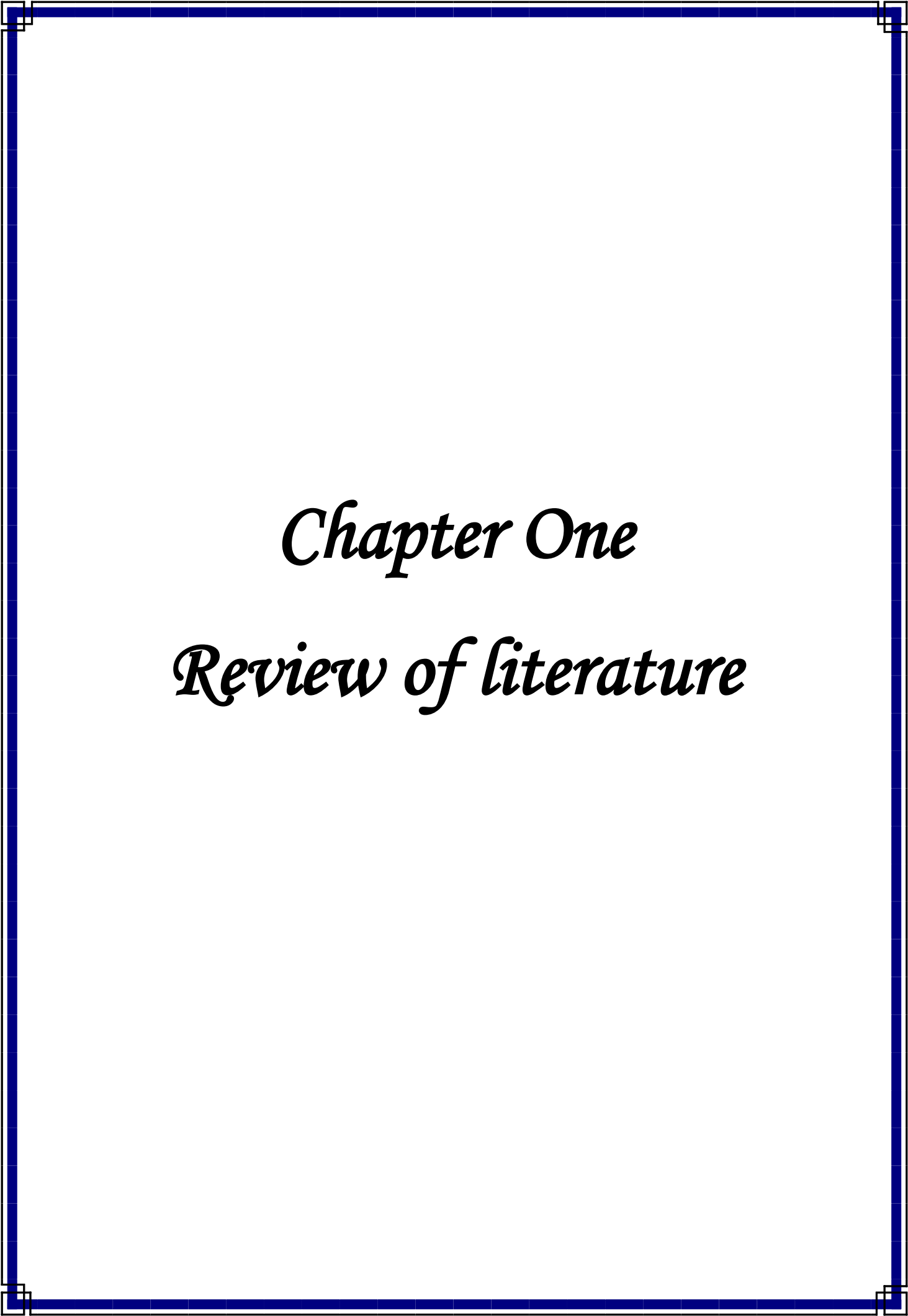
friction, can serve as both structured materials and functional materials (**Chen *et al.*, 2003**). Addition of functional nanoparticles into a polymer–matrix has much attention because of their cost/effective processability, special physical properties such as increased electric/heat conductivities, improved stiffness and strength and high-flexibility (**Guo *et al.*, 2008**).

Inorganic particles are not easily distributed in organic matrices, unless a dispersing agent is used. For this reason silane coupling agents are often used as they can form stable chemical bonds with both inorganic and organic materials. So the saline function as adhesion promoter, that is to improve the binding of nanoparticles to the matrix of polymer and this will result in improvement of the properties of that polymer (**Posthumus *et al.*, 2004**).

Aim of the study

The study was aimed to evaluate the effect of silicon carbide nanoparticles addition to heat cure acrylic denture base material on the following properties:

1. Thermal conductivity.
2. Impact strength.
3. Transverse strength.
4. Surface hardness.
5. Surface roughness.



Chapter One

Review of literature

Review of literature

1.1 Denture base materials

Denture base is the part of denture which rests on the foundation area and to which the teeth are attached (GPT 9, 2017). History of dentures to treat the edentulism back to 700 B.C .Various materials such as wood, ivory, bone, ,rubber and many other materials were used to make dentures. In 1937, *Walter Wright* introduced poly methyl methacrylate (PMMA) material as a denture base material and become the superior over all denture base materials and becomes popular in 1940s due to its good esthetic, easy clinical and laboratory manipulation and being economical (Alla *et al.*,2015).

1.1.1 Requirements of denture base materials

The ideal properties of denture base materials have been formulated. However, none of available materials fulfill all the requirements. For that reason, any material has good proportion of all requirements would be the material of choice, the requirements were listed by (Manappallil, 2007; McCabe and Walls, 2008):

- 1- Good strength and resistance to wear and abrasion.
- 2- Adequate thermal properties.
- 3- Dimensionally stable, no expansion, contraction or warpage during processing.
- 4- Pleasant taste and odor.
- 5- Biocompatibility.
- 6- Color stability.
- 7- Natural appearance.
- 8- Adequate adhesion to plastic, metal and porcelain.
- 9- Easy fabrication and repair.

10- Radiopacity.

11- Does not provide culture for growth of bacteria and fungi and can be disinfected.

12- Reasonable cost and shelf life.

1.1.2. Classification of denture base materials

The denture base materials can be classified into (Manapallil, 2007):

- Metallic denture base material: (Cobalt-Chromium alloy, Titanium, Aluminum, Gold, Nickel chromium).
- Non metallic denture base material: (porcelain, vulcanite, acrylic resin, , poly carbonate polymers, epoxy resin, cellulose and phenol-formaldehyde).

1.2. Polymers

Great development in the science of biomaterials in the last few decades that contribute to increase the life expectancy of human beings. Dental materials are classified into four main groups: metals, ceramics, polymers and composites (Alla *et al.*, 2015).

Polymers are commonly used in dentistry and especially used in prosthodontics that is in removable prosthesis such as partial and complete dentures. However, the polymers are also used in denture teeth, impression trays, temporary crowns and maxillofacial prosthesis. Polymers used in prosthodontics have special chemical and physical features to ensure suitable clinical applications (Powers and Wataha, 2013).

1.2.1. Composition

The term polymer means that the molecule composed of many (poly) parts (mers). The *mer* ending denote the simplest repeating chemical structural unit from which polymer is made. The polymers that are made from one type of

monomer are called *homopolymers*, while those which made from two or more different monomer are called *copolymers*. Polymers in which mer units arranged randomly along the polymer chain they called *random* which is found in normal polymers, while *block* polymers are special type of polymers in which large number of one mer type attached to large number of another mer type as seen in figure (1.1) (Powers and Sakaguchi, 2012).

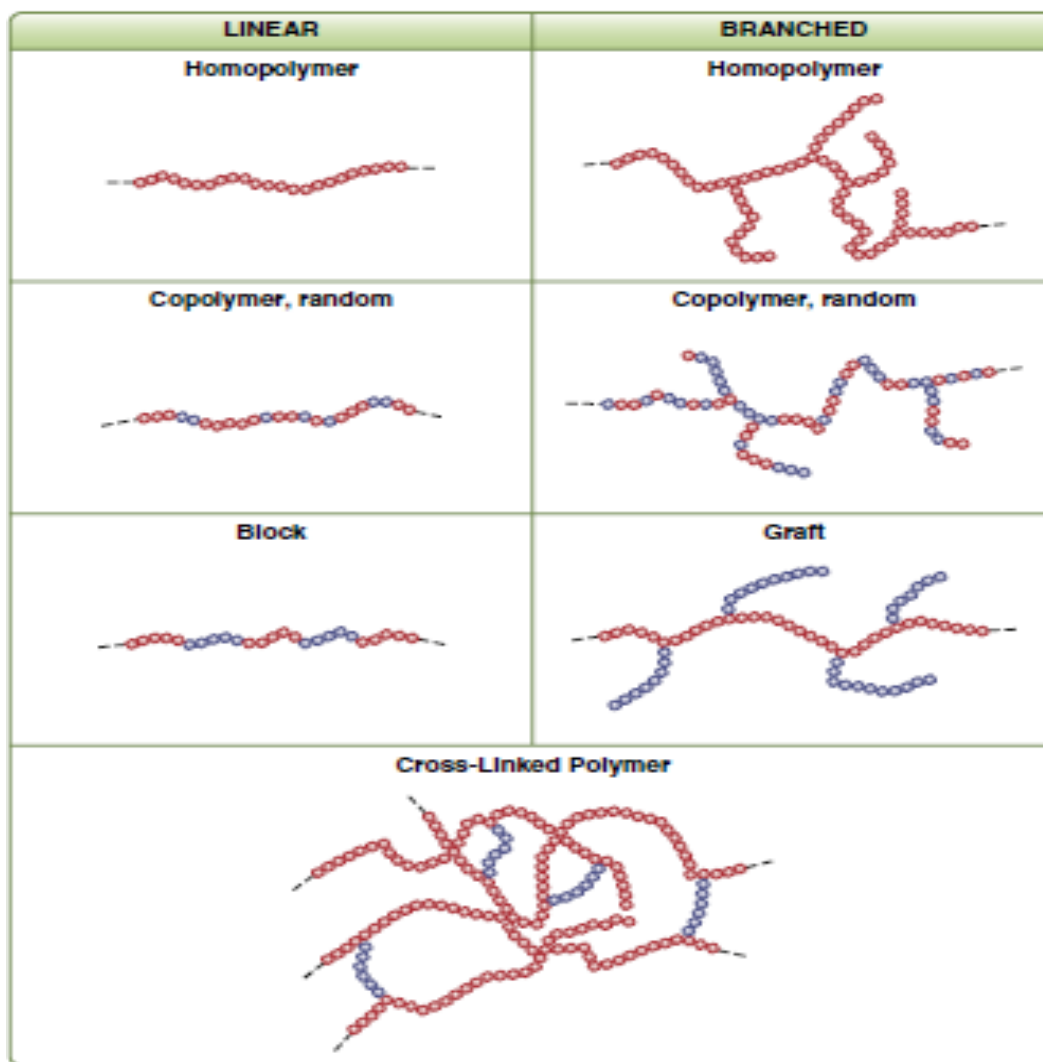


Figure (1.1): Linear, branched and cross-linked homopolymers and copolymers. Red circles: one type of mer unit; blue circle: another type of mer unit; *dashed lines*: continuation of polymer segment.(Powers and Sakaguchi,2012)

1.2.2. Polymerization Reaction

Polymerization is formation of chemical bonds that linking low molecular weight compound (monomers) into long chains of high molecular weight compounds (polymer) that have useful clinical properties which completely different from monomers as shown in figure (1.2) , there are two basic types of polymerization addition and condensation (**Powers and Wataha, 2013**).

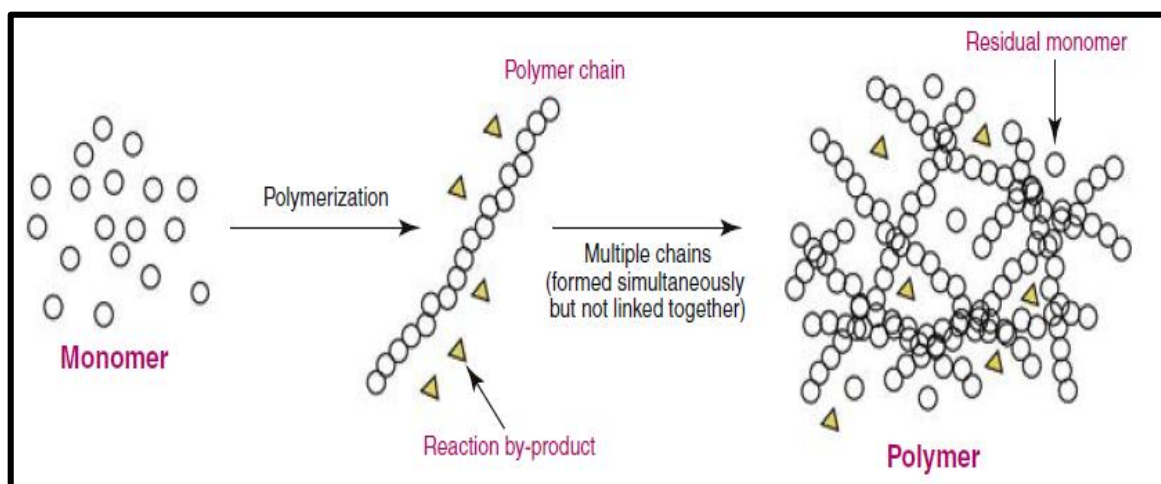


Figure (1.2): Polymerization reaction (Powers and Wataha, 2013).

A) Addition polymerization

Addition polymerization occurs when the reaction between two molecules (either similar molecules or different molecules) to form large molecules without omission of smaller molecules (like water or alcohol) (**Noort, 2013**).

The reaction mechanism is that the monomer is added successively to the end of growing chain. Addition polymerization begins from the active center, adding on polymer at a time quickly to form a chain, the chain is growing indefinitely until all monomer is completed. The process is simple, but is difficult to control. The addition polymerization can produce huge molecule and there is no change in composition during addition reaction because the monomer structure is repeated in polymer in many times (**Anusevice and *et al.*, 2013**). For example conversion of MMA to PMMA as shown in figure (1.3)

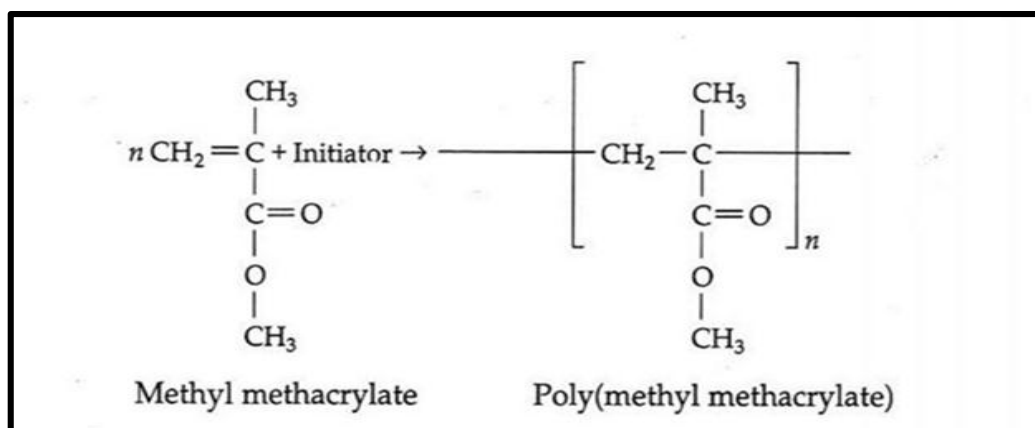


Figure (1.3): Conversion of MMA to PMMA in addition polymerization (Powers and sakaguchi, 2012).

The process of addition polymerization include four stages: (Anusevice *et al.*, 2013; Noort , 2013).

- I. Activation:** The presence of free radicals is necessary for polymerization reaction to occur. They are very reactive chemical compounds that have unpaired electrons. The production of free radical is called activation that occurs, for example, in decomposition of peroxide which provides two free radicals that in turn give more free radicals. Such chemicals, known as initiator that starts polymerization reaction.
- II. Initiation:** In which free radicals react with monomer and initiate polymerization reaction.
- III. Propagation:** Free radical transferred to monomer which can react with another monomer molecule. Repeating the process can generate polymer chain until chains interfere with each other or the free radicals are exhausted.
- IV. Termination:** Termination can occur by either binding of two ends of polymer chain or by exchange of one hydrogen atom from one growing polymer chain to another.

B) Condensation polymerization

Condensation reaction occurs when two molecules or more react to form third, larger molecule with formation of small molecule byproduct like water or alcohol. This mechanism is used in the formation of polysulfide, as well as biological tissues to make proteins, carbohydrates and ribonucleic acid (McCabe and Walls, 2008).

1.3 Acrylic resin

Acrylic resins are groups of thermosetting plastic or thermoplastic depend on cross linking materials that are made from acrylic acid or methacrylic acid. The chemical name is polymethyl methacrylate while in general it is called acrylic resin or methacrylic resin, and the chemical formula $(C_5H_8O_2)_n$. More than 90% of complete dentures use heat cure acrylic resin. These are supplied as poly (methyl methacrylate) powder and methyl methacrylate (liquid). The monomer reacts with polymer to dough like consistency that can be easily handled and it has a wide popularity (Choudhary, 2009).

1.3.1 Physical form

Denture base plastics are commonly supplied as powder-liquid form or gel form (Powers and Sakaguchi, 2012).

A. Gel form

This is the early form of acrylic resin, which is made from monomer and polymer that are premixed and packed as soft cake for immediate use. In spite of the advantage of good accuracy of gel form, their use had been decreased due to their short shelf life (Craig and Powers, 2002).

B. Powder – liquid form

Polymethyl methacrylate denture base material is commonly provided in powder – liquid form. When powder and liquid are mixed together a workable mass is formed (Anusavice *et al.*, 2013).

1.3.2 Chemical composition

1. Powder (polymer)

The powder contains predominantly prepolymerized polymethyl methacrylate in the form of micro-sized beads (or spheres). Most of polymers are modified by small amounts of ethyl, butyl or other alkyl side group methacrylates to produce polymer that is for some extent resistant to impact fracture, the initiator found in the powder like benzoyl peroxide that initiate the polymerization reaction (Powers and Sakaguchi, 2012).

Plasticizers are also incorporated into the beads of powder that help in dough formation; for many years dibutyl phthalate used as external plasticizer, while internal plasticizers can be used instead which contain methacrylate or acrylic monomer. The plasticizers are added to the polymer that they do not participate in polymerization reaction and do not become part in polymer strands they just trapped in polymer network. These chemicals are used to modify the physical properties (making the resin more rubbery). Because the plasticizers are not chemically bonded to the polymer, they easily leached out with time (O'Brien, 2002).

Pure polymer, such as poly (methyl methacrylate) are clear and glass like polymers and occasionally used in this form for construction of denture base. However, pigmentation are added to produce more life like appearance. Sometimes, small fibers are added to produce veined appearance. Pink appearance of acrylic are resulting by cadmium salts, these pigments have good color stability but there is leach of cadmium in only small amounts. Fear of toxicity from cadmium lead to gradual replacement with more safe substances

(McCabe and Walls, 2008). The principle contents of acrylic powder are shown in figure (1.4)

Powder	
Ingredients	Functions
Prepolymerized poly (methyl methacrylate)	<ul style="list-style-type: none"> Undergoes further polymerization. Reduces the polymerization shrinkage.
Copolymers of PMMA (5%) e.g. ethyl or butyl methacrylates	<ul style="list-style-type: none"> Increase the solubility of polymer in the monomer. To produce a polymer somewhat more resistant to fracture by impact.
Initiator (0.2–1.5%) e.g. benzoyl peroxide or di-iso butyl azonitrile	Initiates the polymerization of the monomer liquid after being added to powder.
Plasticizer, e.g. Dibutyl phthalate	<ul style="list-style-type: none"> Increases the solubility of polymer in the monomer. Produces softer and more resilient polymer.
Color pigments, e.g. Mercuric sulfide—Red Cadmium sulfide—Yellow Ferric oxide—Brown Carbon black—Brown	Added to obtain the various tissue like shades.
Opacifiers, e.g. zinc or titanium oxides	Provides opacity.
Dyed synthetic fibers made from nylon or acrylic	To simulate the minute blood vessels in the underlying oral mucosa.
Inorganic particles, e.g. glass fibers, zirconium silicate, whiskers of alumina,	<ul style="list-style-type: none"> To improve the stiffness. To decrease COTE.
Heavy metal compounds, e.g. barium, bismuth, etc.	<ul style="list-style-type: none"> Imparts radiopacity.

Figure (1.4): The principle content of acrylic powder (Alla, 2013).

2. Liquid (Monomer)

The liquid contain non-polymerized methyl methacrylate (MMA), and has a chemical formula as shown in figure (1.5) (Anusavice, 2007).

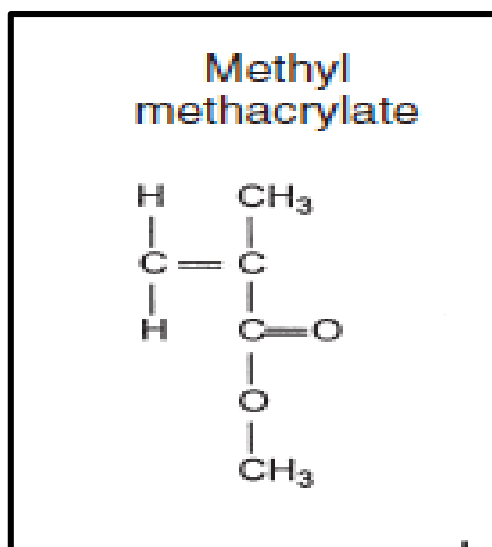


Figure (1.5) Chemical formula of the MMA (Anusavice, 2007).

The monomer gives the building blocks for polymerization and it is flammable highly volatile liquid that is clear and colorless at ambient temperature. It has low viscosity, boils at (100.3°-100.8° C), its density about 0.945 g/ cm³ and heat of polymerization (12.9 Kcal per mol) and distinct odor (Craig *et al.*, 2004; Powers and Sakaguchi, 2006; Choudhary, 2009). It should be stored in a dark container because it will be polymerized when activated by heat, visible or ultraviolet light (Anusavice, 2007).

Inhibitors are added to provide adequate shelf life of the liquid; the inhibitors that are frequently used to prevent premature polymerization is hydroquinone which may be present in concentration of 0.003% -0.1% (Craig and powers, 2002).

Plasticizers, like dibutylphthalate, are added to monomer to have soft and resilient polymer. Plasticizers do not enter the polymerization reaction and not interfere with interaction between polymer molecules. The plasticizers are added also to soft liners. One disadvantage of plasticizer use they leach out from the plastic gradually into oral fluids and result in hardening of denture base (Powers *et al.*, 2013). The dentures made from cold cure acrylic resin contains methyl methacrylate in the form of residual monomer which is capable of

causing irritation, allergic response and inflammation of oral mucosa. Besides that, the residual monomer also produce denture stomatitis and angular cheilitis. Residual monomer present in heat cure acrylic is less than that in self cure acrylic resin (Alla, 2013). The principle component of acrylic monomer are shown in figure (1.6)

Liquid	
Methyl methacrylate	Undergoes polymerization to produce PMMA.
Comonomers	Improves the properties of denture bases.
Inhibitor, e.g. Hydroquinone (0.003%–0.1%)	Prevents premature polymerization and prolongs the shelf-life of the liquid.
Plasticizers, e.g. butyl or octyl methacrylate and dibutyl phthalate	Producer softer and more resilient polymer.
Cross-linking agent, e.g. Ethylene glycol dimethacrylate	<ul style="list-style-type: none"> Improves the physical properties of the denture. Increases resistance to crazing.

Figure (1.6): The principle component of acrylic monomer (Alla, 2013)

The polymer monomer ratio is usually 3-3.5 : 1 by volume or 2.5:1 by weight this ratio of mixing resulting in about 7% polymerization shrinkage (volumetric and linear), if polymer monomer ratio is too high (more polymer/ less monomer) this will result in that not all polymer will wet by monomer and the cured acrylic resin will be granular, also dough will be difficult to manage.

If polymer monomer ratio is too low (less polymer/ more monomer) this will make polymerization shrinkage more than 7% and more porous denture, also dough forming time will prolonged (Powers *et al.*, 2013)

1.3.3 Mode of activation

The process of curing is modified by many researchers, not only to modify the physical and chemical properties but also to improve the working properties (Sada and Hannada, 1994).

1.3.3.1 Heat activation

Heat is required for decomposition of benzoyl peroxide initiator, this decomposition will lead to production of free radicals which in turn will initiate polymerization (**Ismail, 2007**).

A-Water bath curing:

Water bath curing is the most common method for curing of acrylic denture base. The polymerization occurs by application of heat, that are maintained until the polymerization is completed. By heat application the benzoyl peroxide (initiator) will decompose ,the decomposition occurs at relatively low temperature (about 70° C) and lead to production of free radicals. The free radicals will activate the monomer by attaching to them causing the double bond between two carbon atoms to open and in this way will initiate the polymerization (**Craig, 1997**).

The polymerization occurs by heat application of external heat to the flask that contain denture base which is placed in water bath at specific time and specific temperature (curing cycle) and the strength of denture base will depend on the time and temperature of the curing cycle. The polymerization reaction is exothermic reaction and when heating large mass acrylic, the temperature in the center of the mass will rise above that of the investing plaster and the flask. The boiling point of pure monomer is 100.38°C, quick heating of large mass of dough will cause the internal temperature to elevate above this temperature, the monomer boils and produce spherical voids in the hottest part. This will result in gaseous porosity of cured denture base (**McCabe and walls, 2008**).

Acrylic denture base can be polymerized by either two methods (**Craig and Powers, 2002**):

1-*Short curing cycle*: curing the acrylic at 74°C for 1.5-2 hours and then 100°C for 0.5- 1 hour.

2-Long curing cycle: curing the acrylic at constant temperature 74° C for 8 hours or longer (over night).

The choice of the curing cycle will influence the accuracy and impact resistance of acrylic more than mixing ratio. The curing with long curing period gives significantly better physical and mechanical properties (**Jerolimove, 1989**).

The short curing cycle provides faster but incomplete polymerization due to rapidity of heating which result in higher residual monomer and porosity in denture base and this accounts for significantly higher properties for long curing cycle which is greatly associated with microwave and autoclave curing methods (**Banerjee *et al.*, 2010**).

B-Microwave curing

Nishii in **1968** introduced microwave activation method. There are several problems faced with this method like reflection of microwave by metallic flasks. In **1984**, **Kimura *et al.*** solved the problem by production of fiber reinforced plastic flasks, but they were high in cost and easily braked which limit their use. With setting power at 450 Watt, 4.5 minutes are necessary for complete polymerization of 2 flasks, 8.5 minutes for 4 flasks, and 13 minutes for 6 flasks (**Botega *et al.*, 2004**).

The mechanical properties of denture base resin processed by microwave energy were compared to those processed by water bath, it was found that no significant difference between the values of strength of both methods (**Truong and Thomasez, 1988**).

Conventional acrylic resin cured by microwave showed increase in porosity when compared with conventional acrylic cured by water bath, but this significant increase in the porosity is clinically acceptable so it can be used for curing dentures (**Singh S. *et al.*, 2013**).

C-Autoclave curing

Autoclave can be defined as a pressure cooker that has a lid tight to air which traps the steam from boiling water and it was first used by *Muley* 1976. The steam will increase the pressure inside the autoclave which increase the boiling point of water. In spite of the advantages of this technique like ease, simplicity and cost-effectiveness, the major disadvantage is long processing time required (**Banerjee *et al.*, 2010**).

In 2012, **Abdulwahhab and Alnakkash** conducted a study and concluded that there was no significant difference in transverse, hardness and impact strength between acrylic cured by autoclave and that cured by conventional water bath method, but there was significant decrease in porosity for autoclave cured acrylic resin, that is because the pressure will accelerate the polymerization and higher pressure prevent boiling of monomer.

1.3.3.2 Chemical curing

Chemically activated resin is also called chemical curing resin, auto-polymerizing resin or self-curing resin (**McCaby and Walls, 2008**).

They are similar to heat-cure denture base and the only difference is in the manner of polymerization initiation. The composition of monomer is varied by addition of chemical activator in the form of tertiary amine, like dimethyl paratoludin to activate benzoyl peroxide and initiate polymerization. This type of resin provide more residual monomer than heat cure which is irritant to the soft tissue and act as plasticizer that decrease the transverse strength (**Anusavice, 1996**).

The amount of residual monomer released by self cure acrylic resin is higher than that of heat cure acrylic, with the fact that the residual monomer is about 3% to 5% for self cure acrylic while the heat cure produce about 0.2% to 0.5% (**De Oliveira *et al.*, 2014**).

1.3.3.3 Light cure

Since 1984, visible light cure (VLC) denture base resin was available and their use increased for relining and repair of the dentures. They are available in premixed ropes or sheets, powder-liquid system and gel. It require curing unit that emit shorter wavelength blue light in high intensity (**Murata *et al.*, 2007**).

Visible light cure resin show higher hardness than heat cure and self cure acrylic resin and there was high significant difference between impact values of heat cure and visible light cure due to brittle nature of (VLC) resin. However, heat cure, self cure and visible light cure resin have similar mechanical properties in term of rigidity and transverse strength (**Dar-Odeh *et al.*, 1997**). These resins display less porosity than self cure acrylic resin, facilitate fabrication and adjustment, and 25% lighter and less polymerization shrinkage (**Tandon *et al.*, 2010**).

1.3.3.4 Mixed curing (Rapid heat polymerized resin)

These are hybrid acrylic, both chemical and heat activated initiators to permit rapid polymerization without the porosity that expected. After cooling the flask at room temperature, the denture can be deflasked, finished and polished in conventional manner (**Tandon *et al.*, 2010**).

The polymerization occurs within 20 minutes in water bath and one of its disadvantages was that it may cause tissue irritation because of high percentage of residual monomer (**Bartoloni and Walker, 2000**).

1.4 Modification of acrylic resin denture base

Clinical failure of complete or partial prosthesis made from PMMA resulting from fracture either due to fatigue or impact forces of mastication. Flexural fatigue of dentures as evidenced by midline fracture due to the stress concentration around the micro cracks, repetitive nature of masticatory load

results in propagation of cracks which weakens the denture base and finally results in fracture (**Alla *et al.*, 2013**).

To improve the strength of denture base, it can be made in three routes (**Von Fraunhofer, 2000; Saritha *et al.*, 2012**):

- Production of various alternatives to PMMA like polycarbonates, nylon and polyamides.
- Chemical modification of PMMA by addition of rubber in the form of butadiene styrene.
- Addition of fibers and/or nanoparticles.

1.4.1 Alternative materials to PMMA

After introduction of PMMA, most dental researches aimed to produce a material with high strength, good dimensional stability, low residual monomer, increased resistance to infections and increased radiopacity. Polycarbonate has used to produce dentures with good strength and high performance but it was injection molded technique and need complex equipment for processing (**Dhir *et al.*, 2007**).

1.4.2 Chemical modification of PMMA

The patient with high risk of dropping, the denture can be made from polymer reinforced with butadiene-styrene rubber. The result was high impact strength and low possibility to distortion. In this type the PMMA reinforced with rubber that will be scattered through the matrix (**Craig *et al.*, 2002**). The rubber particles are bonded to acrylic matrix and they supplied in powder and liquid form and conventionally processed. The monomer here either contain little or no cross- linking agent which differ from that of conventional one (**O'Brien, 2002**).

1.4.3 Application of nanotechnology in dentistry

Nanotechnology is the characterization, designing, application and production of devices, systems and structures by controlling the size and shape at nanoscale (**Dutta and Gupta, 2006**). Nanoscale points to objects of length ranging from 1-100 nanometer, a nanometer is a billionth of a meter: 10^{-9} m (**Hornyak et al., 2008**).

While nanodentistry is the technology and science of keeping perfect oral health with the use of nanomaterials likes tissue engineering, nano additives and nano coatings (**Freitas, 2000**).

The primary goal of using nanotechnology with dental materials to increase mechanical properties, high resistance to abrasion ,decrease the shrinkage of dental composite and to improve the esthetic of ceramic and composite (**Tsanka et al.,2009**), nano materials can be applied as following:

1. Nanocomposites

Nanocomposite is a multiphase material in which one phase has one, two or three dimensions of less than 100 nanometer. The thermal, optical, mechanical, electrical and electromechanical properties are completely different from those which make the composite like increased strength and high thermal and electrical conductivities (**Kamigaito, 1991; Ajayen et al., 2003**).

Nanocomposite differ from conventional composite because of high surface to volume ratio of reinforcing phase. This can reach up to 1000 m²/g filler. As a result, the interface is the determinant of macroscopic response of nanocomposite (**Michler and Baltá-Calleja, 2005**). The development of nano composite occurs in response to discouraging issues of polymerization shrinkage, strength, microhardness and wear resistance of polymers in dental applications (**Khurshid et al., 2015**).

2. Nano solutions

Nanosolutions are solutions with dispersed nanoparticles, which can be added to different paints, solvents and polymers, in them nanoparticles will be dispersed homogenously (**Jhaveri and Balaji, 2005**). Since nanoparticles are stable, that are not cluster nor settle out of dispersion, no shaking of the bottle is required, in contrast to larger size of fillers will settle out of solutions and require routine shaking before use (**Kumar and Vijayalakshmi, 2006**).

3. Impression materials

Nanofillers are added into vinylpolysiloxanes impression material, providing unique addition silicone impression material. This material has good flow, better detail production, fewer voids and improved hydrophilic properties (**Kumar and Vijayalakshmi, 2006**).

4. Bone replacement material

Nanotechnology was being used in bone replacement in which bone like nanoparticles were created with the aid of ultrasound and inserted into the human body in a paste form (**Jhaveri and Balaji, 2005**).

5. Polymer nanocomposites

Polymer nanocomposite are polymers which reinforced by small amounts (less than 5% by weight) of nano-sized particles (**Denault and Labrecque, 2004**). They are different from conventional filled composite, in the latter the reinforcement occurs in microns. The importance of polymer nanocomposite is to provide properties not present in the resin, without sacrificing resins' inherent mechanical properties, processibility or adding extra weight (**Vaia and Wagner, 2004**). The nanocomposite has many advantages, such as strength (without affecting the other mechanical properties) and improved heat resistance more than traditional polymers prepared with fillers in micro-sized scale (**Jordan et al., 2005**).

In any composite, there are three main material components: the matrix, the reinforcement (nano-filler) and the interfacial region. The interfacial region is the connection between matrix and filler and its properties variant from bulk matrix due to its proximity to the fillers (**Vaia and Wagner, 2004**).

In **2014**, **Jasim and Ismail** studied the effect of silanized alumina on some physical and mechanical properties of PMMA and they found that the addition of alumina nanoparticles to acrylic resin will improve the thermal properties and transverse strength at the same time this addition decrease water sorption and solubility. While in **2015**, **Muklif and Ismail** found that the addition of silanized alumina nanoparticles and polypropylene fibers to PMMA improved thermal properties, impact strength, surface hardness and roughness while non- significant difference in transverse strength.

The silanized ZnO nanoparticles and treated polyethylene fibers were added to PMMA and resulted in highly significant increase in impact strength and surface hardness, while significant decrease in water sorption and solubility and highly significant difference in transverse strength between groups (**Mohammed and Ismail, 2018**).

1.4.3.1 Factors affecting the properties of nanocomposite

1. Particle size

Decreasing the particle size will increase the surface area, and this will increase surface area to volume ratio which will improve the strength, electrical properties and becomes chemically reactive (**Vaia, 2002**).

2. Particle stiffness

High quantity of soft/elastic filler will increase the impact toughness but will decrease the modulus of elasticity, while increasing the hard/rigid fillers will increase the modulus of elasticity (**Viana, 2006**).

3. Effect of interfacial adhesion

Treatment of the filler surface of particle will increase interfacial adhesion which in turn affect the bond strength (**Miyagawa and Drzal, 2004**).

4. Nature of matrix material

- Thermoset resin: cross linked chains that does not melt when heated.
- Thermoplastic resin: long chains that soften when heated.
- Elastomers: rubber like or elastic (**Dharan and Tan, 2007**).

5. Distribution of particles within the matrix

When the nanoparticles are tightly bond and uniformly distributed within the polymer matrix, they will enhance fracture resistance of polymer. Despite of that, the nanoparticles may agglomerate due to high surface energy and may decrease the properties (**Deshmane et al., 2007**).

1.5 Silicon carbide (SiC)

Silicon carbide (SiC) has been used in the industry since last century, the synthesis method introduced by *Cowless* in 1885 and by *Acheson* in 1892. Silicon carbide represent important structural ceramic due to its special properties, these properties result from highly covalent (up to 98%) chemical bonding between silicon and carbon atoms (**Izhevskiy et al., 2000**).

The chemically stable form of carbon and silicon is silicon carbide (SiC) with closely packed double layer of Si and C atoms. The Si - C bilayers will determine the crystal structure of number of SiC polytype. There are about 200 polytypes (different crystal structure) of SiC in the present time, but the most common ones are the cubic (also called β -SiC) and hexagonal polytypes. The cubic form show excellent biocompatibility in vitro, also it is easy to nucleate, grow at low temperature and it has few commercial uses (**Ponraj et al., 2016**). The two principle polytypes of SiC are shown in figure (1.7)

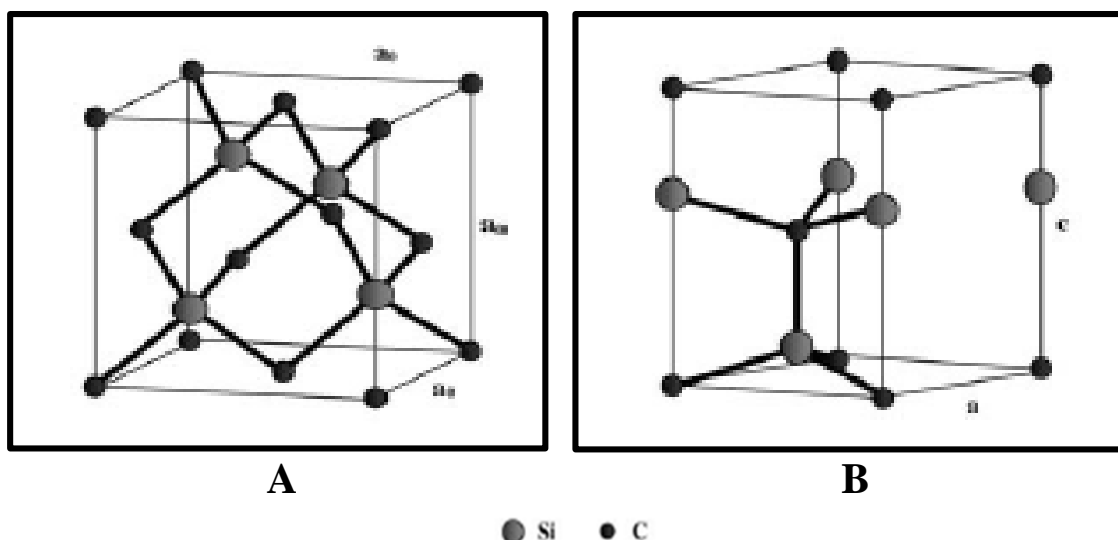


Figure (1.7): The basic units of SiC crystal structure, (A) hexagonal modification and (B) cubic modification (Presser and Nickel, 2008)

SiC is an important ceramic which has different applications. It has special properties such as thermal stability and conductivity (3.3 times that of Si), high hardness, elevated melting point, high strength and oxidation and erosion resistance. These properties make SiC very suitable for high temperature, high power electronic devices as well as abrasion and cutting applications. Since 1991 SiC is well known semiconductor and it can be used in applications where conventional semiconductors like silicon cannot perform reliably or adequately. SiC was manufactured using the traditional *Acheson* technique which initiated in 1892, (the raw materials for this process are quartz sand and petroleum cokes. The reaction is carried out by electrical resistance heating of the mixture of quartz sand and petroleum cokes to temperatures up to 2300°C. The SiC produced in the Acheson process is obtained in the form of large chunks. The silicon carbide produced in this way has a hexagonal crystal structure). Several fabrication methods are used such as: chemical vapor deposition (CVD), physical vapor deposition (PVD), liquid phase sintering (LPS) and metal alloying (MA) (Abderrazak and Hmida, 2011).

SiC is compatible with micromachining techniques and has special mechanical and chemical properties, so that it is a convenient material for biomedical microsystem. There are new application of SiC nanoparticles in nanomedicine (**Ponraj, 2016**) :

1. Medical implants field.
2. Cancer treatment applications.
3. Biomimetics.
4. Bio-labeling application.
5. Protein adsorption and filter.
6. Biomorphic ceramics.
7. Bio-sensing application.

SiC is a well known abrasive material in dentistry. They can be coated on a paper or polymer packed polishing disc, which are very effective in microfilled resin restoration (**Jefferies, 2007**).

1.6 Silane coupling agent

Different applications in industry, medicine and dentistry require connecting of dissimilar organic and inorganic materials for specific technical purpose. Due to difference in nature of chemical bonding of these materials, the interaction is very weak at the interfacial layer. This problem can be solved with introduction of coupling agent (**Lung and Matinlinna, 2012**).

Silane is a chemical compound that contains silicon (Si) atom or atoms with covalent bond to carbon atoms. The silanes could be monofunctional (in which one Si atom attached to three alkoxy groups), bifunctional (two Si atoms attached to three alkoxy group for each Si atom) or trifunctional (three Si atoms attached to three alkoxy groups for each Si atom) as shown in figure (1.8) (**Matinlinna et al., 2004**).

When filler particles bonded to resin matrix, this allows the flexible polymer matrix to transfer stresses to more rigid and stiffer (high modulus) filler particles. A properly applied silane coupling agent can improve physical and mechanical properties and decrease leaching by preventing water from penetrating along the resin-filler interface (**Anusavice, 2007**).

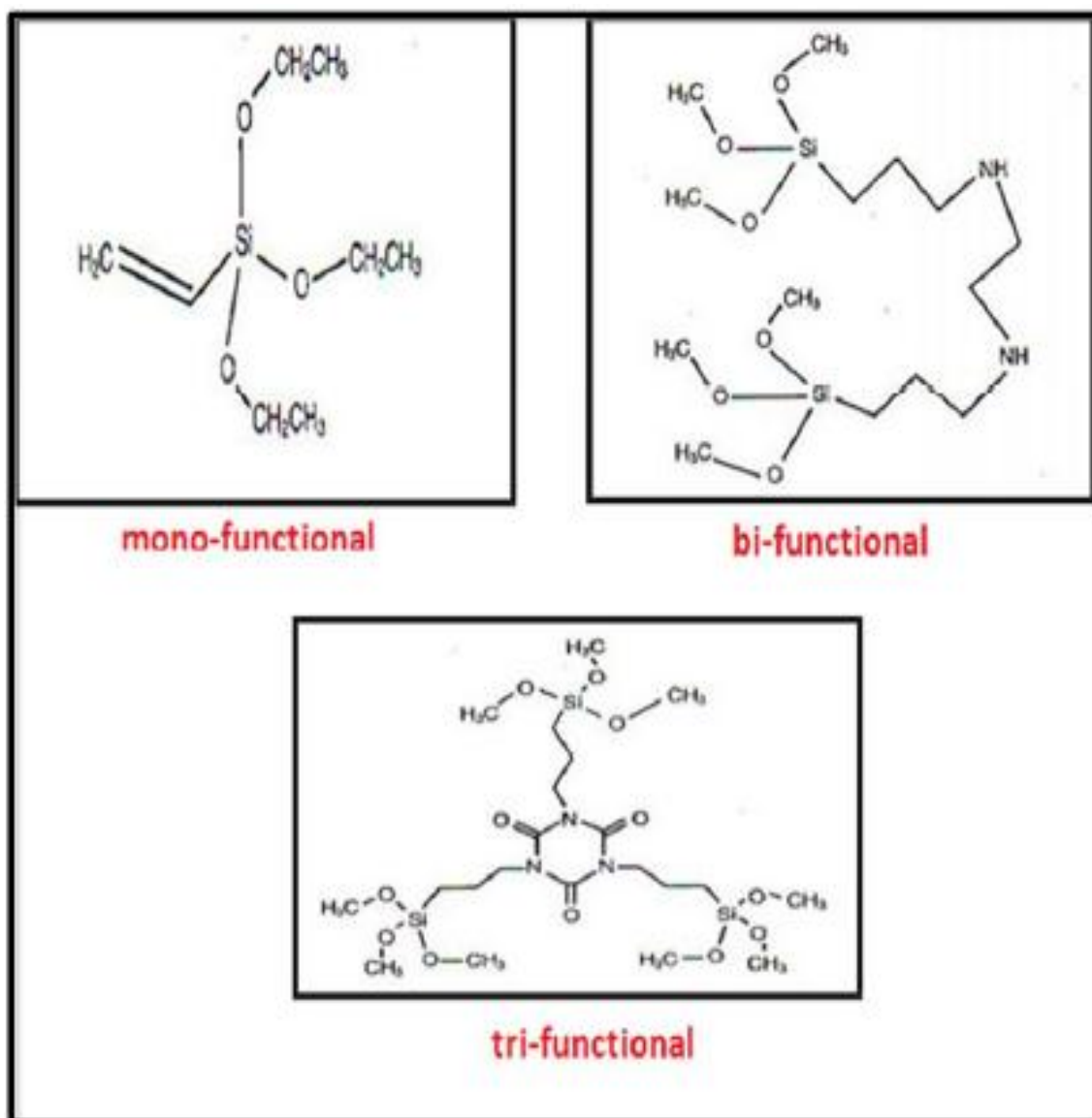


Figure (1.8): Chemical structure of silane (Matinlinna *et al.*, 2004)

Silane coupling agent plays important role in the composite, its functions were summarized by the following (**Powers and Sakaguchi, 2012**):

- It improves the mechanical properties of the composite and reduces the pulling of the fillers from matrix throughout clinical wear.
- It forms interfacial bridges that form strong binding between resin matrix and filler.
- The resultant interfacial phase provide medium to spread the stress to the matrix and adjacent filler particle.
- It provides hydrophobic medium that reduce the absorption of water by composite.

The silane coupling agent adsorbed on the fillers making bi-layer, chemisorbed and physisorbed silane layers (**Zulkifli, 2009**). The chemisorbed layer attached to the surface of filler by covalent bond. The physisorbed layer loosely bond over the first layer by hydrogen bond and van der Waals force among silane molecules. The mechanical and physical properties created by the layers of silane depend on amount of coupling agent, nature of silane, pH, drying condition used in silanization and rate of hydrolysis and condensation (**Arksornnukit et al., 2004**). There are many forms silane coupling agents designed for special bonding between different fillers and resin matrix (**Yuen et al., 2008**). The solubility parameter is used to consider the penetration of silane coupling agent into resin matrix (**Suzuki and Ishida, 2003; Loch et al., 2007**).

Tri-methacryloxypropyltrimethoxysilane (MPS) was selected based on the solubility parameter for methyl methacrylate MMA and PMMA bonding (**Krasnoslobodtsev and Smirnov, 2002**).

Tri-methacryloxypropyltrimethoxysilane (MPS) is the main silane coupling gent used in dentistry to modify the surface of glass and silica fillers mixed with resin matrix (**Matinlinna et al., 2004**), the chemical structure is seen in figure (1.9).

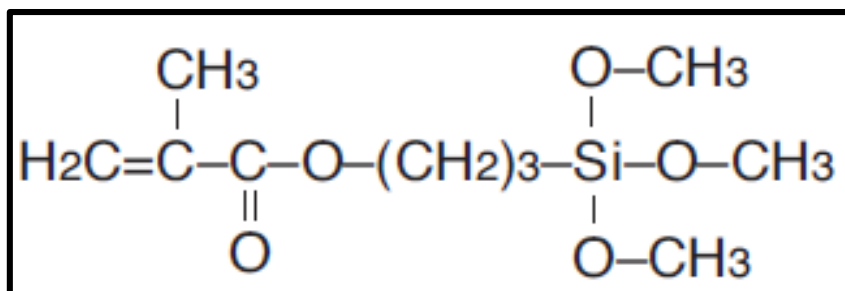


Figure (1.9): Structural formula of 3-methacryloxypropyltrimethoxy silane (MPS)
(Taira *et al.*, 2012).

In 2007 Khalid *et al.* found that when using methacrylic acid as a coupling agent in the formation of TiO₂-PMMA nano-composite, the result is that the nano-composite show better elastic properties and have applications in dental composite and bone cement.

Adding alumina fillers silanized with 0.1% weight (MPS) result in significant increase in flexural strength and wear resistance (Chaijareenont *et al.*, 2012).

1.7 Some properties of PMMA

1. Thermal conductivity

Thermal conductivity is one of the important thermal properties of dental materials and can be simply defined as the capability of the material to transfer heat (Bagby and gladwin, 2013).

The difference in thermal conductivity of denture base materials, in the same manner result in difference of soft tissue response. A metal base is good thermal conductor and can cause instant tissue response, while acrylic resin delayed response to thermal changes. The good thermal conduction is preferred for the denture base to maintain good health of the supporting tissue by making the heat rapidly conducted to and from the tissue by denture base (Powers and Sakaguchi, 2006).

The thermal conductivity of a material is the quantity of heat in calories or joules per second passing through a body of 1 cm thickness with cross-section 1 cm^2 when the difference in temperature is 1°C . The thermal conductivity is measured under steady-state condition in which temperature gradient does not change. According to second law of thermodynamics, heat flows from areas of higher temperature to areas of lower temperature (**Anusavice *et al.*, 2013**).

In **1998 Messersmith *et al.*** stated that studies showed the physical characteristics of denture base material can affect the acceptance of patient to the prosthesis by changing sensory experience of food during mastication. The thermal characteristics of denture base material can affect the ability to sense transient temperature change at the palate.

Acrylic resin is poor thermal conductor this may cause the patient to consume a drink that is too hot without realizing this, which make the tissues in the back of oral cavity are continuously exposed to temperature change in food / liquid intake (**Noort, 2002**). Thermally conductive polymer offers new possibilities for replacing metals in several application plus the advantages of polymers such as light weight, corrosion resistance and ease of processing. Therefore, the current interest is to improve the thermal conductivity of polymers by addition of nanofillers with high thermal conductivity (**Han and Fina, 2011**).

Therefore, to enhance the thermal conductivity of polymers, carbon based thermal conductive fillers added to polymer matrix to enhance thermal conductivity (**Wu *et al.*, 2017**).

2. Impact strength

Impact strength is the measure of energy absorbed by a material when sudden blow applied to break it (**Powers and Sakaguchi, 2006**). Impact strength is important for acrylic denture base material because it has tendency to break if they dropped on hard surface (**Powes and Wataha, 2013**).

The addition of plasticizer into denture base will improve the impact strength, but compressive strength and hardness will decrease. Large number of methods for measurement of impact strength are available, but the most common types are those using swinging pendulum which fall into two categories (**Craig and Powers, 2002**):

1. Charpy test, the specimen is supported horizontally at both ends.
2. Izod test, the specimen is clamped at one end and held vertically

Zappini et al. in 2003 compared the impact strength of seven heat polymerized denture base resin, they found that the impact strength influenced by loading condition and specimen geometry.

3. Transverse strength

Transverse strength is described in dental, engineering and technical literature as modulus of rupture or flexural strength, it sometimes called three point bending (3PB) test (**Craig and Power, 2002**). It can be defined as the resistance of material to fracture (**Jagger, et al., 2002**) as shown in figure (1.10).

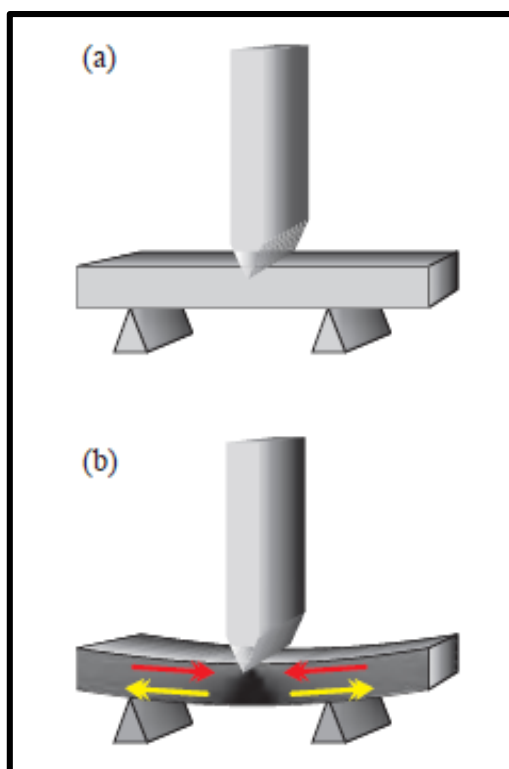


Figure (1.10): (a) Diagrammatic representation of three points bending test. (b) Bending of the beam introduce both tensile (yellow arrows) and compressive (red arrows) stresses (McCabe and Walls, 2008).

Transverse strength measurements are used more frequently than tensile and compressive strength, because transverse strength very closely resemble the type of loading to which the denture subjected inside the mouth and it obtained when load applied in the center of a beam supported at both ends (**powers and sakaguchi, 2006**). The amount of residual monomer influence the transverse strength, the less the amount of residual monomer the higher will be the transverse strength (**Hamza *et al.*, 2004**).

For brittle materials, the flexural strength can significantly influenced by surface defect such as notches or any imperfection can cause stress concentration on the surface and lead to fracture of the material at lower force than expected. The fracture is initiated on the side of the specimen which subjected to tension force (**McCabe and Walls, 2008**).

4. Surface hardness

The ability of material to resist indentation . The material is said to be hard when it resist hard indenter such as diamond (**Powers and Wataha, 2013**). Many methods can be used to test surface hardness and choosing the optimum test will depend on the hardness range and type of material (**Powers and Sakaguchi, 2012**).

Unalan and Dikbas in 2007 found that there was an improvement in the surface hardness of acrylic with addition of 5%wt. , 10%wt. and 15% wt. of silane treated mica and glass fillers.

Hu et al. in 2009 found the addition of nanozirconia to acrylic cause slight increase in hardness at 5%wt. concentration and great increase in hardness at 15% wt. concentration. **Safi in 2011** added modified ZrO_2 nanoparticles to acrylic and cause increase in hardness and this increase of hardness is directly proportional to the increase in concentration of modified ZrO_2 nanoparticles.

5. Surface roughness

Surface roughness is defined as fine irregularity present on the surface due to manufacturing process. Irregularities and porosities are present on denture surface played major role in reducing the activity of denture cleaning agent and hence increased stain and plaque retention (**Zissis et al., 2000**).

The surface properties of any denture base material are very important because the studies on these materials has shown direct relationship of roughness with accumulation of plaque and adherence of candida albicans. For prosthetic and dental restorative materials, and conventional polishing method of PMMA with lathe and pumice provide more smooth surface and decrease surface roughness (**Barbeau et al., 2003**).

Ogle *et al.* in 1986 studied the surface of dental stone, surface of heat cured resin, auto polymerizing acrylic resin and visible light cure acrylic resin. The result showed that the heat cured resin had the smoothest surface.

Abdul Ammer in 2006 mixed titanium powder and zinc oxide with PMMA powder and found the roughness of PMMA increased with increase the amount of zinc oxide and titanium powder.

Safi in 2011 found the roughness of PMMA was not significantly increased with addition of ZrO_2 nanoparticles.

Jasim and Ismail in 2013 found that no change in surface roughness was seen when acrylic resin was mixed with silanized Al_2O_3 nanoparticle.



Chapter Two

Materials and Method

Materials and Methods

2.1. Materials

The materials used in the study are shown in figure (2.1)

- Dental pumice. Silky rock (Turkey).
- Distilled water (Iraq).
- Ethanol as solvent 99.9% concentration (Iraq).
- Heat curing acrylic resin (Vertex, regular heat curing denture base material, Netherlands) polymer and monomer.
- Separating medium (ISODENT, Finland).
- Silicon carbide nanopowder (SiC, Beta, 99.9% purity, <80 nm, cubic). (Nanoshel, USA).
- Toluene analytical reagent (GCC Gainland Chemical compound, UK).
- Tri-Methacryloxypropyltrimethoxysilane (MPS) coupling agent, (Cheng du micxy chemical co. ltd. 2530-86-0. China).
- Type III hard dental stone, Elite rock (Zhermack, Italy).



Figure (2.1): Some of the materials used in the study.

2.2 Equipments and instruments

1. Computer controlled universal testing machine (WDW-20, Laryee Technology Co., Ltd., China).
2. FTIR (Fourier Transform Infrared Spectrophotometer, SHIMADZU, Japan).
3. SEM (Scanning electron microscope) (FEI, Inspect S50, Netherlands).
4. Rotary pump coater (Quorum, Q150R ES, UK).
5. Thermal conductivity testing machine (HOT DISK, Sweden).
6. Impact testing machine (Tmi, testing machine Inc. Amity Vielle, New York, USA).
7. Profilometer (test for surface roughness). (TEREN, DaLian, China).
8. Durometer (Shore D hardness tester , China).
9. Magnetic stirrer (LABINCO, Netherland).
10. Probe sonication apparatus (Soniprep-150, England).
11. CNC-CO2 laser engraving cutting machine (JL-1612, Jinan Link Manufacture and Trading Co., Ltd., China).
12. Plastic patterns.
13. Electronic balance (accuracy 0.0001g, Germany).
14. Dental metal flasks (Broden, Sweden).
15. Clamps (Hanau, USA).
16. Hydraulic press (Germany).
17. Dental vibrator (Bego, Germany).
18. Water bath (England).
19. Rubber bowel, wax knife, spatula, vernier and ruler (China).
20. Prosthetic hand piece (Marathone, China).
21. Finishing burs: fissure burs, acrylic burs and sand paper burs.
22. Lathe polishing machine (Germany).
23. Disposable syringe and polyethylene cup (China).

2.3 Methods

2.3.1 Pilot study

Pilot study had been conducted to select which concentration of silicon carbide nano particles can be used in the study as filler for heat cure acrylic denture base.

Three specimens for each concentrations of (0.5%wt.), (0.25%wt.) and (0.125%wt.) of SiC in addition to threespecimens for control group were prepared in each test. The tests that performed were thermal conductivity, impact strength and transverse strength. The total specimens in pilot study were 36 specimens as shown in table (2.1).

Table (2.1):Specimen grouping and numbering for pilot study

Concentration of nanoparticles	Thermal conductivity test	Impact strength test	Transverse strength test
0% SiC group	3 specimens	3 specimens	3 specimens
0.125%wt SiC group	3 specimens	3 specimens	3 specimens
0.25%wt SiC group	3 specimens	3 specimens	3 specimens
0. 5% wt SiC group	3 specimens	3 specimens	3 specimens

According to the results obtained from the pilot study, the addition of (0.25% wt.) and (0.125% wt.) result in greatest values of impact strength and transverse strength among the studied groups and also because at concentration 0.5% wt. the color of specimens turns to dark grey which is objectionable therefore, the selected concentrations are (0.25% wt.) and (0.125% wt.) were used to complete the main study as shown in table (2.2).

Table (2.2): The means values of tests used in pilot study

Amount of nano SiC	Impact strength Kj/m ²	Transverse strength N/mm ²	Thermal conductivity W/m.k
Control group 0% SiC	0.26	88.56	0.437
0.125% wt. SiC	0.25	87.35	0.734
0.25% wt. SiC	0.24	85.13	0.897
0.5% wt. SiC	0.20	84.87	0.908

2.3.2 Specimen grouping

One hundred fifty (150) specimens were prepared from heat cured acrylic resin. The specimens were divided into 5 groups according to the used tests. Each group consisted of 30 specimens which are subdivided into:

- 1) Control group of 10 specimens of acrylic resin without SiC nano particles.
- 2) 10 specimens of acrylic resin with 0.25%wt. SiC nano particles.
- 3) 10 specimens of acrylic resin with 0.125%wt. SiC nano particles.

As shown in figure (2.2)

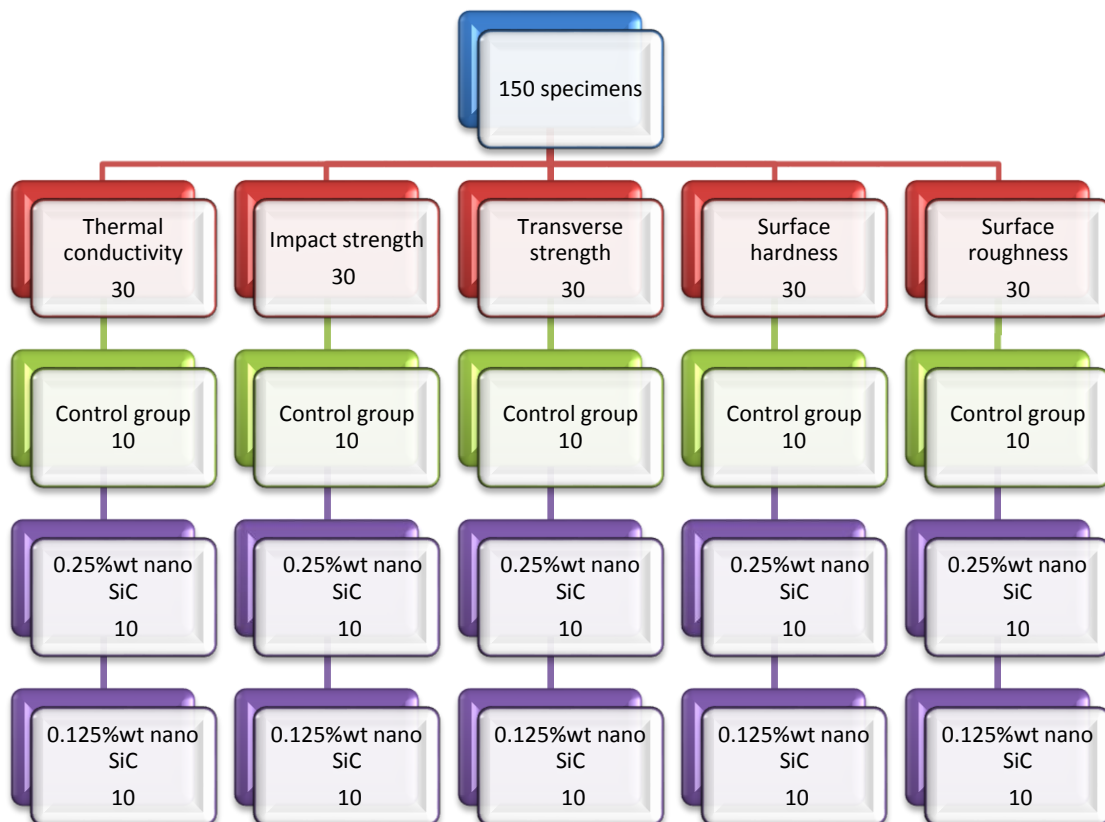


Figure (2.2): Specimen grouping and number of specimens in each group.

2.3.3 Fourier Transform Infrared (FTIR)

Silicon carbide nanoparticles were treated with silane coupling agent in order to introduce reactive groups onto nanoparticles for better binding to polymer matrix.

Tri-trimethacryloxypropyltrimethoxysilane (MPS) was selected based on solubility parameters of PMMA and methyl methacrylate (MMA) (**Choi and Chung, 2003**). The amount of (MPS) was selected based on previous study which use 25% wt. silane coupling agent (**Jasim, 2013**) who added alumina nanoparticles to heat cured acrylic resin denture base material.

One hundred milliliter aqueous solution of ethanol (70% volume) was prepared by diluting absolute ethanol with distilled water and the PH of the solution was adjusted to 4.5 with 99.9% acetic acid and PH meter. After that, 25% wt. MPS was added into ethanol aqueous solution and stirred. This solution is stored in polyethylene cup and covered for 5 minutes for hydrolysis and silanol formation. Then, 100g of silicon carbide nanoparticles added to MPS solution. The mix was stirred with magnetic stirrer for 30 minutes then, the mix was sonicated with probsonication apparatus for 30 minutes and the solution left to dry at room temperature for 14 days (**Pisaisit *et al.*, 2012**).

The (FTIR) spectrophotometer was made to determine whether or not functional group of MPS are attached to nanoparticle by analyzing characteristic vibrations of functional groups (**Shi *et al.*, 2004**).

2.4 Preparation of test specimen

2.4.1 Plastic patterns preparation

Three different plastic patterns was made to prepare mould for acrylic specimens, these plastic patterns were made by cutting plastic plate with laser

cutting machine into the desired shape and dimension according to the required test as shown in figure (2.3).

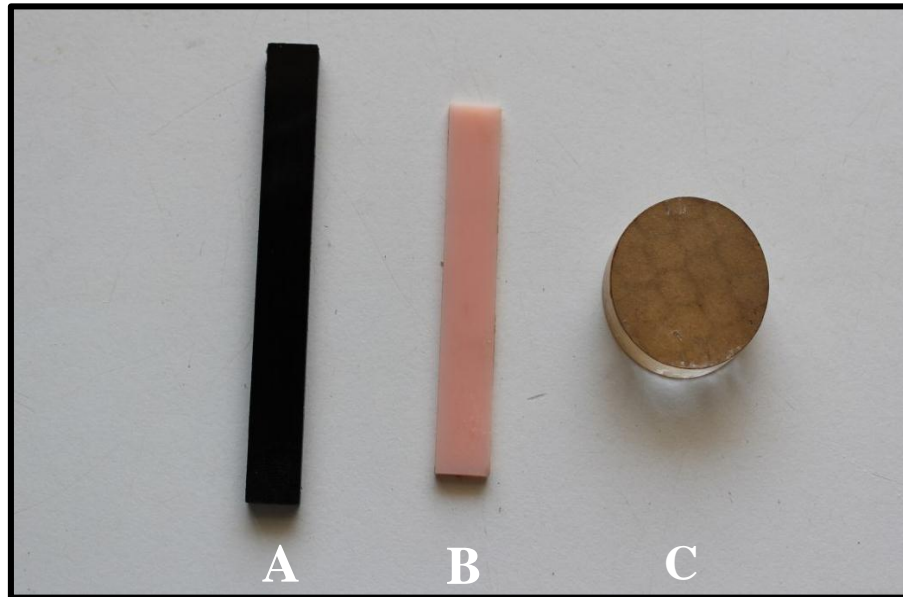


Figure (2.3) Plastic patterns A. Impact strength pattern. B. Transverse strength, surface hardness and surface roughness pattern. C. Thermal conductivity pattern.

2.4.2 Mould preparation

The same conventional way for complete dentures processing is followed to make acrylic specimens. Plastic patterns are coated with separating medium (cold mold seal) and allowed to dry. Dental stone is mixed according to manufacture instruction (100g of powder with 20 ml of water) and added to the lower half of the flask and vibrated to prevent air bubbles formation. The plastic patterns inserted approximately a half of their depth and the stone allowed to dry as shown in figure (2.4). After that the lower half coated with separating medium and allowed to dry, then the upper half is placed and filled with stone and vibrated then the lid of flask is placed and left to set. Following the setting of stone the two halves are separated and the plastic patterns are removed to create mould cavity for acrylic specimen, then separating medium was placed and allowed to dry after that the flask ready for packing of acrylic specimen.



Figure (2.4): Plastic patterns in the lower half of the flask.

2.4.3 Preparation of acrylic resin

The preparation of acrylic resin was made by mixing polymer and monomer according to manufacture instruction (2.3g to 1ml). The amounts of polymer, monomer and silicon carbide nanofiller in different concentrations that used in the study are shown in table (2.3) by using electronic balance with accuracy of (0.001g) as shown in figure (2.5).

Table (2.3): Mixing ratio of acrylic resin and SiC

Groups	Amount of nano SiC (g)	Amount of polymer (g)	Amount of monomer (ml)
Control	0	23	10
0.125% wt. SiC	0.0287	22.9713	10
0.25% wt. SiC	0.0575	22.9425	10



Figure (2.5): Electronic balance.

2.4.4 Addition of silicon carbide nanofillers

In order to make the dough with particular concentration of nanofiller, the measured amount of nano SiC was added to the monomer and the nano particles are well dispersed in the monomer by using probe sonication apparatus (120 W, 60 KHz) for 3 minutes to break them into individual nano particle as shown in figure (2.6) (**Mohammed *et al.*, 2009**).



Figure (2.6): Monomer and nanofillers are sonicated in probe sonication apparatus

After that the materials are mixed and manipulated according to manufacture instructions. The monomer with nano powder is immediately mixed with acrylic powder in order to reduce the possibility of particle aggregation and phase separation, then the mix is covered and left until dough stage was reached.

2.4.5 Packing of acrylic resin

Packing of acrylic started after the acrylic resin reached the dough stage, the acrylic removed from the mixing jar and then placed in the mould cavity which was previously coated with separating medium, and the two parts of the flask closed together and the closed flask placed under the hydraulic press as shown in figure (2.7), and the pressure slowly applied until the flask is tightly closed (at 100 psi) and the excess acrylic flows out, then the flasks are clamped with flask clamp and it is ready for curing as in figure (2.8).



Figure (2.7): Flasks under the pressure of hydraulic press

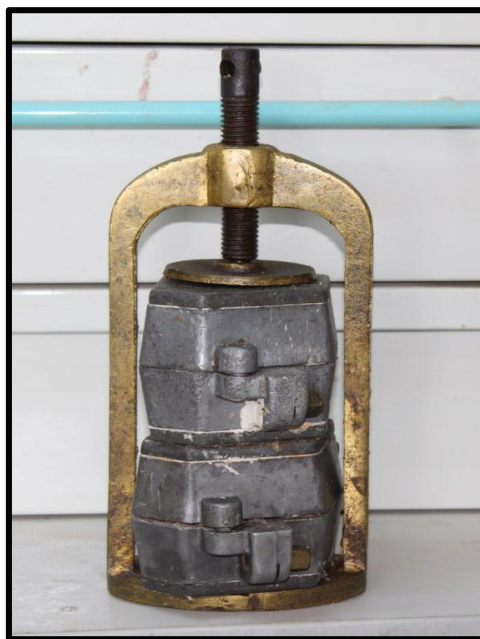


Figure (2.8): Flasks are clamped with flask clamps

2.4.6 Curing of acrylic resin

The curing of acrylic resin is performed by placing the clamped flasks into the water bath and the temperature begin from room temperature and raised to the 100°C the clamps were left for 30 minutes according to manufacture instructions as in figure (2.9). After complete curing, the metal flask is allowed

to cool at room temperature for about 30 minutes then the flasks are opened and the acrylic specimens are removed carefully from the mould.

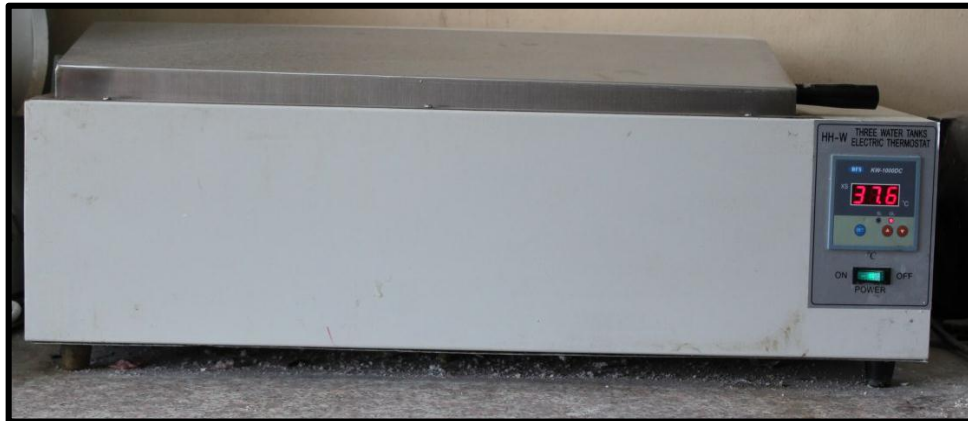


Figure (2.9): Water bath for acrylic resin curing

2.4.7 Finishing and polishing

All acrylic resin specimens (except specimens for surface roughness test) are finished with finishing burs and polished with lathe polishing machine. The speed of polishing machine is about 1500 rpm to prevent excessive heat which will distort the specimens and the polishing was made with rage wheel until glossy surface using pumice that mixed with water.

2.5 Experimental tests

The following tests were performed for the prepared acrylic resin specimens:

1. Thermal conductivity test.
2. Impact strength test.
3. Transverse strength test.
4. Surface hardness test.
5. Surface roughness test.

2.5.1 Thermal conductivity test

A. Test specimens

The specimens of heat cure acrylic resin are in the shape of disk with dimensions of (40mm in diameter and 2.5mm thickness) according to instrument specification as in figure (2.10)

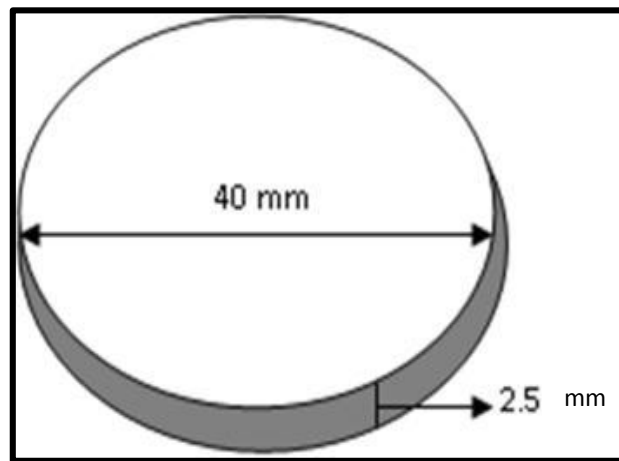


Figure (2.10) Thermal conductivity test specimen dimensions

B. Parts of thermal conductivity machine

- **Hot disk (Thermal contact analyzer)**

The hot disk thermal constant analyzer as shown in figure (2.12) can be used for measuring the thermal transport properties for a large variety of materials with thermal conductivities between 0.005 and 500 W/m.K. Naturally the parameter heating power measuring time and radius of disk, by which the experiment controlled and should be selected with care to have results within given limits of accuracy as in figure (2.12).

- **Hot disk sensor**

The hot disk sensor consist of electrically conducting pattern in the shape of double spiral extend out of thin sheet of Nikel. The Nikel was chosen because of its high thermal conductivity and known temperature coefficient of resistivity. The conducting pattern was supported on both sides with thin electrically insulating material as in figure (2.11).

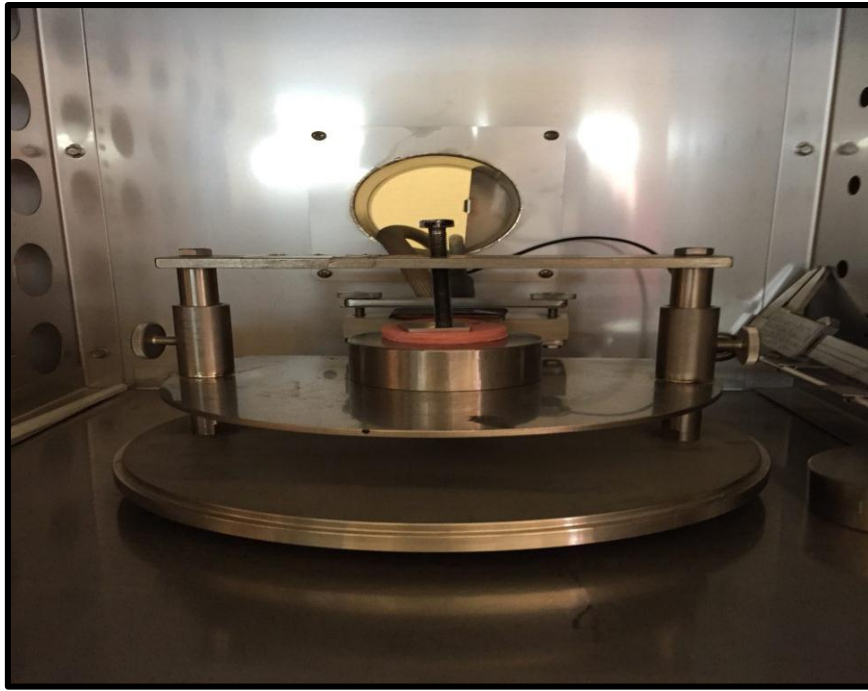


Figure (2.11): Hot disk sensor.

C. Testing procedure

The equipment are connected to the computer which was programmed for the test. By selection the experiment type, the method will be selected automatically. The experiment is called (transient plane source) as in figure (2.12).



Figure (2.12): Thermal conductivity testing machine

Steps of testing method (according to manufacture instructions):

1. Starting the hot disk unit by turn on the switch on the back side of the unit.
The hot disk should be turned on 60 minutes prior to starting experiment.
2. Turn on the computer and start the hot disk analyzer software.
3. Prepare the stainless steel plates between which studied specimen will be placed.
4. Select the experimental parameter (parameter of polymer).
5. Placing each specimen on the sensor of the hot disk and take the reading from the computer after 15 minutes.

2.5.2 Impact strength test**A. Test specimens**

The specimens of acrylic resin for impact strength were prepared with dimensions of (80mm length \times 10mm width \times 4mm thickness) (**ISO 179, 2000**) as in figure (2.13). The test specimens were stored in distilled water at 37°C for 48 hours before the test performed (**ADA specification No. 12, 1999**).

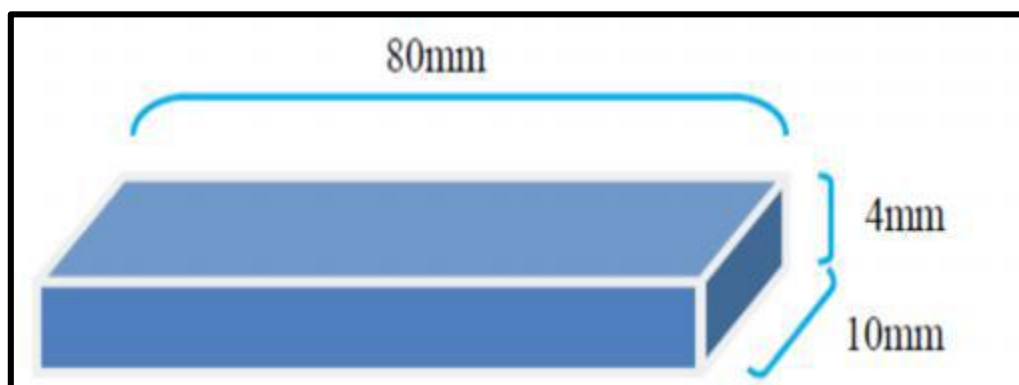


Figure (2.13) Impact strength test specimen dimensions.

B. Test procedure

The impact strength test was performed following the procedure recommended by ISO 179 with impact testing device as in figure (2.14).

Each specimen was supported horizontally at end and struck by swinging pendulum of 2 Joules. The scale gives the reading of impact energy in joules. The charpy impact strength was calculated in kilo-joules per square meter by the following equation:

$$\text{Impact strength} = \frac{E}{b.d} \times 10^3 \text{ (ISO No. 179, 2000) where:}$$

E: The impact energy in Joules

b: The width of specimens in millimeters

d: The depth of specimens in millimeters

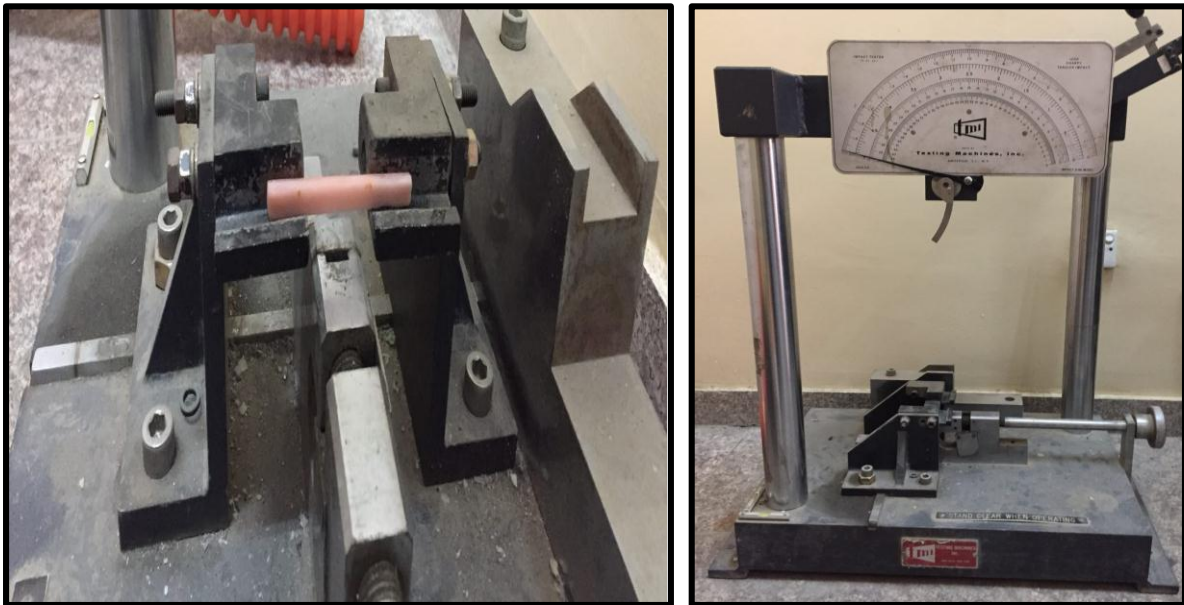


Figure (2.14) Impact strength test

2.5.3 Transverse strength test

A. Test specimen

Specimens that used in this test were prepared with dimensions of (65mm length x 10mm width x 2.5mm thickness) as in figure (2.15). All of the specimens are kept in distilled water at 37°C for 48 hours before being tested according to ADA specifications No.12, 1999.

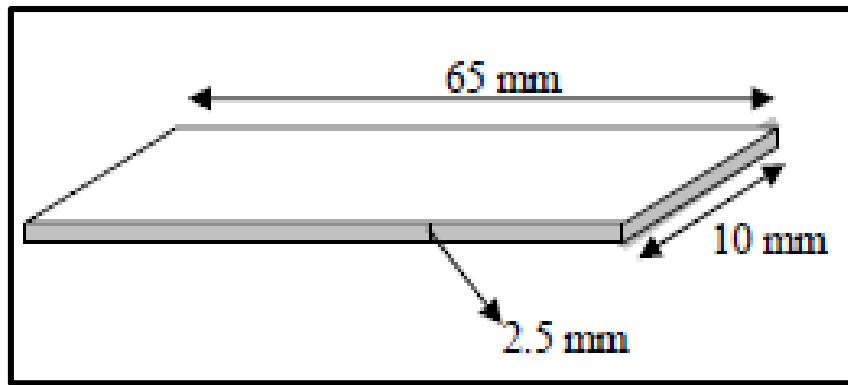


Figure (2.15) Transverse strength test specimen dimensions

B. Test procedure

The test was performed with Instron universal testing machine as seen in figure (2.16), the specimens was positioned on bending fixtures that made of two parallel supports with (50 mm) apart, and the load was applied across the head with speed 1mm/min. through a rod that placed centrally between the supports to make deflection until fracture occurs as in figure (2.17).

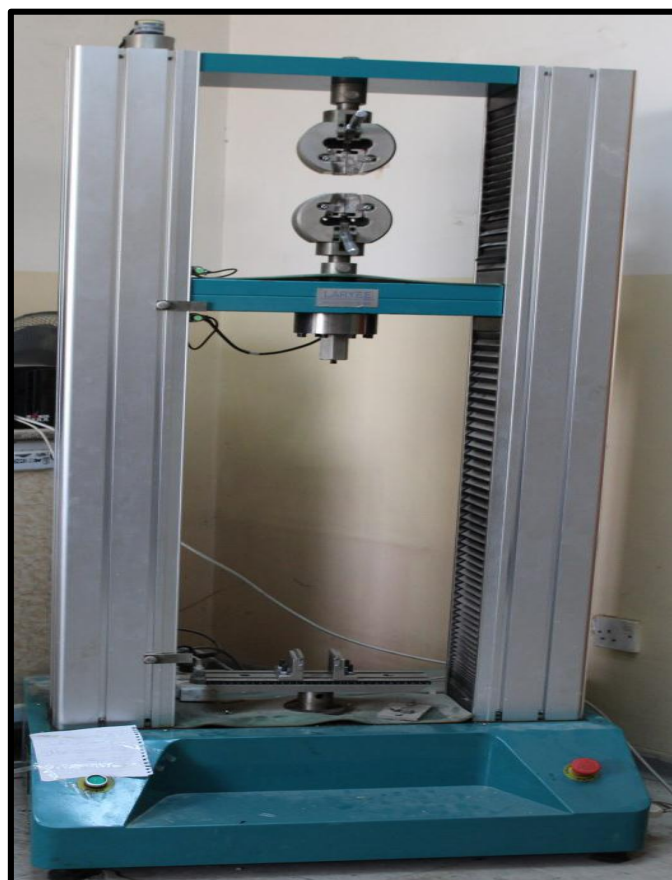


Figure (2.16) Instron universal testing machine

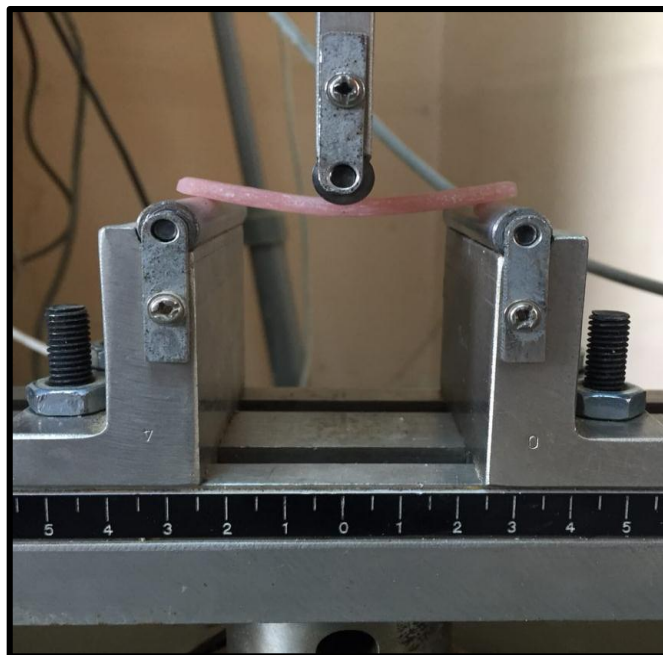


Figure (2.17) Specimen bending before fracture

The transverse strength was calculated using the following formula:

$$T = \frac{3PL}{2bd^2} \text{ (ADA specification No.12, 1999), where:}$$

T: Transverse strength (N/mm²)

P: Maximum force applied on specimens (N)

L: Distance between supports (mm)

b: Width of specimens (mm)

d: Depth of specimens (mm)

2.5.4 Surface hardness test

A. Test specimen

The specimens of acrylic resin for surface hardness test were prepared with dimensions of (65mm length × 10mm width × 2.5mm thickness) as shown in figure (2.18). All the specimens were kept in distilled water at 37°C for 48 hours before the test was performed (**ADA specification No.12, 1999**).

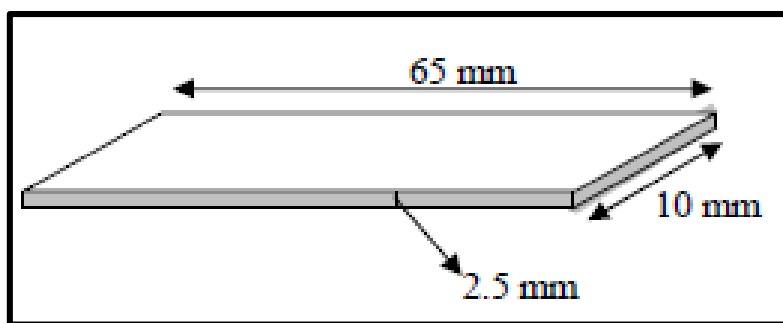


Figure (2.18) Surface hardness test specimen dimensions

B. Testing procedure

Surface hardness was performed by (shore D) durometer hardness tester according to ADA specification No.12, 1999 which is suitable for acrylic resin.

The device consists of spring loaded indenter (0.8 mm in diameter) which is attached to digital scale starting from 0 to 100 unit as in figure (2.19). The usual method was to press on the indenter and record the maximum reading as in figure (2.20). Three readings were taken two at each end and one on the center and the mean of three readings was recorded.



Figure (2.19) Shore D hardness tester



Figure (2.20) Specimen testing with hardness device

2.5.5 Surface roughness test (Ra)

A. Test specimen

The test specimens of acrylic resin for surface roughness test were prepared with dimensions of (65mm length \times 10mm width \times 2.5mm thickness) as in figure (2.21). All of specimens were stored in distilled water at 37° C for 48 hours before the test performed (**ADA specification No.12, 1999**).

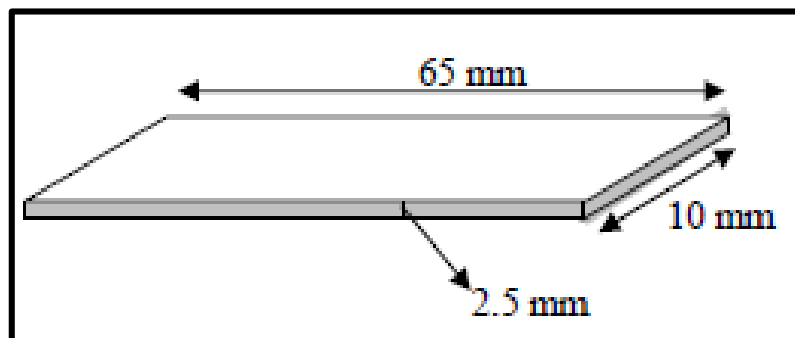


Figure (2.21) Surface roughness test specimen dimensions

B. Testing procedure

The microgeometry of test specimens was studied using profilometer (Figure 2.22). This device consists of surface analyzer (sharp stylus made from diamond) to trace the profile of surface irregularities. The maximum distance the stylus can move is 11 mm and it records all the peaks and recesses that characterize the test specimen surface, three areas were chosen one in the middle and two at both ends and the mean of three readings was calculated.

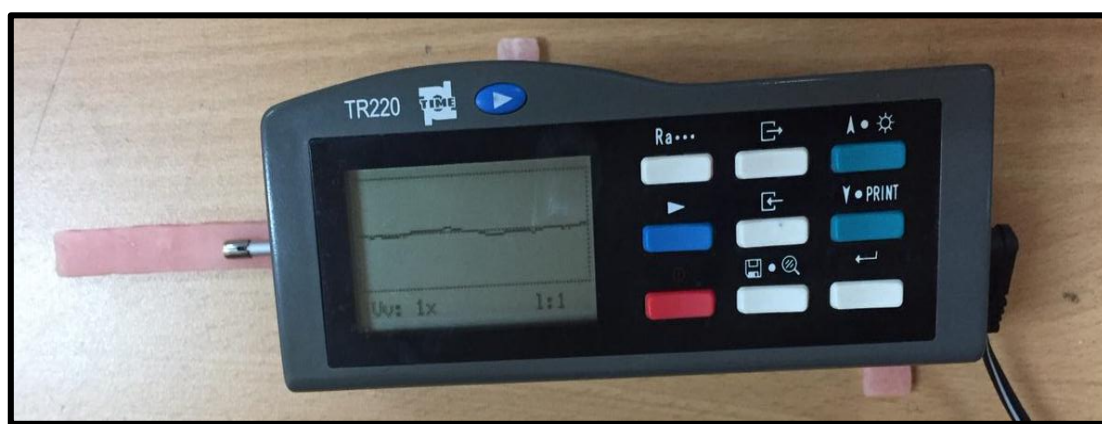


Figure (2.22) Digital profilometer

2.6 FTIR (Fourier Transformed Infrared) Spectroscopy

Fourier transform infrared test was performed to SiC nanopowder, silane (tri-methacryloxypropyltrimethoxysilane) and the resulting powder after nanopowder had been silanized to determine whether or not there is chemical bond between SiC and silane coupling agent. A little amount of the material is scraped and placed directly on special plate of FTIR spectrophotometer for test as in figure (2.23).



Figure (2.23) FTIR spectrophotometer

2.7 Scanning Electron Microscope (SEM)

The scanning electron microscope was performed to test the control specimen of acrylic resin and the specimen containing SiC nanoparticles (0.25% wt.) in order to show the nanoparticles distribution within matrix of polymer with magnification up to 2000X (figure 2.24).



Figure (2.24) Scanning electron microscope

The specimens for scanning electron microscope was prepared by cutting a small piece from acrylic resin specimen (only small enough to be mounted on special plate of SEM) , then they are painted with gold by rotary pump coater (plasma sputter) as shown in figure (2.25) and Tungsten gas, after that the specimens are examined with scanning electron microscope .



Figure (2.25): Rotary pump coater.

2.8 Energy Dispersive X-ray Spectroscopy (EDS)

To determine whether the silicon carbide nanoparticles get blended with the matrix of polymer (PMMA) or not, EDS test was done. This test was performed to determine the elemental analysis or chemical characterization of the specimen. Two specimens were studied one is control specimen and the other contain SiC nanoparticles of concentration 0.25% wt. (Figure 2.26).



Figure (2.26) EDS testing machine

2.9 Statistical analysis

The data of the specimens was collected and computerized using (SPSS) statistical package for social science version 20.

The statistical analysis include the following:

1. Descriptive statistics which include:

- Mean
- Standard deviation (SD)
- Standard error (SE)
- Minimum (Min)
- Maximum (Max)
- Bar charts representing the mean of readings

2. Inferential statistics

ANOVA (one way analysis of variance) for assessing the difference between more than two groups. If the difference was statistically significant Tukey HSD was used to examine the difference between each 2 groups.

P value

$P \geq 0.05$ (NS) Non- significant

$P < 0.05$ S Significant

$P \leq 0.01$ (HS) High significant

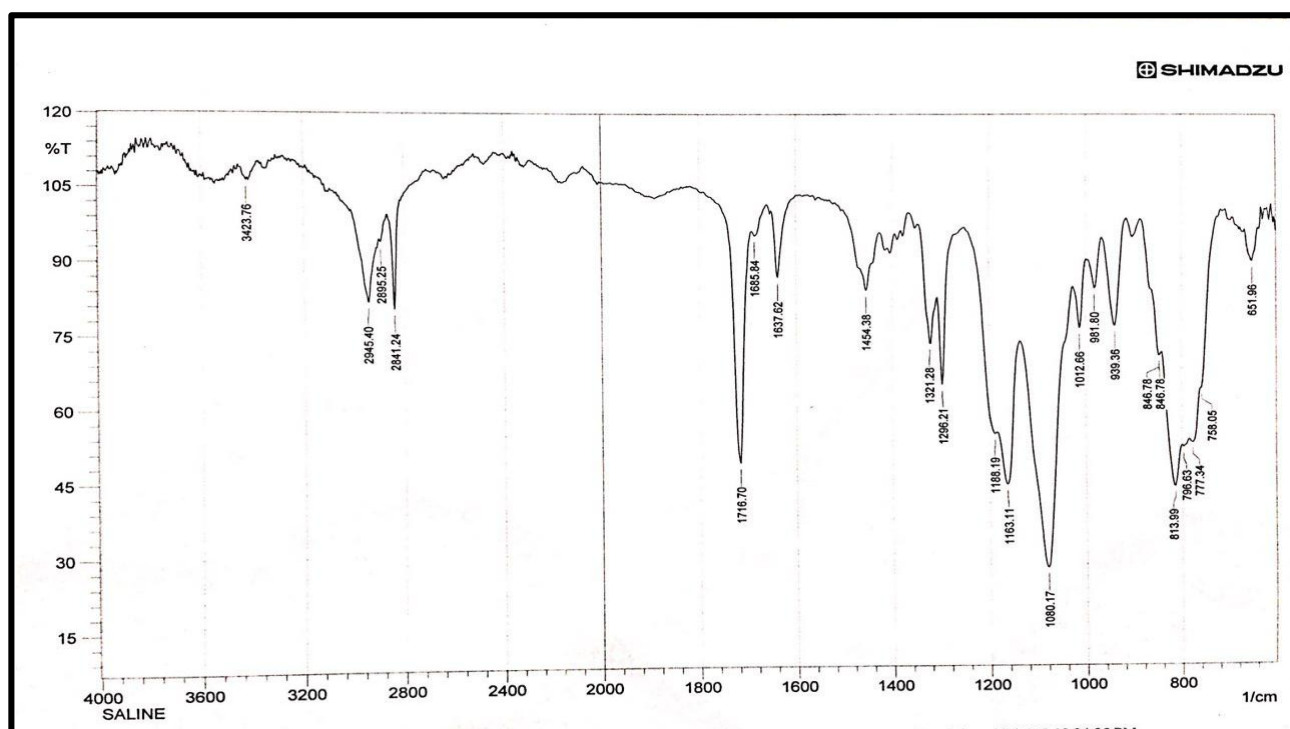
Chapter Three

Results

Results

3.1 Characterization of silanated silicon carbide nanofillers (SiC)

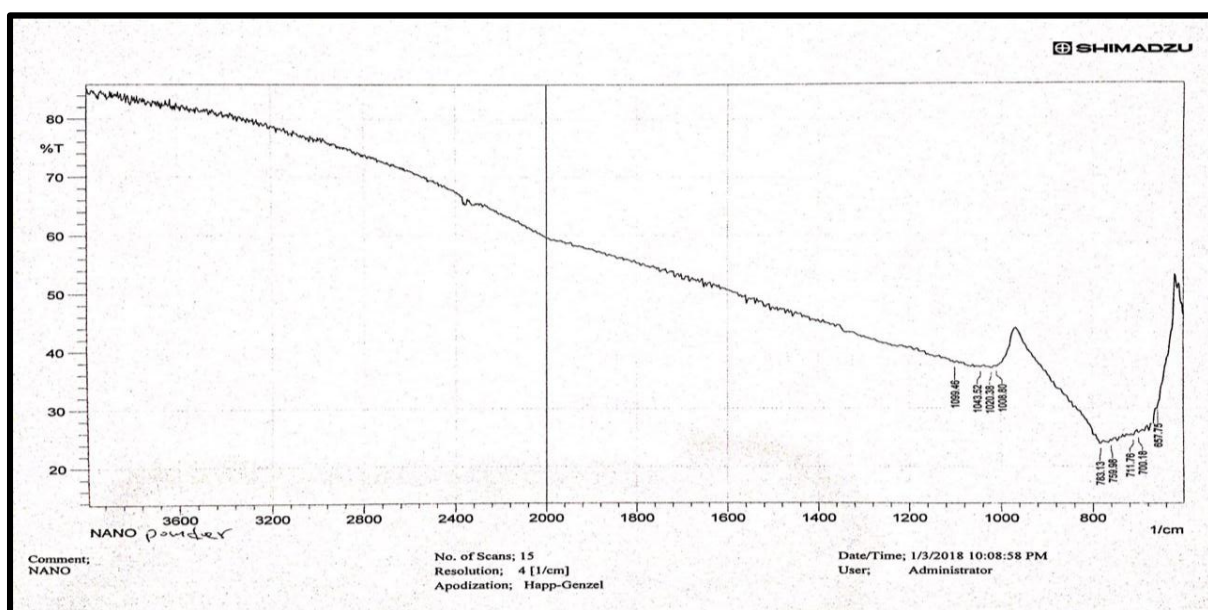
Characterization of absorption bands of the material was indicated by using FTIR. The absorption bands of MPS can be assigned to the presence of the functional groups, which are predominate peaks at 2945 cm^{-1} and 2841 cm^{-1} can be attributed to the (C-H) stretching and the characteristic (C=O) stretching occurs at 1716 cm^{-1} and the stretching (C=C) occurs at 1637 cm^{-1} , for (CH₂- and CH₃) occurs at 1454 cm^{-1} , groups of peaks between 1296 cm^{-1} and 1163 cm^{-1} can be attributed to (C-O-C) stretching as in figure (3.1).



Wave number (cm⁻¹)

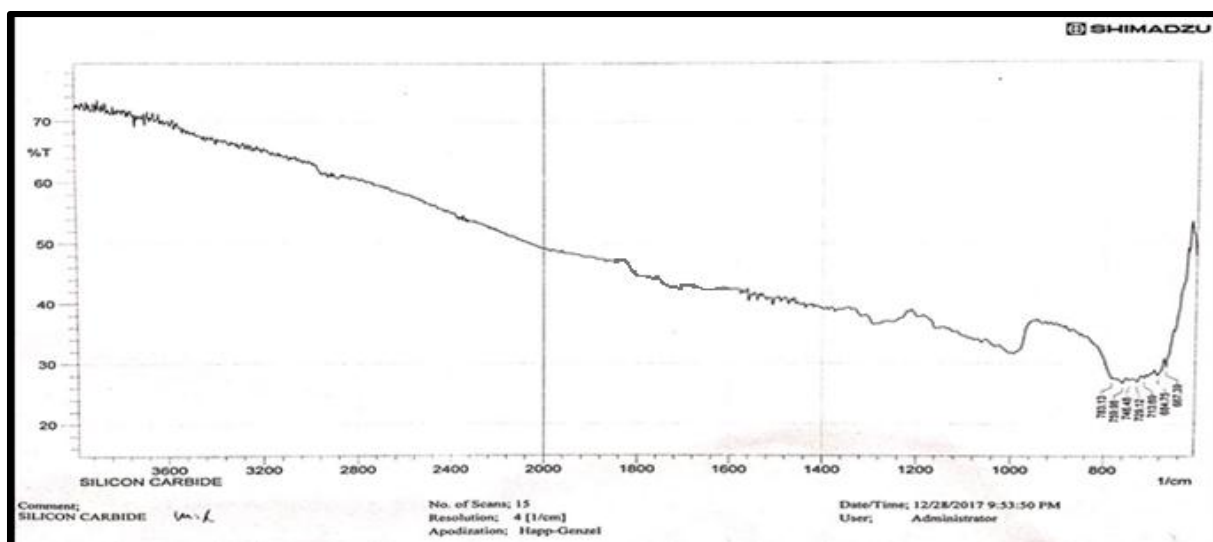
Figure (3.1) FTIR spectrum of Tri-methacryloxypropyltrimethoxysilane (MPS)

The FTIR result of SiC after silanization in figure (3.3) shows the same absorption peaks of SiC before silanization as seen in figure (3.2) and there is no new peaks appeared, so there is no chemical bond between the silane coupling agent and the nanoparticles SiC.



Wave number (cm^{-1})

Figure (3.2) FTIR spectrum of silicon carbide nanoparticles before silanization



Wave number (cm^{-1})

Figure (3.3) FTIR spectrum of silicon carbide nanoparticles after silanization

3.2 Microscopic test

Scanning electron microscope was made for control specimen and experimental specimen with SiC 0.25% wt. at 2000X magnification as in figures (3.4) and (3.5).

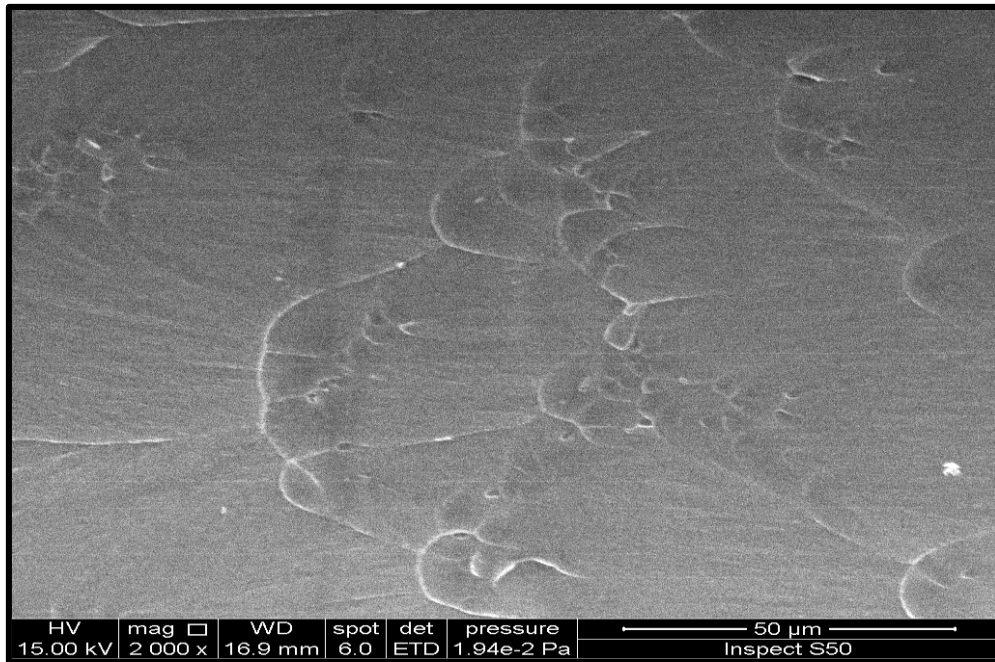


Figure (3.4) Scanning electron microscope of control specimen

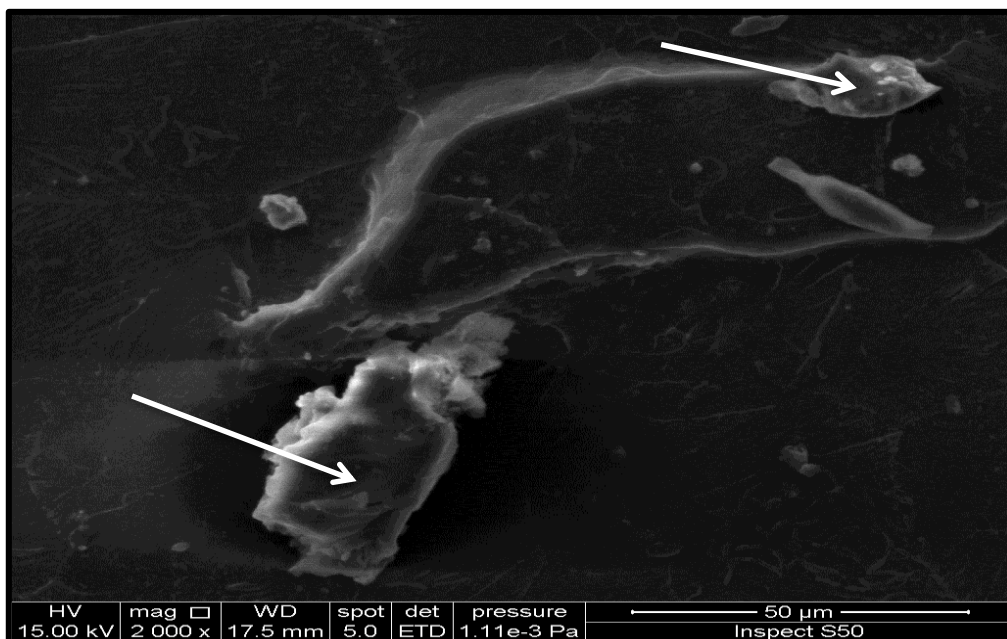


Figure (3.5) Scanning electron microscope of experimental specimen with SiC nanoparticles aggregation (white arrows)

As appear in the figure (3.5) the nanoparticles are not uniformly distributed throughout the specimen and they aggregate in groups within the polymer matrix (white arrows).

3.3 Energy Dispersive X-ray Spectroscopy (EDS)

EDS diagrams for heat cure acrylic resin before the addition and after the addition of 0.25%wt silicon carbide nano powder are shown in figures (3.6 and 3.7) respectively, the difference between the two diagrams indicate the incorporation of silicon carbide nano powder in the polymeric matrix of acrylic.

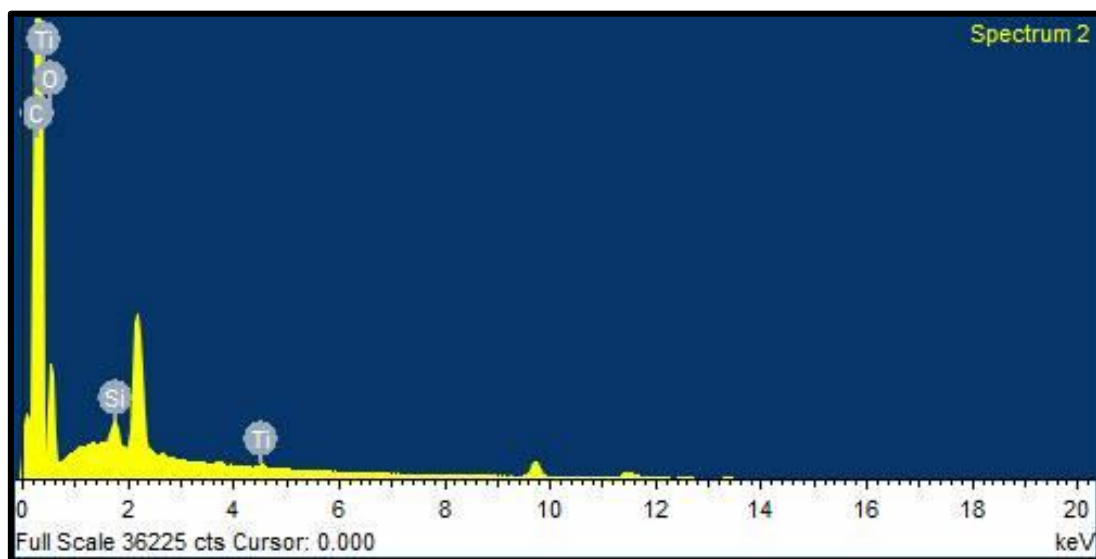


Figure (3.6) EDS diagram before the addition of silanated SiC nano powder

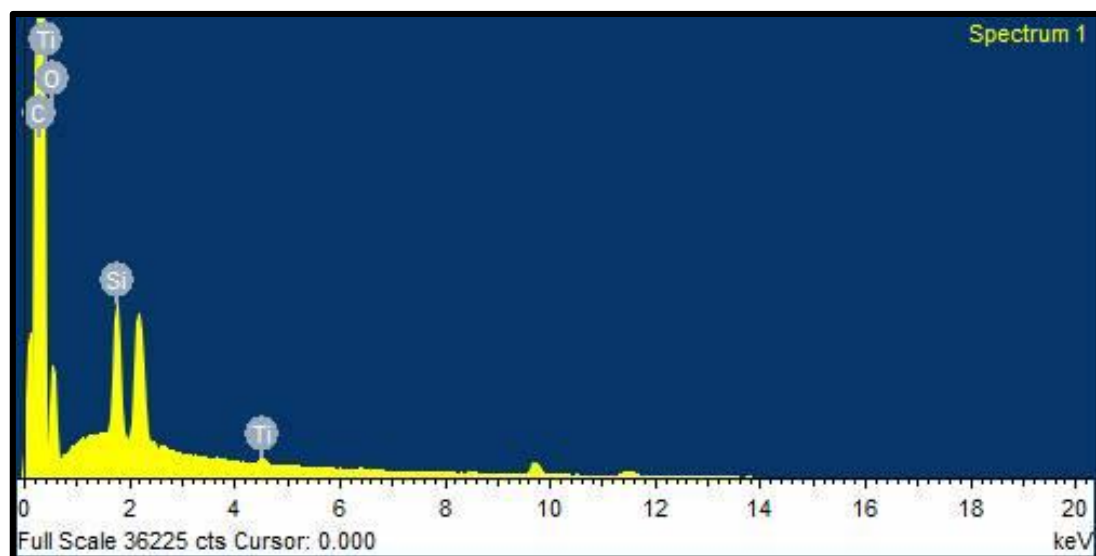


Figure (3.7) EDS diagram after the addition of silanated SiC nano powder

3.4 Thermal conductivity test

The mean, standard deviation, standard error, minimum and maximum values of experimental specimens measuring thermal conductivity test results are presented in table (3.1). Figure (3.8) represent the mean values of the thermal conductivity result.

The highest mean value of thermal conductivity appear in 0.25% wt. SiC group (0.972 W/m.K), while the lowest mean value was in control group.

Table (3.1): Descriptive statistics of thermal conductivity (W/m.K) among groups.

Studied group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	.622	.081	.026	.516	.746
0.125%	10	.819	.073	.023	.722	.959
0.25%	10	.972	.067	.021	.865	1.082

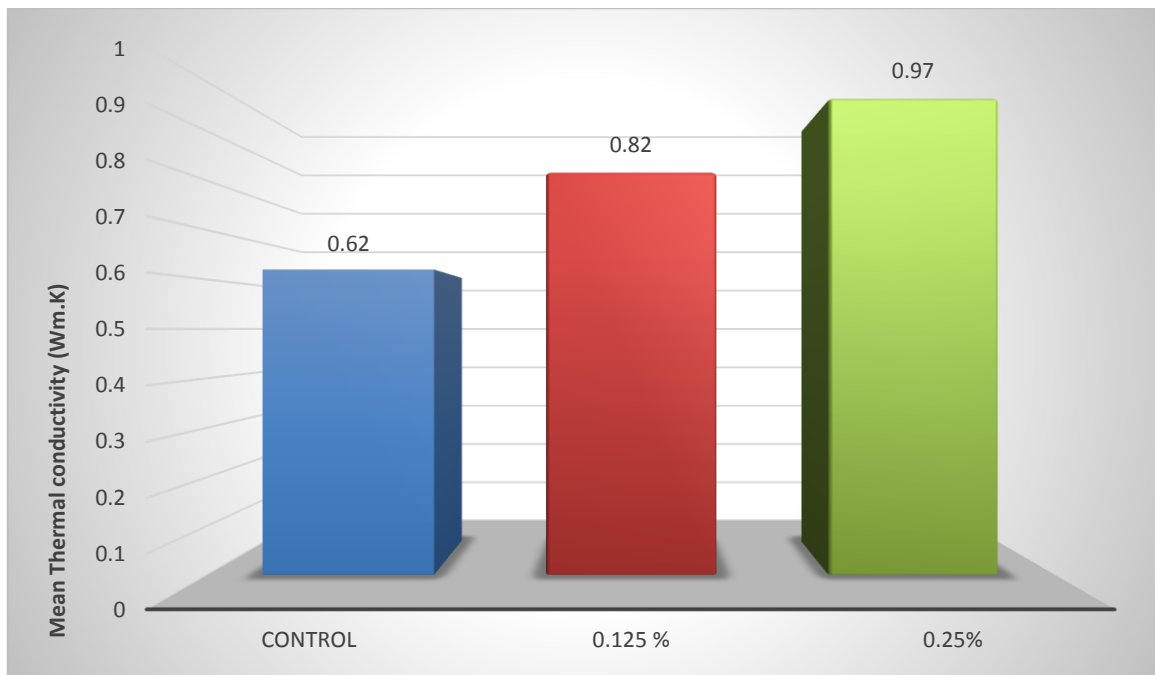


Figure (3.8) Bar chart of mean values of thermal conductivity among studied groups

One way (ANOVA) suggested a highly significant statistical difference among groups as in table (3.2).

Table (3.2): One way ANOVA for thermal conductivity among groups.

	Sum of Squares	df	Mean Square	F	Sig.	Effect size
Between groups	.615	2	.308	56.457	.000 (HS)	0.898
Within groups	.147	27	.005			
Total	.762	29				

The source of difference was more investigated by further complement analysis of data, **Tukey HSD** (Honestly significant difference) as in table (3.3). The table show highly significant difference among test groups.

Table (3.3) Multiple comparisons of thermal conductivity between groups using Tukey HSD

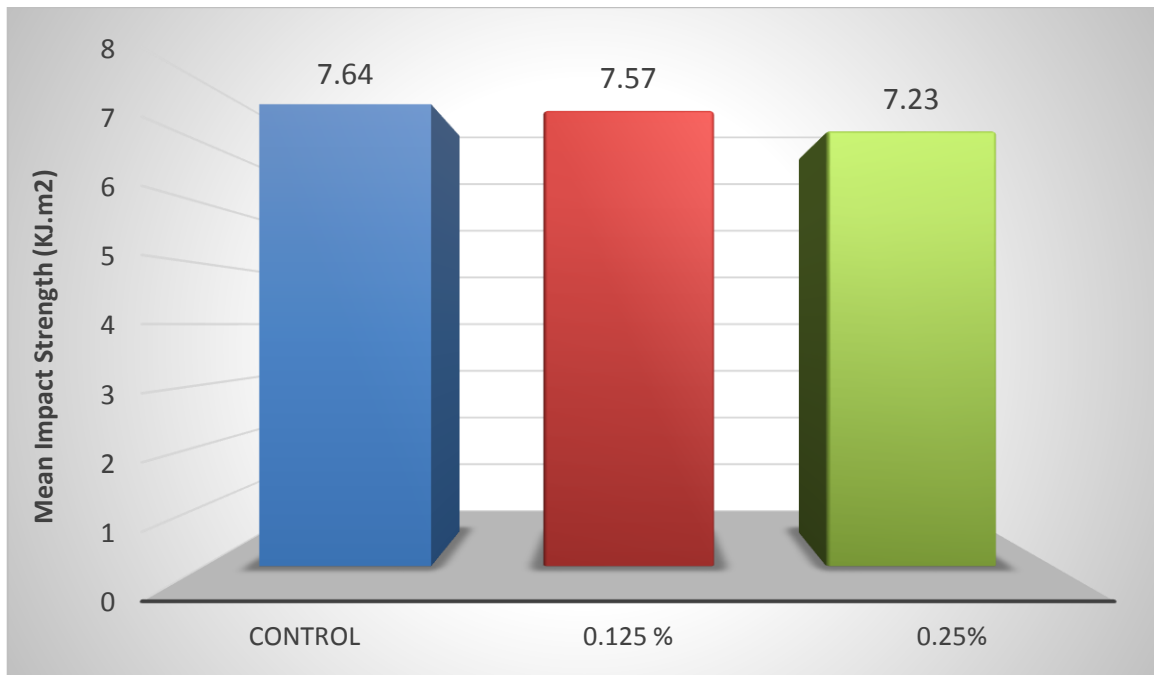
Multiple Comparisons of thermal conductivity (W/m.K) between groups using Tukey HSD					
(I) groups	(J) groups	Mean Difference (I-J)	Sig.	95% Confidence Interval	
				Lower Bound	Upper Bound
control	0.125%	-.196	.000	-.2782	-.1146
	0.25%	-.350	.000	-.4317	-.2681
0.125%	0.25%	-.154	.000	-.2353	-.0717

3.5 Impact strength test

Mean values, standard deviation, standard error, minimum and maximum of impact strength test results are presented in table (3.4). Figure (3.9) represent mean values of impact strength results. The highest mean value of impact strength is related to control group, while the mean value of 0.25%wt SiC nanoparticle group is (7.234 KJ/m²) which is the lowest mean value.

Table (3.4): Descriptive statistics of impact strength (KJ/m²) among groups.

Studied group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	7.644	.713	.225	6.730	8.790
0.125%	10	7.572	.888	.281	6.140	9.210
0.25%	10	7.234	.853	.270	5.420	8.120

**Figure (3.9) Bar chart of mean values of impact strength test among studied groups**

One way (ANOVA) test revealed a non significant difference among studied groups as in table (3.5)

Table (3.5): One way ANOVA for impact strength among groups.

	Sum of Squares	df	Mean Square	F	Sig.	Effect size
Between groups	.958	2	.479	.710	.501 (NS)	0.224
Within groups	18.223	27	.675			
Total	19.181	29				

3.6 Transverse strength test

Descriptive statistics of transverse strength are presented in table (3.6). Figure (3.10) represent the mean values of transverse strength result.

The highest mean value is present in control group, while the lowest mean value is for 0.25% wt SiC group (85.790 N/mm²).

Table (3.6): Descriptive statistics of Transverse strength MPa (N/mm²) among groups.

Studied group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	87.840	1.366	.432	85.500	89.500
0.125%	10	86.980	2.427	.767	82.700	89.900
0.25%	10	85.790	2.745	.868	81.300	89.500

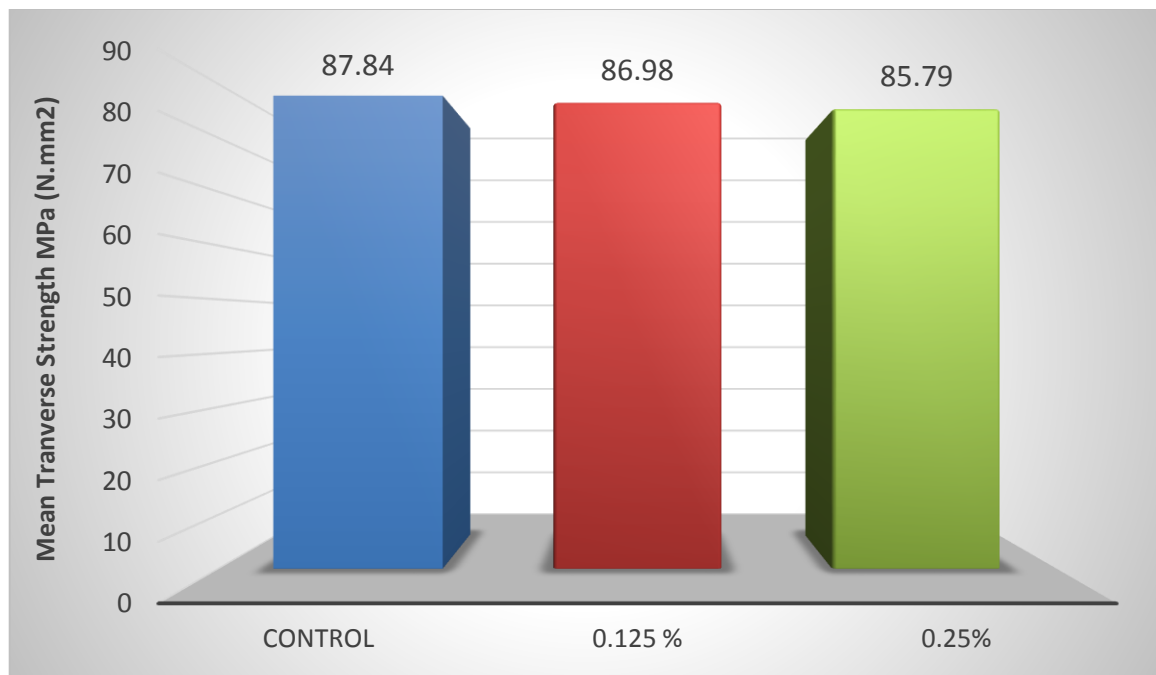


Figure (3.10) Bar chart of mean values of transverse strength test result among the studied groups

One way (ANOVA) revealed a non significant difference among groups as in table (3.7).

Table (3.7): One way ANOVA for transverse strength among groups.

	Sum of Squares	df	Mean Square	F	Sig.	Effect size
Between groups	21.194	2	10.597	2.079	.145 (NS)	0.365
Within groups	137.609	27	5.097			
Total	158.803	29				

3.7 Surface hardness test

Descriptive statistics of surface hardness test are shown in table (3.8). Figure (3.11) represent mean values of the study groups.

The highest mean value is (89.340) for 0.25%wt. SiC group, while the lowest value for control group.

Table (3.8): Descriptive statistics of Hardness (IU) among groups.

Studied group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	84.720	1.883	.595	82.200	87.400
0.125%	10	87.400	2.548	.806	83.300	90.900
0.25%	10	89.340	2.196	.694	85.500	93.000

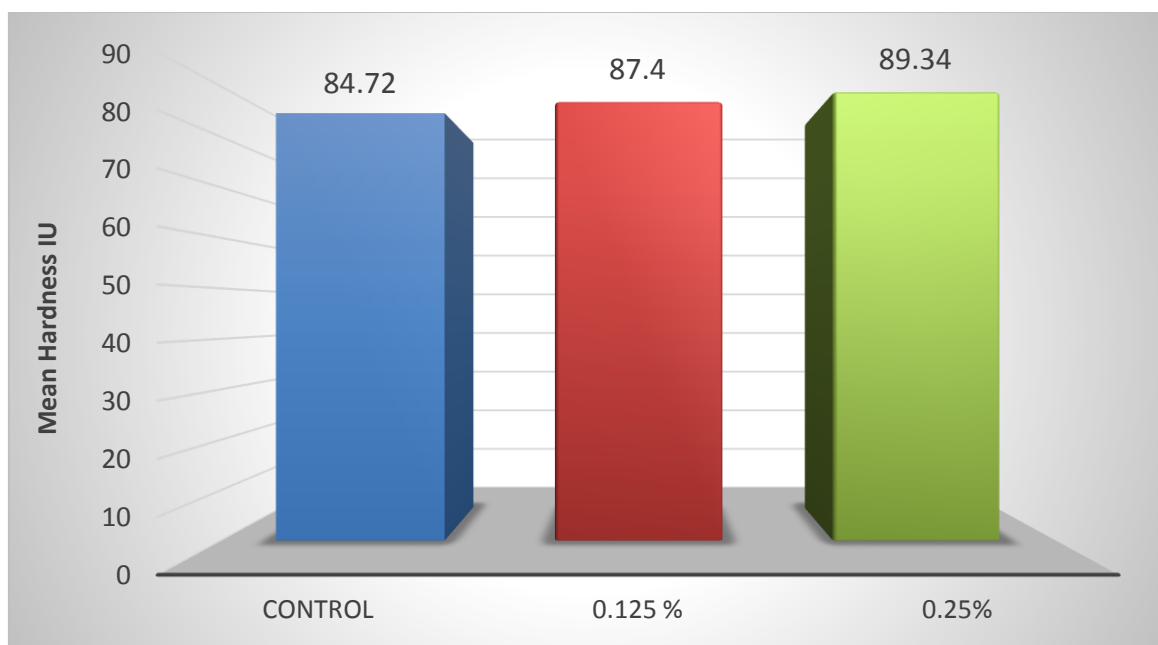


Figure (3.11) Bar chart of mean values of hardness test among groups

One way (ANOVA) test revealed a highly significant difference among the studied groups as in table (3.9)

Table (3.9): One way ANOVA for surface hardness among groups.

	Sum of Squares	df	Mean Square	F	Sig.	Effect size
Between groups	107.635	2	53.817	10.866	.000 (HS)	0.668
Within groups	133.720	27	4.953			
Total	241.355	29				

The source of difference was more investigated by analysis of data , Tukey HSD as in table (3.10). In the table the test results showed a highly significant difference among test groups.

Table (3.10) Multiple comparisons of hardness among groups using Tukey HSD

Multiple Comparisons of Hardness (IU) between groups using Tukey HSD					
(I) groups	(J) groups	Mean Difference (I-J)	Sig.	95% Confidence Interval	
				Lower Bound	Upper Bound
control	0.125%	-2.680	.031	-5.148	-.212
	0.25%	-4.620	.000	-7.088	-2.152
0.125%	0.25%	-1.940	.145	-4.408	.528

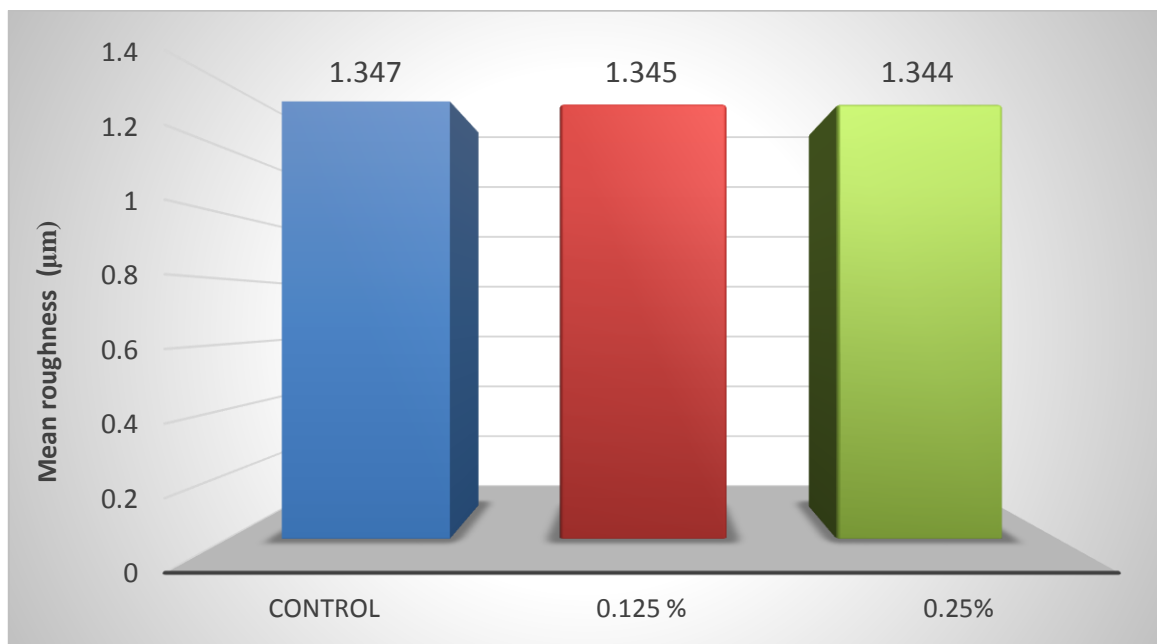
3.8 Surface roughness test

The means, standard deviation, standard error, minimum and maximum of the surface roughness test result are present in table (3.11). Figure (3.12) represent the mean values of the surface roughness result.

The control group showed the highest mean value, while 0.25% wt. nano SiC showed the lowest mean value (1.344 μm).

Table (3.11): Descriptive statistics of Roughness (μm) among groups.

Studied group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	1.347	.303	.096	1.010	1.810
0.125%	10	1.345	.241	.076	1.001	1.611
0.25%	10	1.344	.223	.071	1.077	1.658

**Figure (3.12) Bar chart of surface roughness test among studied groups**

One way (ANOVA) test revealed a non significant difference among studied groups as in table (3.12).

Table (3.12): One way ANOVA for surface roughness among groups.

	Sum of Squares	df	Mean Square	F	Sig.	Effect size
Between groups	.000	2	.000	.000	1.000 (NS)	0.005
Within groups	1.799	27	.067			
Total	1.799	29				



Chapter Four

Discussion

Discussion

PMMA is the material of choice for denture base fabrication. It was introduced by *Dr. Walter Wright* in 1937 and it continues to be used because of its favorable working characteristics, ease of processing, accurate fit, stability in oral environment, inexpensive equipment and superior esthetic. Despite of these good properties, there is need to improve some properties such as poor thermal properties and inferior mechanical properties of PMMA (**John *et al.*, 2001**).

One approach to resolve this problem was to incorporate some types of reinforcements into the denture base polymer. The concept of combining nanoparticles within a matrix of material in new composite reinforcement has been shown to be very successful (**Leszczyn'ska A *et al.*, 2007**).

In the present study some properties of acrylic denture base material were evaluated after the addition SiC nanoparticles to improve some properties with minimum effect on others.

The exceptional properties of SiC are the main reasons behind the research and development of this material and it is the stable form of silicon and carbon. The SiC has remarkable thermal properties (SiC has thermal conductivity comparable to copper and silver) also it has high resistance to harsh environment like high temperature, acidic and basic media and oxidizing media. SiC has exceptional biocompatibility and hemocompatibility, thus it is suitable for application inside the human body. These properties attributed to the nature of Si-C bond (**Ponraj, 2016**).

4.1 Thermal conductivity

One of the important thermal properties of dental materials is thermal conductivity or ability to transmit heat which obtained from determining the rate at which heat can be transmitted through a given cross sectional area of the specimens of material during a given time interval (**Powers and Sakaguchi, 2012**).

PMMA also has low thermal conductivity compared with gold or cobalt alloy denture base materials and this can present problems during denture processing as the heat produced can not escape , leading to a temperature rise and this may lead to porosity during fabrication. From the patient's point of view, the problem with low thermal conductivity is that in edentulous patients wearing full dentures, the palate is covered by the denture base; consequently, the ability to sense transient temperature change at the palate may be affected by the thermal characteristics of the denture base material. Also thermal characteristics of denture base can become important factors affecting the gustatory response (**Abdulhamed & mohammed, 2010**).

Tables (3.1) and (3.2) and figure (3.8) showed that there was a highly significant increase in the values of thermal conductivity with the addition of silicon carbide nanoparticles. This may be due to that the particles gradually contacting with each other to form chain like or network like structure which is called heat conductive pathways and these pathways permit transition of heat from one side of the specimen to another side and bridge the insulating effect of polymer. Thus, the polymer will exhibit high thermal conductivity. The higher the filler loading, the more significant heat conductive pathways resulting in enhancement of heat conductive performance of the material (**Yu *et al.*, 2018**).

4.2 Impact strength

The amount of power needed by unit area of material when it is damaged by sudden strike like falling denture from hand is the impact strength so the denture must have enough impact strength (**Craig and power 2002**).

The outcome of the study revealed the highest impact strength value for control group as compared with 0.125%wt. and 0.25% wt. SiC as shown in tables (3.4) & (3.5) and figure (3.9). Among the studied groups there is non significant decrease in impact strength compared to control group.

The reason behind this decrease could be the nanoparticles agglomeration, nanoparticles have high surface energy and tend to stick together and agglomerate (**Balkees *et al.*, 2018**).

The agglomeration may also be due to difficulty in dispersion of nanofillers in the matrix of the polymer by conventional techniques (using probe sonication apparatus) to be homogenous causing them to agglomerate due to affinity of nanoparticles to aggregate (as shown in SEM figures 3.4 and 3.5), with increase in nanoparticles addition (as with 0.25% wt. nanoparticles) the impact strength decreased because at higher concentrations there will be more improper distribution within the acrylic matrix (**Salman *et al.*, 2017**), and this explains why the impact strength of (0.25% wt. nanoparticles) is less than that of (0.125% wt. nanoparticles) group.

Another possible cause is that in the PMMA containing nanoparticles, the bonding at the interface between the inorganic SiC nanoparticles and organic polymer matrix is poor. When the force is placed, stress will be concentrated at the interface between the filler and the matrix. The stress cannot be effectively transferred; therefore, it will cause cracks and finally induce material failure, and the PMMA matrix revealed brittle behavior. In such material the crack propagated rapidly and lead to specimen fracture, this indicate a weak

resistance to crack propagation and minimal energy was required to fracture the specimen (**Jiangkongkho *et al.*, 2018**).

In this study the addition of SiC nanoparticles to PMMA at concentrations of 0.125% wt. and 0.25% wt. have a non significant effect on the impact strength of PMMA.

4.3 Transverse strength

The transverse strength test, one of the mechanical strength tests, is especially useful in comparing denture base materials because it represents the stress which applied to the denture during mastication (**Craig and power, 2002**). The transverse (flexural) strength is a combination of compressive, tensile and shear strengths, all of which directly reflect the resistance of a material to fracture (**Anusavice,2002; Jagger *et al.*,2002**).

This test simulates the flexural loading that an upper denture may receive during mastication. The evaluation of acrylic denture base by a transverse bending test was based on a three point loading system, since it reflects the loading arrangement in the clinical situation (**Dhuru, 2004**).

As shown in tables (3.6) and (3.7) and figure (3.10) there was a non significant decrease in the transverse strength with addition of SiC nanoparticles as compared with control group. The decrease in the flexural strength depends on particle distribution which results in decrease in flexural strength, also the voids content were found to be higher due to of agglomeration of nano-filler at higher concentrations lead to their improper distribution in the matrix (**Kumar *et al.*, 2018**) as shown in SEM figures (3.4) and (3.5). Another possible cause of decreased flexural strength is that the nanoparticles may act as impurities of polymerization that causes decrease the degree of polymerization ,thus more unreacted monomer will be left over within the matrix that result in decrease in the flexural strength (**Balkees *et al.*, 2018**).

With increasing the amount of SiC nanoparticles, the distance between particles will decreased which allow for increased chances of nanoparticles agglomeration. This agglomeration generates stress concentration at the agglomerated particles which decrease the mechanical properties (**Jiangkongkho *et al.*, 2018**), that explain the decrease in flexural strength at higher concentrations (0.25%) wt. as compared with lower concentrations of nanoparticles (0.125% wt.).

This decrease is not significant from statistical point of view, therefore; it will not affect the function of PMMA-SiC composite produced.

4.4 Surface hardness

Power and Sakaguchi in 2012 defined the surface hardness as resistance to surface penetration or indentation. In this study Shore D hardness test is used which is more appropriate for acrylic resin material (**Unlane and Dikbas in 2007**).

As shown in tables (3.8) and (3.9) and figure (3.11), the result of hardness value showed highly significant increase with addition of silicon carbide nanoparticles compared with control group. The increase in hardness is direct proportional with increase in silicon carbide content.

The polymer reinforced with SiC showed higher surface hardness because with the addition of SiC nanoparticles, the distance between the particles inside the matrix will be decreased. This decrease in the inter particle distance increases the bonding strength between the particles and cluster together which result in surface accumulation of hard material particles SiC in the acrylic matrix spaces which result in improved behavior in term of hardness (**Antil *et al.*, 2017**).

The increase in hardness is concentration dependent that means increase was slight with low concentration of nanoparticles (as in 0.125 %wt.

nanoparticles) due to low network density, while highly increase in hardness of the nanocomposite with higher concentrations (as in 0.25% wt. nanoparticles) was attributed to increase in the accumulation of particles of hard material into the acrylic resin matrix (**Hudson *et al.*, 2000**).

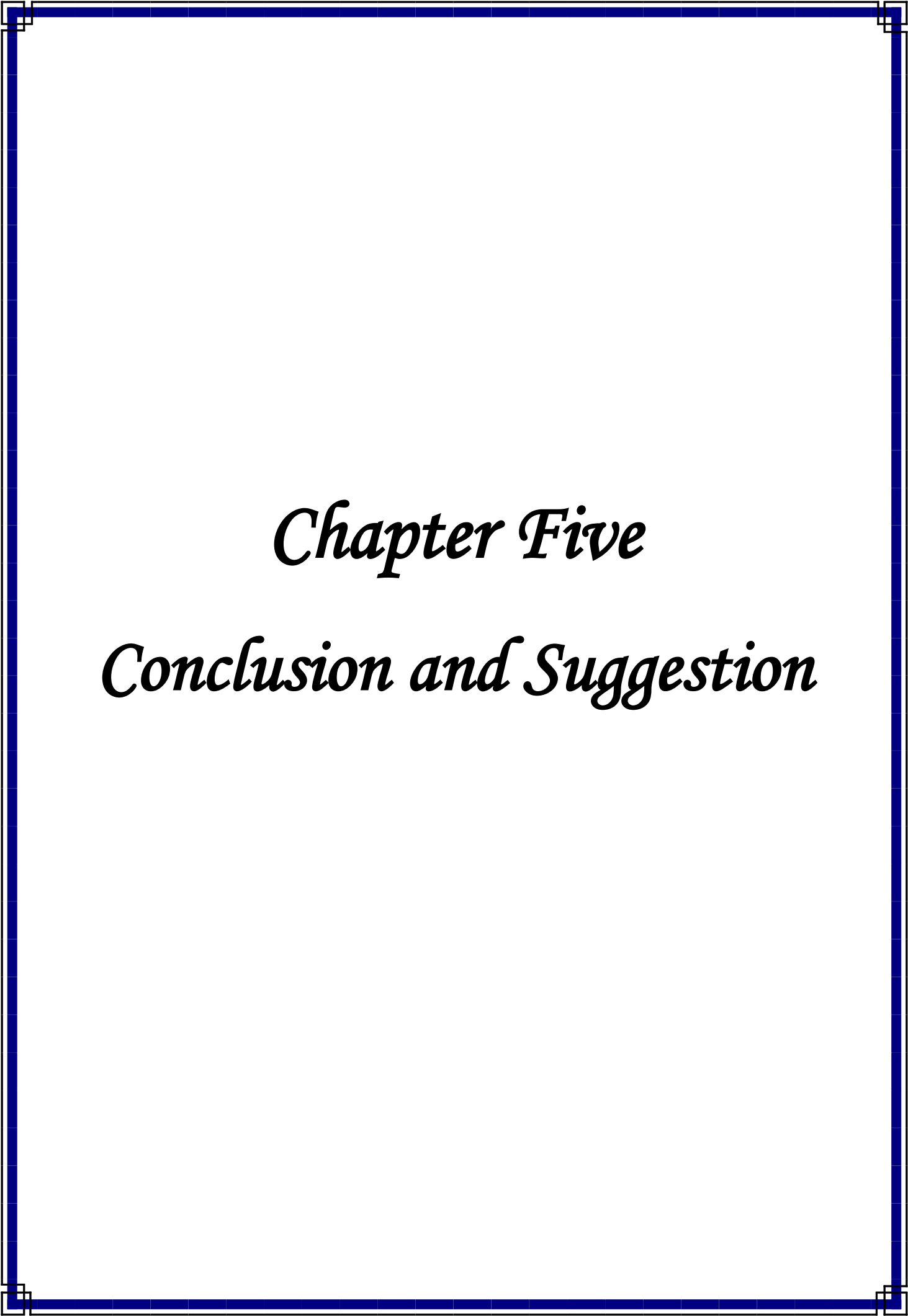
4.5 Surface roughness

Achieving a smooth surface with extremely fine or no surface scratches has always been a prime objective for resin restoration ,since the increase in surface roughness had detrimental effect on the aesthetic of the denture; the smooth shiny surface of the acrylic resin help to resist the buildup of stain, debris and plaque (**Harrison *et al.*, 2004**).

Surface roughness of the denture base is very important, as it affects the health of the tissue in direct contact with the denture .Most microorganisms present intraorally, particularly those responsible for caries, periodontal disease and denture stomatitis, can only survive in the mouth if they adhere to a non-shedding oral surface and begin to form colonies (**Morgan and Wilson, 2001**).

Tables (3.11) and (3.12) and figure (3.12) showed non-significant change with different percentages of added SiC nanoparticles and this may be due to that the silicon carbide nanoparticles have very small size, also surface roughness test is concerned with the outer surface and not the inner surface of the nanocomposite so when small percentage of nanoparticles when added to the acrylic resin only few particles were involved on the surface of the specimens (**Al momen, 2000**), so that when small percentage of nanoparticles added to the acrylic resin it will have a very small effect on the surface roughness.

Therefore; the SiC nanoparticles will not significantly affect on surface roughness when compared to control group.



Chapter Five

Conclusion and Suggestion

Conclusions & Suggestions

5.1 Conclusions

Within the limitation of the study, the following conclusions were reached:

1. The addition of SiC nanoparticles into heat cured acrylic denture base resin increased the thermal conductivity property and it is concentration dependant.
2. Addition of silanated SiC nanoparticles to heat cured acrylic denture base material improved the surface hardness and the improvement is concentration dependent.
3. There is little reduction in surface roughness of acrylic denture base reinforced by silanated SiC nanoparticles.
4. The impact strength and transverse strength with addition of SiC to heat cured acrylic denture base were not significantly decreased.

5.2 Suggestions

On the basis of this study, this work can be extended to include:

1. Evaluation the effect of silicon carbide along with any type of fibers (like poly ethylene fibers) on different properties of PMMA .
2. Investigate the effect of adding silanated nano SiC to chemical cure or hybrid acrylic resin.
3. Studying the effect of adding silicon carbide nanofillers on other properties of acrylic resin such as dimentional stability, adabtibility, water solubility, water sorption raioopacity and color stability.
4. Studying the effect of SiC nanoparticles on the mechanical properties of soft liners.

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Appendices

Appendices

**Appendix I data represent the values of Thermal conductivity (W/m.K)
for each concentration**

	Control	0.125%	0.25%
1	0.542	0.734	0.933
2	0.516	0.749	0.964
3	0.654	0.822	0.996
4	0.580	0.785	0.929
5	0.739	0.880	1.082
6	0.644	0.959	1.076
7	0.655	0.850	0.865
8	0.746	0.722	0.934
9	0.612	0.831	0.961
10	0.536	0.856	0.983

**Appendix II data represent the values of Impact strength (KJ/m²) for
each concentration**

	Control	0.125%	0.25%
1	6.73	7.64	7.44
2	8.72	8.28	5.42
3	6.82	6.14	6.16
4	7.34	7.22	7.13
5	8.79	9.21	8.06
6	7.78	7.89	8.12
7	7.18	6.60	7.52
8	7.33	7.15	7.24
9	7.71	8.25	7.94
10	8.04	7.34	7.31

**Appendix III data represent the values of Transverse strength (N/mm²)
for each concentration**

	Control	0.125%	0.25%
1	89.2	88.1	88.7
2	86.6	88.5	87.1
3	88.2	87.3	85.6
4	86.9	86.7	89.5
5	89.1	88.4	86.7
6	85.5	89.9	84.3
7	89.5	82.7	82.1
8	88.3	85.5	84.5
9	88.6	83.4	81.3
10	86.5	89.3	88.1

**Appendix IV data represent the values of Surface hardnessfor each
concentration**

	Control	0.125%	0.25%
1	85.2	84.9	91.1
2	86	90.9	91.3
3	83.5	83.3	89.5
4	85.7	88.8	93
5	87.3	88.6	87.7
6	82.6	84.1	85.5
7	82.2	89.1	90.3
8	87.4	89.8	88.7
9	84.2	87	89
10	83.1	87.5	87.3

Appendix V data represent values of Surface roughness (μm) for each concentration

	Control	0.125%	0.25%
1	1.241	1.001	1.077
2	1.729	1.271	1.554
3	1.010	1.278	1.568
4	1.045	1.520	1.498
5	1.470	1.586	1.173
6	1.645	1.014	1.658
7	1.025	1.585	1.216
8	1.142	1.611	1.162
9	1.353	1.472	1.454
10	1.810	1.114	1.080

الخلاصة

بيان المشكلة:

الراتنج الاكريلي (PMMA) هي المادة الأكثر استخداما في تصنيع اطعم الاسنان. المادة بعيدة عن المثالية في تلبية المتطلبات ، مثل انخفاض التوصيل الحراري و صلادة سطح غير كافية و زيادة في خشونة السطح .

الهدف من الدراسة:

الغرض من هذه الدراسة لتقييم تأثير اضافة الحبيبات النانوية لمسحوق كاربيد السيليكون SiC بتركيز (0.125% بالوزن و 0.25% بالوزن) على بعض الخواص الفيزيائية والميكانيكية مادة الراتنج الاكريلي الحراري.

المواد والطرق:

تم تحضير مائة وخمسين عينة (150) ثم قسمت الى خمسة مجاميع وفقا للاختبارات التي اجريت وكل مجموعة تتكون من (30) عينة وهذه المجموعة قسمت الى ثلاث مجاميع اخرى كالآتي : (مجموعة السيطرة) ومجموعة الراتنج الاكريلي المقواة (0.125% بالوزن من مادة كاربيد السيليكون النانوية) ومجموعة الراتنج الاكريلي المقواة (0.25% بالوزن من مادة كاربيد السيليكون النانوية). تم فحص تجارب التوصيل الحراري وقوة الصدمة والقوة المستعرضة و صلادة السطح و خشونة السطح تم بحثها.

النتائج:

نتائج هذه الدراسة اظهرت ان عند التركيز 0.125% بالوزن من مادة كاربيد السيليكون النانوية ادى الى زيادة ملحوظة بكلا التوصيل الحراري و صلادة السطح بينما هنالك انخفاض غير ملحوظ بالقوة المستعرضة وقوة الصدمة و خشونة السطح مايكرون عند اضافتها لمادة قاعدة الطقم الراتنج الاكريلي.

اظهر التركيز 0.25% بالوزن من مادة كاربيد السيليكون النانوية زيادة ملحوظة في نتائج التوصيل الحراري و صلادة السطح بينما هنالك انخفاض غير ملحوظ بالقوة المستعرضة وقوة الصدمة و خشونة السطح عند اضافتها لمادة قاعدة الطقم الراتنج الاكريلي.

الاستنتاج:

اضافة مسحوق كاربيد السيليكون النانوي الى مادة قاعدة الطقم الاكريليك يحسن التوصيل الحراري وصلادة السطح وخشونة السطح بينما هنالك انخفاض غير ملحوظ في نتائج القوة المستعرضة وقوة الصدمة.



جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة بغداد
كلية طب الأسنان

تأثير اضافة حبيبات كاربيد السيليكون النانوية على بعض الخواص الفيزيائية والميكانيكية لمادة قاعدة طقم الراتنج الاكريلي الحراري

رسالة مقدمة الى مجلس كلية طب الاسنان- جامعة بغداد
كجزء من متطلبات نيل درجة الماجستير في صناعة الاسنان

قدمت من قبل

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