



**EFFECT OF INSERTION TECHNIQUES
ON
THE SHEAR BOND STRENGTH OF
LOW-SHRINKAGE (Silorane)
COMPOSITE RE-STORATIVE
MATERIALS**

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**This thesis was submitted in partial fulfilment of the
requirement for Master's Degree in dental materials**

**University of Benghazi
Faculty of Dentistry**

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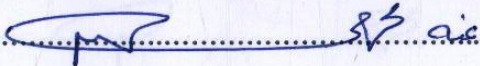
Department of Prosthodontics-Dental Materials

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Dedication

I dedicate this work to : My father's spirit, who supported me at the start of this work, hoping he would be proud of me, my husband, who finished what my father started, my mother for all her sacrifice and love, and my sisters for their encouragement and support.

Asma Abdelmawla

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Asma Abdelmawla

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List of abbreviations and symbol

Bis-GMA : Bisphenol Glycidyle Metha acrylate.

UDMA : Urethan Di Metha acrylate.

TEGDMA : Tri Ethylene Glycol Di Metha acrylate.

MMA : Methyl Metha acrylate.

EDMA : Ethylene Di Metha acrylate.

MPS : γ -Metha cryloxy Propyl trimethoxy Silane.

BHT : Butylated Hydroxy Toluene.

DC : Degree of Conversion.

OIL : Oxygen Inhibition Layer.

RBCs : Resin Based Composites.

MBCs : Methacrylate Based Composite.

SBS : Shear Bond Strength.

TEC : Tetric EvoCeram.

°C : Centigrade

MPa : Megapascal

nm : nanometer

Vol% : percentage content by volume

Wt% : percentage content by weight

Effect of insertion techniques on the shear bond strength of low-shrinkage (Silorane) composite restorative materials.

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Abstract

Objective: to evaluate the shear bond strength of low shrinkage (Silorane) based composite with different insertion techniques (layering and bulk) and storage conditions (wet, dry) and compare with conventional methacrylate composites.

Materials and methods: 108 specimens were fabricated by used cylindrical shaped mould for SBS test, prepared from three types of composite materials, low shrinkage (Filtek Silorane), and two MBCs resins, Tetric EvoCeram, and Filtek Supreme resin composites, subdivided to 12 groups according to insertion technique (layering, bulk) and the storage condition (wet, dry at $\pm 37^{\circ}\text{C}$ for one week) ($n=9/\text{groups}$). The SBS were measured with a crosshead speed of 0.5 mm/min. Ultimate shear strength data (MPa) from all tested groups were submitted to analysis of variance (one-way ANOVA) and the Tukey test ($p = 0.05$). The fractured surfaces of the test samples were visually evaluated by a light microscope at 20 and 40 times magnification. Fractures were classified as either adhesive or cohesive or mixed.

Results: Filtek Supreme (MBCs) specimens inserted in bulk technique and stored dry showed the highest mean SBS (36.45 MPa) values. Silorane and Tetric Ceram (MBCs) were lower in their all groups (26.45, 26.78, 27.50 MPa for Silorane, 27.51, 27.02, 27.29, 28.21 MPa for Tetric Ceram resin specimens) except for Silorane specimens inserted in layers which was further decreased by wet storage (21.90 MPa), this was slightly similar to the result of Filtek Supreme specimens inserted in layers and stored dry, which showed the lowest (21.35 MPa) SBS among the groups.

Conclusion: insertion technique type and storage condition didn't significantly affect SBS of Silorane resin in compare with methacrylate resin, except for the specimens inserted in layers and wet condition, there was a significant decrease in SBS values. This finding support the clinical reliability of insertion the Silorane composite in bulk with a cavity deeper than 2 mm depth.

Chapter I

Introduction.

Introduction:

Composites are essentially made of three basic components: resin based organic matrix, organic filler particles or organic dispersed phases and an organic-inorganic bonding agent, a Silane coupling agent. Organic matrix is made of monomers that converted into polymers and form cross-linked three-dimensional network.^(1, 2)

For more than 50 years, Dimetacrylate-based dental composite resin has been used in dentistry offering a significant number of benefits and advantages. During this time, many developments in filler technology and initiation systems have considerably improved composite physical properties and expanded their clinical applications. However, the polymerization shrinkage resulting from the conversion of Dimetacrylate monomers into long cross-linked polymeric chains (free radical polymerization), remain a major drawback.^(1,3) Polymerization shrinkage and the related polymerization stress, both contributed different challenges such as reduced marginal integrity and post-operative sensitivity.^(4,5) Imperfect margins result in marginal staining and eventually secondary cavities, and represent the most important reason for the replacement of existing insufficient composite fillings.⁽⁶⁾

There are many strategies and approaches that have been proposed to reduce the magnitude and effect of polymerization shrinkage and the related polymerization stress. Moreover, some of them intend to attain this goal by using different strategies from clinical techniques by changing the insertion technique of resin composite in dental cavities, or by changing to filler technology and light cure methods.⁽⁷⁾ Others aimed at changing the matrix chemistry (the monomer matrix) of the resin composite. However, insertion techniques would be in the incremental technique, polymerization would be more uniform and efficient through the composite's entire thickness. This will

reduce the possibility of composite flow.⁽⁸⁾ In addition, increments applied in a layering technique depend on their bonding together in an oxygen inhibition layer formed on the surface of previous polymerized composite increment in atmosphere.^(8,9) Conversely, for cavities restored in bulk, a gradient of conversion due to light attenuation through the composite, would allow the composite to flow from the bottom of the restoration, which might result in internal gaps as the viscosity of material compromised adaptation to the cavity wall.⁽⁸⁾

Apart to the changes in the filler particles amount, shape, size or surface treatment, the main approaches adapted so far are to change the monomer structure and chemistry.⁽¹¹⁾

In the last decade, a new low-shrinkage Silorane-based resin composite material was developed from the reaction of oxirane and siloxane molecules. The reduced polymerization shrinkage property attributed to compensating the volumetric shrinkage by opening of the oxirane ring during polymerization (cationic polymerization). It is also aided by its highly hydrophobic nature, due to the presence of siloxane species.⁽⁶⁾

Previous studies confirmed “living” polymerization in the Silorane system,⁽¹⁰⁾ in which the degree of cure continued to increase even 20 minutes after photo initiation.⁽¹²⁾ This in return, raised the question of whether the insertion in bulk could be used.⁽¹³⁾ In a cationic polymerization reaction, the oxygen inhibition layer which is known to allow good interfacial bonding between increments of composite, is eliminated and reduced.⁽¹⁰⁾ Therefore, it is important to evaluate the composite-composite interfacial bonding strength properties of the Silorane-based resin as the material still has polymerization shrinkage to some extent. This is particularly true in the deep cavities where incremental technique is still the technique of choice.⁽¹⁴⁾

Chapter II

Background and Literature review

2.1. Brief history of tooth-coloured restorative material:

The first tooth-colored restorative material, silicate cement, was introduced to restore the anterior teeth in 1870. The major advantage of these cements were better tooth colour match, ⁽¹⁵⁾ and their anticariogenic property due to the fluoride content. On the other hand, the drawbacks of these cements are that they were brittle, stained quickly,⁽³⁾ underwent dissolution in oral fluid, lost their translucency, exhibited surface crazing, and lacked adequate mechanical properties.⁽¹⁶⁾

The development of the first polymeric tooth-colored acrylic resin in dentistry was in 1940. It was based on poly (methacrylate) powder, methyl methacrylate monomer (Figure1), benzoyl peroxide, and n,n- dimethylparatoluidine. Upon mixing, polymerization will occur, by which covalent bonds are formed between the molecules to form a large molecule and the continuous phase.⁽¹⁶⁾ This unfilled acrylic resin was used as an anterior restorative material because of its tooth-like appearance. However, this was for a short period of time due to a variety of problems associated with an increase in discoloration, lack of colour stability, recurrent tooth decay, and pulp reactions.⁽¹⁷⁾

These problems are mainly attributed to the polymerization shrinkage, a large coefficient of thermal expansion, and monomer leaching.^(2,3)The inherent drawbacks and the use limitation of the unfilled resin, led to the development of a filled acrylic resin by Dr Rafael (Bowen) in the 1960s.⁽¹⁾ Improvements in the formulation of the resin matrix (Bis-GMA) were achieved by adding the filler particles and coupling agents. The resulting composites have better mechanical properties and wear resistance. ⁽¹⁶⁾

In composite material technology, the term “composite” refers to a multiphase material formed from a combination of materials that differ in composition or form that remain bonded together yet retain their identities and properties.⁽¹⁷⁾

Composite, the universally used tooth-colored direct restorative material, was developed by combining Dimetacrylate (epoxy resin and methacrylic acid) with silanized quartz powder, the relative amounts and types of each of these components varies with each product and its clinical indication. Thanks to the components' properties (aesthetics, and advantages of adhesive technology), composites have taken the place that was occupied by amalgam.⁽¹⁸⁾

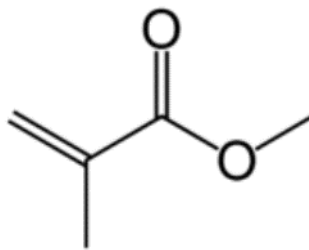


Figure 1: Structure of methyl methacrylate.⁽¹⁾

2.2. Composition of resin-based composite:

The resin based composite restorative materials that are used in dentistry have three major components:

- I. An organic resin matrix (continuous phase).
- II. An inorganic filler (dispersed phase).
- III. A coupling agent.⁽¹⁶⁾

Other components include:

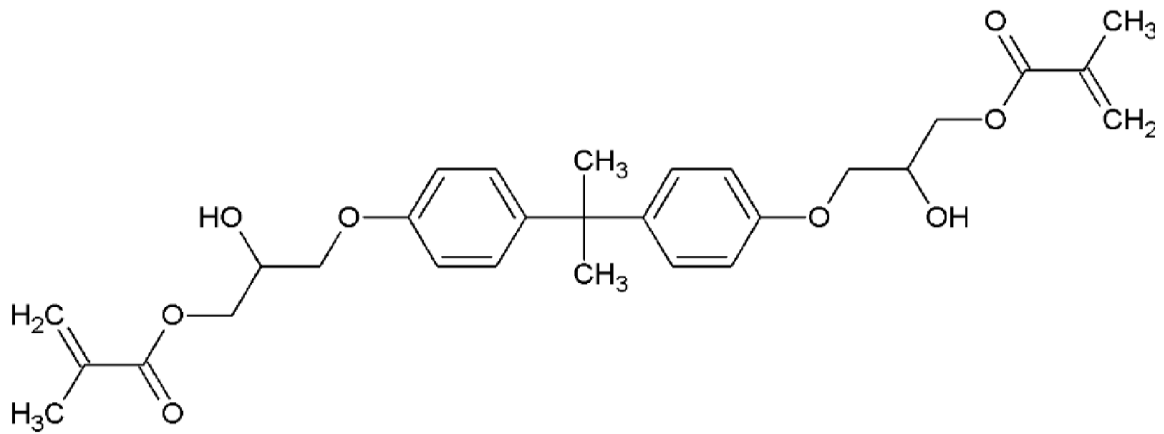
1. Initiator accelerator systems.
2. Inhibitors.
3. Stabilizer.
4. Pigment (Optical modifier).
5. Viscosity controllers such as :
 - a. TEGDMA (triethylene glycol dimetacrylate) (Figure 2).
 - b. MMA (methyl methacrylate)
 - c. EDMA (ethylene glycol dimetacrylate) (Figure 2).⁽¹⁷⁾

2.2.1. Organic resin matrix:

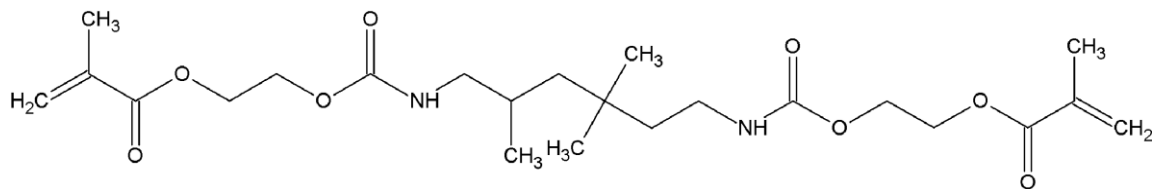
The organic matrix of composite resins is made up, in essence, of a system of mono-, di- or tri-functional monomers. In 1962, Bowen introduced the monomer bisphenol A-glycidyl methacrylate (Bis-GMA) (Figure 2) by attaching Methylmethacrylate groups to epoxy monomer. (Bis-GMA) has a relatively high polymerization rate and low hardening shrinkage. It has some drawbacks, however, such as its high viscosity because of its high molecular weight and lack of colour stability which gives it a tendency to become yellower.⁽¹⁹⁾

Other high molecular weight monomers have since been developed and introduced in commercial materials to overcome the limitations of Bis-GMA based systems. UDMA (Figure 2) formulations based on urethane dimethacrylate, have become increasingly common due to this monomer's low

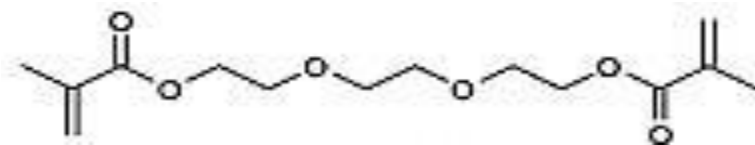
viscosity and high flexibility in relation to Bis-GM. UDMA copolymers, in general, present higher flexural strength, elastic modulus and hardness.⁽²¹⁾ Both Bis-GMA and UDMA have high molecular weight and are highly viscous. To facilitate the manufacturing process and clinical handling, it is diluted with other low-viscosity monomers (low molecular weight) which are considered viscosity controllers, such as triethylene glycol dimethacrylate (TEGDMA) (Figure 2), methyl methacrylate (MMA) or ethylene glycol dimethacrylate (EDMA).⁽²¹⁾



Structure of Bis-GMA



Structure of UDMA



Structure of TEGDMA

Figure 2: 3 Diagrams representing the chemical structure of monomers commonly used in resin-based composites.⁽²⁰⁾

2.2.2. Inorganic phase (filler):

The disperse phase of the resin composite is composed of an inorganic filler. This provides mechanical reinforcement to the mixture and allows light transmission and scattering that gives an enamel-like translucency to the material.⁽²²⁾ Filler loading has been shown to influence the strength, elastic modulus, wear resistance, water absorption, coefficient of thermal expansion and most importantly lower polymerization shrinkage of the material.⁽²³⁾ In addition, the size of the filler influences the polishability of the restoration.⁽²⁴⁾ Examples of the fillers used in dental composites are ground quartz, pyrolytic silica, aluminium silicate, lithium-aluminium silicates, borosilicate glasses and various other types of glass, including some containing oxides of heavy metals such as barium which render the composite material radiopaque.⁽²³⁾

In chronological developments, the filler particle size was reduced from macro filler to micro fillers where the size of particles was just few micro meters. Then came the hybrids, a combination of micro and mini fillers,⁽²⁵⁾ thus improving the strength and handling properties. With the advent of Nanotechnology, the filler particle size went down to as low as a few nanometres there by enabling us to have very high filler loading for use in both the anterior and posterior regions. Thus it became easy to develop exclusive posterior composites having very high compressive strength and low wear rate.⁽²⁶⁾

2.2.3. The interfacial phase (coupling agent):

The most commonly used Silane (Coupling agent) are the organosilanes such as γ -methacryloxy propyltrimethoxysilane (MPS) (Figure 3). The unique dual functionality of Silane coupling agents can form chemical bridges that unite disparate organic and inorganic components.⁽²⁰⁾ This is exemplified by their use in creating an adhesive interphase in silica-reinforced polymeric dental

composites (Figure 4).⁽²⁷⁾ Stable adhesive bonding of the filler to the resin is essential for the strength and durability of the composite and it may also act as a stress absorber at the filler-resin interface.⁽²³⁾ Lack of an adequate bond will permit dislodgment of the filler from the surface or ready penetration of water along the filler-matrix interface. As result, the manufacturer coats the surface of the filler with a suitable 'coupling agent'.⁽²⁰⁾

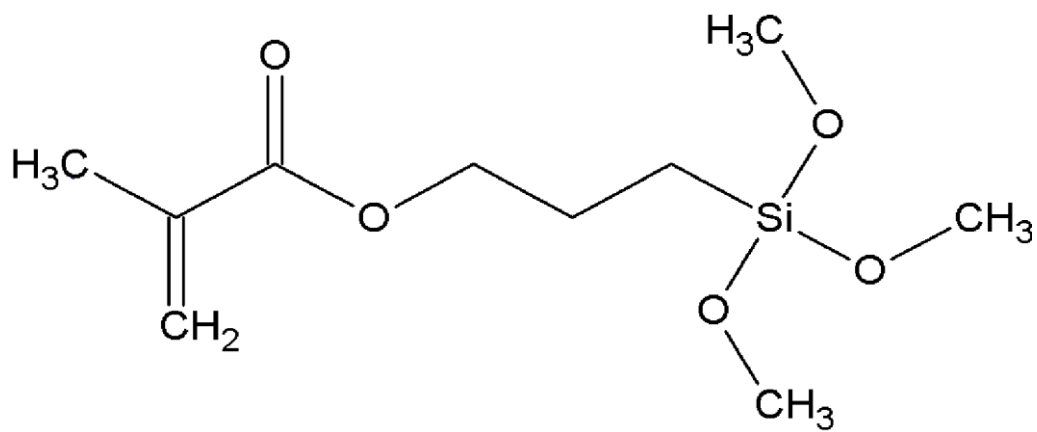


Figure 3: Chemical structure γ -methacryloxypropyl tri-methoxysilane.⁽²⁰⁾

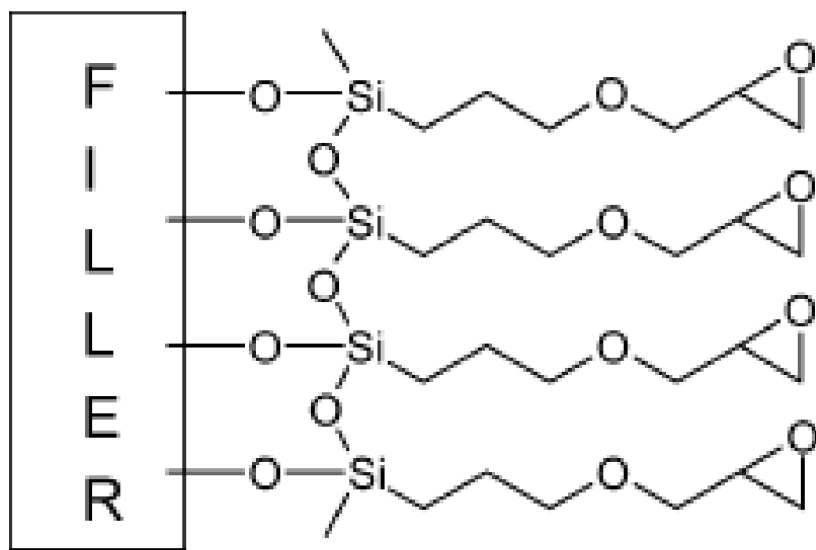


Figure 4: Silane Molecule.⁽²⁰⁾

2.2.4. Initiator and accelerator:

The polymerisation of composites can be attained by chemical means (self-curing) or by activation using visible light (light-curing). In chemical activation, polymerization is achieved by the reaction between an organic amine and a peroxide to produce free radicals, which in turn, attack the carbon double bonds and cause polymerisation. The components are supplied as a two-pack system, one containing a benzoyl peroxide initiator and the other a tertiary amine activator such as dimethyl- or dihydroxyethyl-p-toluidine (DMPTI or DHEPTI). Polymerisation starts as soon as the two pastes are mixed.^(15, 17) Light-activated materials are supplied as a single paste and alpha diketone (Camphoroquinone) is the most frequently encountered photo-activator used in combination with a tertiary aliphatic amine reducing agent (4-n,n-dimethylamino-phenyl-ethanol, DMAPE). Rapid- photo curable composites are used in conjunction with plasma-arc lights. Dual-cure composites are also available, comprising initiators and accelerators that allow light-cure activation followed by self-curing.⁽¹⁶⁾

2.2.5. Inhibitor:

Inhibitors or stabiliser system is used to prevent unwanted polymerisation when the material is exposed to room light or to maximise the product's storage life prior to curing. Butylated Hydroxy Toluene [BHT] in concentration of 0.01wt% is the most common used inhibitor.^(19,28)

2.2.6. Pigments:

Optical modifiers or pigments are added to resin composite to give the material a natural appearance to resemble the tooth and also to achieve good colour stability. The most commonly used opacifiers are titanium dioxide and aluminium oxide (0.001 to 0.007 wt %). These optical modifiers can affect

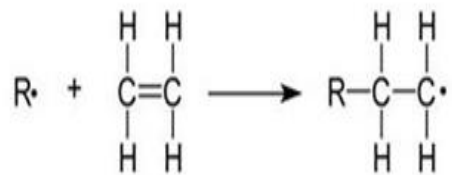
transmission of curing light through layers of resin composite and thus the darker the shade, the more curing time is needed.⁽¹⁶⁾

2.3. Photo-initiated polymerization.

Resin polymerisation is characterised by three processes; initiation, propagation and termination (Figure 5). As described earlier, the photo initiator is responsible for the initiation of the reaction by the production of free radicals(R). These promote polymerizations of monomers, these being formed when Photo-initiators are excited by irradiation (either UV or visible light),⁽²⁹⁾ which can then open the double bonds at both ends of the monomer leading to cross-linking and propagation.^(30, 8)

Typically, commercial dental composites are bi-functional methacrylate monomers, i.e. they have two reactive methacrylate groups per monomer in which following a free-radical-induced polymerization reaction, two carbon to carbon double bonds will have the capability to react and convert to a polymer.^(30, 8) This degree of conversion (DC) is a measure of the amount of methacrylate carbon double bonds (C=C) which have reacted to form covalent bonds with adjacent methacrylate groups.⁽³¹⁾ The DC is a critical factor that greatly influences several properties related to the composite restoration longevity such as the solubility, color stability, mechanical properties and even biocompatibility.⁽³²⁾ This type of reaction is strongly inhibited by free-radical scavengers such as oxygen in the surrounding atmosphere .^(8, 33)

– Initiation



– Propagation

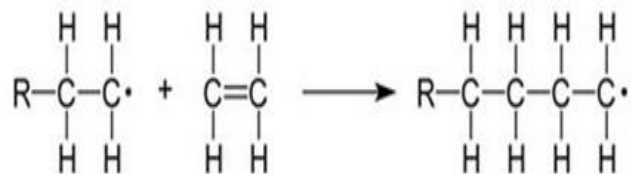
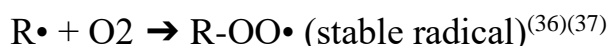


Figure 5: Representation of the initiation and propagation process during photopolymerisation.⁽²⁹⁾

2.4. The oxygen inhibition layer:

The inhibition resulting from oxygen diffusing from the atmosphere into curing resins, is responsible for the formation of soft, sticky superficial layers commonly found on freshly polymerized resins, referred to as oxygen inhibition layer.⁽³⁰⁾ This is due to the oxidation of radicals into stable species known as peroxides, which have low reactivity toward the methacrylate carbon-carbon double bonds thus retarding the formation of a polymer.^(8, 34, 35)



The oxygen inhibition layer can be defined as a layer of inhibited or retarded monomer present on the surface layer of methacrylate resins cured in the presence of oxygen. As result of that, the efficiency of initiation is reduced, leading to significant retardation (or even inhibition) of the polymerization.⁽³⁴⁾ The degree of oxygen inhibition during polymerization and the thickness of OIL may potentially be affected by factors such as monomer functionality and structure, the type and concentration of photo-initiators, the type of fillers, temperature, and polymerization conditions.⁽³⁸⁾ The oxygen inhibited layer is primarily composed of unreacted monomers and oligomers which are purported to have a similar composition to the uncured resin, although with partially consumed photo-initiators and co-initiators possessing a liquid-like consistency.⁽³⁹⁾

In the incremental insertion technique, which is recommended for ultimate success of resin based dental composite, the bond strength between different layers becomes important. The oxygen inhibition layer and it's undesirable reactions of photo-initiated polymerization, not only readily adopts the overlying increment to increase contact area, but also allows materials on both sides, where a covalent bond is established with the newly applied composite material,

to cross and bend together to form an inter-diffused zone. Copolymerization can then take place to produce a chemical bond.^(39, 40)

All these actions will tend to strengthen dental composite layer-layer interaction.^(8,30,40) However, it remains unclear whether the OIL serves a beneficial purpose in interfacial resin composite bond strength as a number of studies state that the oxygen inhibited layer is detrimental to bond strength due to its brittleness.^(38,35) Another studies concludes that the oxygen inhibited layer made no significant difference in bond strength between increments.^(30,41,36) On the other hand, a recent study by Al Hmedat *et al*,⁽⁴²⁾ showed that the presence of OIL will achieve maximum shear bond strength of composite increments.

2.5. Requirements of resin composite for dental restoration:

Firstly, functional properties, including enhanced longevity of the restoratives by excellent mechanical properties, These are high strength, fracture toughness, surface hardness, optimized modulus of elasticity, low wear, low water sorption and solubility, low polymerization shrinkage, low fatigue and degradation, and high radiopacity for better detection of the material during the removal of a composite restoration.⁽³¹⁾ Secondly, biological properties including good biocompatibility (systemic and local), no postoperative pain or hypersensitivity, preservation of tooth integrity in terms of not causing fractures or cracks, as well as caries-inhibiting abilities are required.⁽⁴³⁾ Finally, aesthetic considerations, such as good colour matching and stability (translucency, shades), optimum polishability, long-term surface gloss, absence of marginal or surface staining, and a good long-term anatomical form should also be fulfilled.^(6,43)

2.6. Classification of dental composite:

To improve many of the previously -mentioned properties, the size of filler particles incorporated in the resin matrix of commercial dental composites has continuously decreased over the years, from traditional to the Nano-composite materials.^(43,44) The Lutz & Philips (1983) classification,⁽⁴⁵⁾ which is still popular, allows composites to be ordered according to filler size into four main types (Figure 6).⁽¹⁸⁾ These types are:

1. ***Conventional or Macro filled resin composites***, had filler particles with a size of 10 –100 μm and their disadvantages were poor finish and relatively high wear. The most common used fillers were quartz and strontium or barium glass. Quartz filler had good aesthetics and durability but suffered from absence of radiopacity and high wear of antagonist teeth.^(27,44)
2. ***Microfilled resin composites*** were introduced in the late 1970s. They contain colloidal silica filler with a particle size of 0.01 – 0.05 μm . The small size made it possible to polish the resin composite to a smooth surface finish. A problem was in obtaining a high filler load. Compared to macrofilled resin composites, the microfilled did not have such good physical properties.^(27,44,45)
3. ***Hybrid resin composites*** were introduced to solve shrinkage problems of resin composites. The first introduced hybrid resin composites contained large filler particles of a size of 15–20 μm as well as colloidal silica of a particle size of 0.01– 0.05 μm .⁽²³⁾
4. ***Nanofilled resin composites*** contain filler particles with a size range from 20-1400nm, and are claimed to provide increased aesthetics, strength and durability.⁽²³⁾ Nanofilled composites are also available as Nano-hybrid composites, resulting from the introduction of Nano-sized particles into micro-hybrid composites.⁽²⁸⁾ This, consequently, reduces the polymerization

shrinkage and increases the mechanical properties. These characteristics are superior to those of microfilled composites.^(24,28)

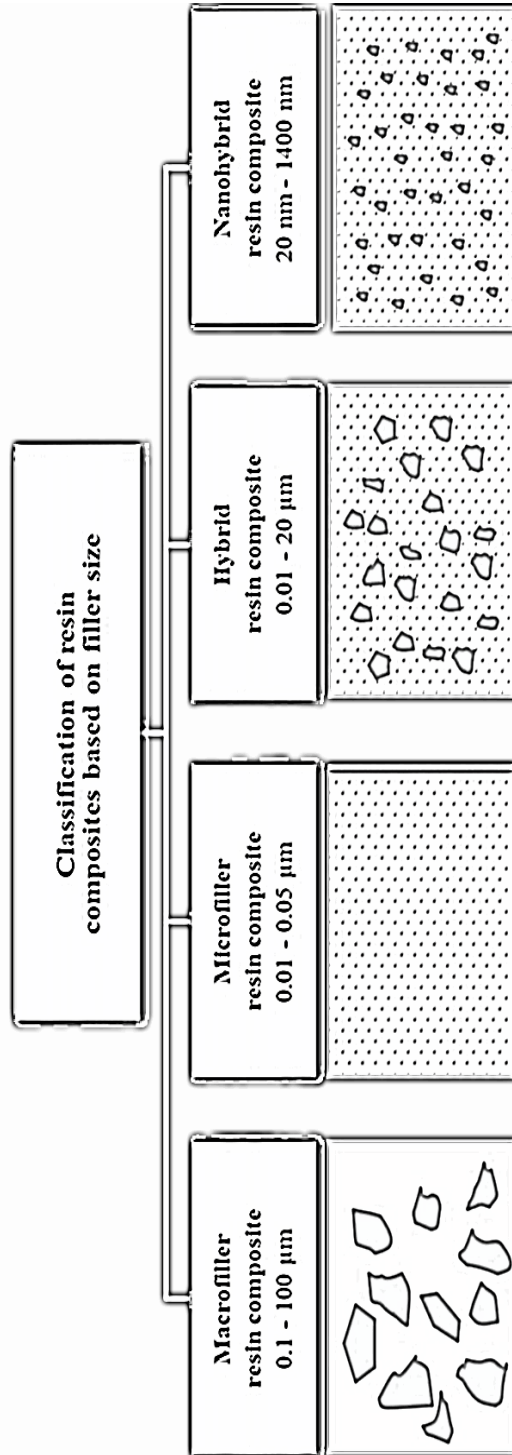


Figure 6: Classification of resin composites based on filler size.⁽⁴⁶⁾

2.7. Polymerization shrinkage:

Polymerization shrinkage is considered a major drawback of all dimethacrylate-based resin-matrix composites and is a critical limitation of dental resin-matrix composite.⁽⁴⁶⁾ This volumetric reduction occurs due to the formation of polymer out of monomers, where covalent bonds are created. In addition, intermolecular distance and free volume are reduced, which results in a shrinkage of the resin-matrix composite in the range of 2-6% .^(4,46,47) Polymerization shrinkage generates stress, resulting in debonding when the shrinkage stress surpasses the tooth-restoration interface bond strength.⁽⁴⁶⁾ This in turn, leads to a number of potential clinical problems such as penetration of saliva, bacteria, and other irritating substances through the debonded interface resulting in the postoperative hypersensitivity, secondary caries and pulpal inflammation.⁽⁴⁸⁾ This, finally, leads to restoration failure and replacing the whole composite materials.⁽⁴⁹⁾

2.7.1. Factors affecting the stress from polymerization shrinkage:

Stress from polymerization shrinkage is influenced by:

1. Chemical composition of the resin matrix and filler load percentage.
2. Incremental filling of RBCs.
3. Modulus of resin elasticity.
4. Polymerization rate.
5. Cavity configuration or the C-Factor.⁽⁵⁰⁾

2.7.1.1. Resin matrix/Filler load percentage:

Composite resins consist of polymer matrix and filler material. Shrinkage is a direct function of the volume fraction of polymer matrix in the composite. The more monomer entities unite into polymer chains and form networks, the higher the composite contraction.⁽⁵¹⁾ On the other hand, the space occupied by filler

particles does not participate in the curing contraction. Therefore, composites that contain lower levels of inorganic filler particles are more likely to produce high levels of polymerization stress.⁽⁴⁹⁾ Modifications to traditional composite chemistry can result in materials that produce lower polymerization stress levels.^(52,50)

2.7.1.2. Incremental filling of RBCs:

Concerns have been raised about the validity of this widely-practiced technique. As the RBC materials does not polymerise completely at the time of placement, in addition, the RBC does not fully shrink (only 70-85% shrinkage) at placement.⁽⁴⁸⁾ Consequently, when the subsequent increment is placed, it may not fully compensate the shrinkage of the underlying layer.^(53,51) It has also been suggested that the main advantage of using an incremental placement technique is to reduce shrinkage stress which is achieved by reduction of the C-factor via decreasing the volume of the restoration through reducing depth and/or diameter of the cavity. In turn this reduces the interface stress developed by the RBC polymerization.⁽⁵⁴⁾

2.7.1.3. Modulus of elasticity:

The elastic modulus is a measurement of a material's stiffness or rigidity, the lower the modulus the more elastic the material. The most rigid RBCs may generate the highest stress where as low viscosity resins are generally more flexible and therefore have a low elastic modulus which, purportedly, enables the material to absorb the stresses generated by shrinkage.⁽⁵²⁾

2.7.1.4. Polymerization rate (curing protocol):

When composite resin polymerizes, it goes through various stages of polymerisation. The most important stage regarding stress formation is when the material begins to solidify; this point is referred to as the gel point.⁽⁵⁵⁾

Shrinkage stresses are developed post-gelation of the resin, at which stage the material can no longer flow sufficiently to absorb the contraction caused by polymerisation.⁽⁵⁵⁾ Prior to gelation of the monomer, there is adequate monomer flow to overcome the stress developed.⁽⁵⁴⁾ Therefore, any forces developed before the gel point, contribute minimally to the overall stress of the polymer. Thus, a major area of interest is to delay the onset of the gel point by different curing regimes. However, Christensen *et al*,⁽⁵⁶⁾ suggested that different curing techniques resulted in no significant differences in the reduction of stress. Other techniques such as soft-start, two-step and pulse delay have also been used to try to decrease stress development,⁽⁵⁵⁾ although much controversy exists as to whether any significant reduction in stress can be achieved.^(50,56,57)

2.7.1.5. The C-Factor:

C-Factor is the ratio between bonded and unbonded surface area of the restoration, an increase in this ratio results in increased polymerization stress. For example: three-dimensional cavity preparations (Class I) have the highest polymerization stress.^(46,53) Controversially, another study by Pfeifer *et al*, reported that for a given restoration, stress generation was more influenced by cavity depth and diameter, where increasing depth and/or diameter caused an increased stress production, regardless of C-factor values.⁽⁸⁾

Furthermore, the authors concluded that the C-factor may only be useful to compare stress generation where similar volumes of restorative material are tested. Therefore the Configuration factor as a significant issue in stress generation in RBC restorations will continue to be debated in dental literature.⁽⁵⁷⁾

2.7.2. Alternative methods for reducing shrinkage in dental composites:

There have been many attempts to minimize the stress from polymerization shrinkage either by:

2.7.2.1. Improving curing technique and methods:

The composite exhibited improved physical properties when cured at a low intensity and with slow polymerization vs. higher intensity and faster polymerization. For example; “soft-start” polymerization enables, for as long as possible, the pre-gelation phase to permit slow polymerization by increasing light intensity more gradually from the curing units as this may reduce the polymerisation shrinkage stress.^(60,61) Moreover, Meereis *et al*, stated that the use of an alternative photo-activation mode (intermittent light, exponential, soft-start or pulse delay modes) was shown to be an effective strategy for reducing and controlling stress development in resin-based dental materials.⁽⁶¹⁾

2.7.2.2. Improving placement technique:

Another approach which has been proposed to overcome the polymerization shrinkage stress is the use of different restorative insertion techniques (Figure7).⁽⁷⁾ By using specific restorative techniques, stresses resulting from polymerization shrinkage might be reduced. However, it is not clear which restorative technique should be used to reduce the shrinkage and resulting stresses.⁽⁶²⁾

Applying the composite in increments instead of using a bulk technique, is suggested to overcome the limited depth of cure of the material and to reduce the shrinkage stresses. It initially reduces micro leakage at the interface (resin-tooth) eliminating undesirable consequences.⁽⁶³⁾ The clinically advised maximum thickness of RBCs material during incremental placement, is around 2-3 mm (dependent on material and curing light properties).^(31, 65) If the RBC is layered any thicker this will reduce the energy transferred to the bottom level of the cavity, leading to inadequate polymerization of the deeper segments of the composite,⁽⁶⁵⁾ which may adversely affect the longevity and overall mechanical,

physical and biological (increases its cytotoxicity) properties of the restoration.⁽⁴⁷⁾

Unfortunately, layering technique has certain disadvantages, such as the possibility of contamination, failures in bonding between resin composite layers, and void formation.^(66, 68) All of this increases the treatment time/chair time, mainly when compared with the bulk insertion technique.⁽⁶⁸⁾ Conflicting results have been reported regarding the efficacy of incremental filling technique.⁽⁶⁹⁾ According to Abbas *et al*,⁽⁶⁶⁾ by using cuspal deflection measurements in premolars, multiple increments induced higher shrinkage stress and cuspal deflection.

On the other hand, Deliperri *et al*, recommended the use of a bulk technique because it reduces the stress at the cavo-surface margin.⁽⁷⁾ In contrast, Park JK *et al*, and Kwon Y *et al*, state that using “modified bonded disc method” and a universal testing machine respectively, had shown considerably reduced cuspal deflection with incremental filling compared to the bulk filling technique.^(62,70)

In other studies, different layering techniques have been shown to be unrelated to polymerization shrinkage stresses and cuspal deformation. Kuijs *et al*, used 3D FEA (Finite Element Analysis) to show that the differences produced by various filling techniques were smaller than expected.⁽⁴⁷⁾ In the experimental setups with tooth models, Kwon Y, *et al*, have seen that the magnitude of the cuspal deflection was not significantly different among the groups using different filling techniques.⁽⁷⁰⁾ Moreover, Opdam *et al*, have reported that there is no difference between bulk or layering techniques when pain or microleakage is concerned.⁽⁷¹⁾

Despite the controversy over the advantages of incremental build-up of composites, this technique has been broadly recommended in direct resin composite restoration as it is expected to decrease the C-factor (the ratio of

bonded surface to unbonded free surface).⁽⁶²⁾ The presence of a high C-factor is a risk for debonding within the resin–dentin interface .⁽⁷²⁾

This C-factor can be minimized by increasing the free surface and decreasing the bonded surface. This can be done by using an incremental layering technique which can be through either a horizontal layering technique or an oblique layering technique (Figure 7).⁽⁷²⁾

For deep Class I cavities, a study made by Nikolaenko, *et al*,⁽⁷²⁾ recommended that horizontal layering is the most promising way to get a good bond to the cavity floor. In another study by Jafarpour, *et al*, it is shown that there were no significant differences in cusp deformation among different incremental insertion techniques neither horizontal nor oblique (Figure 7).⁽⁷³⁾

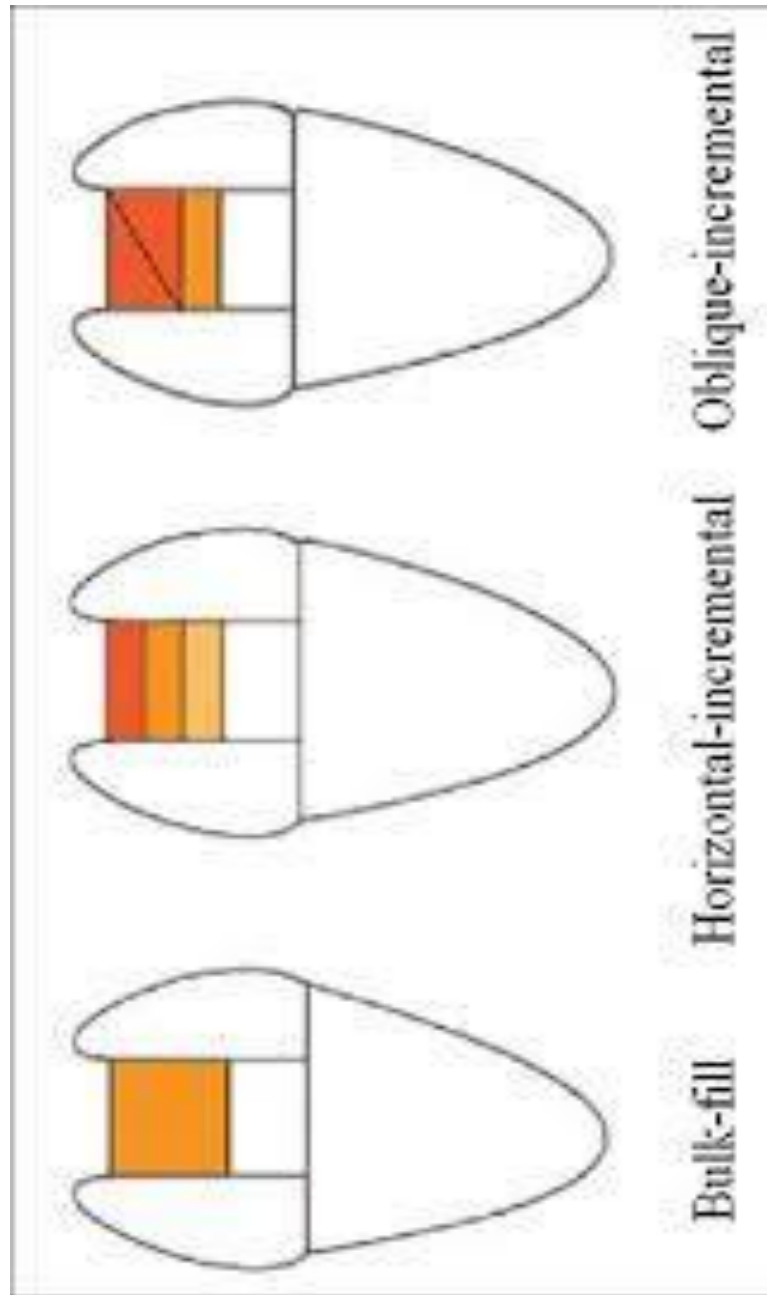


Figure 6: Schematic diagram illustrating the different composite placement techniques.⁽⁷³⁾

2.7.2.3. Improving composite resin composition and formulation:

In recent years, dental manufacturers have tried to address the deficiencies in dental composite restoratives through the development of enhanced dental composite formulas with reduced shrinkage, or reduced shrinkage stress.⁽⁴⁴⁾ The ideal dental composite would undergo zero, or at least low, shrinkage whilst setting. Zero shrinkage would ensure that the material remained physically adjacent to the tooth surface if originally placed there.⁽⁷⁴⁾

One of the promising contributions, in this sense, refers to the use of nanotechnology in novel material formulation. It consists in the production of materials and functional structures in the range of 0.1 to 100 nm (Nano scale). This is done through several physical and chemical methods where quartz, glass and ceramic fillers are converted in Nano fillers,⁽²⁴⁾ this in turn, higher filler content in composites lead to reduced shrinkage stress.⁽⁷⁵⁾ However, the dimensional stability of the composite restorative material is compromised by the polymerization reaction of the matrix phase.⁽⁶⁾ The conversion of the monomer molecules into a polymer network, is accompanied by a closer packing of the molecules. This leads to polymerization shrinkage,⁽³¹⁾ therefore, modification and alteration in formula and composition of the resin matrix, aimed at reducing the polymerization shrinkage and improving the marginal adaptation.⁽⁷⁶⁾

Since Bowen introduced Bis-GMA (bisphenol glycidyl dimethacrylate), in 1963⁽¹⁾ as the monomer system for dental composites, resin matrix composition changed and modified in various ways leading to superior physical, chemical and mechanical properties as well as clinical longevity.⁽⁶⁾ These changes were developed over time and contributed in very different directions to the product profile of the composites polymerize by their methacrylate functionality such as Triethylene glycol methacrylate, Polyacid-modified resin composites or

compomers,⁽⁷⁷⁾ and so called Ormocer (organically modified ceramics),⁽⁴³⁾ these have their common basis in the radical polymerization of methacrylate.⁽²⁸⁾ However, the two major drawbacks of dental composites, polymerization shrinkage and the related polymerization stress still have to be worked on.⁽⁶⁾

2.8. Development of a Silorane-based composite system:

Many years ago, efforts to overcome clinical deficiencies of RBCs have led to the development of new matrix materials.⁽⁷⁸⁾ In the last decade, 3M ESPE introduced Silorane, the new cationic ring opening monomer, as alternatives to methacrylate, the components of the RBC polymer matrix, due to its hydrophobicity and low polymerization shrinkage.^(6,79) Silorane is hybrid systems that contain both siloxane and oxirane-based monomers (Figure 8).⁽⁶⁾

The individual components of the Silorane base resin, combined to provide two main advantages: low polymerization shrinkage, due to ring opening polymerization of the oxirane monomer which exhibit approximately 1% shrinkage compared to 1.5-5% that is typical of traditional methacrylate. Secondly, increased hydrophobicity due to the hydrophobic nature of siloxane species.^(6,9,10,81,82)

The reduction in shrinkage stress has been attributed to the slow started polymerization associated with the cationic ring-opening polymerization which may last up to 20 minutes with delay at the gel point.^(12,79) This is achieved by opening of the oxirane ring during polymerization that extends their linkage through ring opening, flattening and extending toward each other (Figure 9).^(6,10) This in turn, will compensate the contraction-induced stress of resin composite.⁽⁸²⁾ Commercially silorane-based resin, is available as Filtek P90 (3M ESPE). It is a microhybrid composite composed of silorane matrix (23.0%), filled with fillers (76.0%) which are fine quartz particles and radio-opaque

yttrium fluoride (filler size between 0.1 and 2 μm),⁽⁶⁾ initiators (0.9%), stabilizers (0.13%), and pigments (0.005%).⁽⁸⁰⁾

Moreover, like the methacrylate based composite, Silorane-based composite also contains Camphorquinone, so that currently available dental curing units can be used for polymerization initiation.⁽¹¹⁾

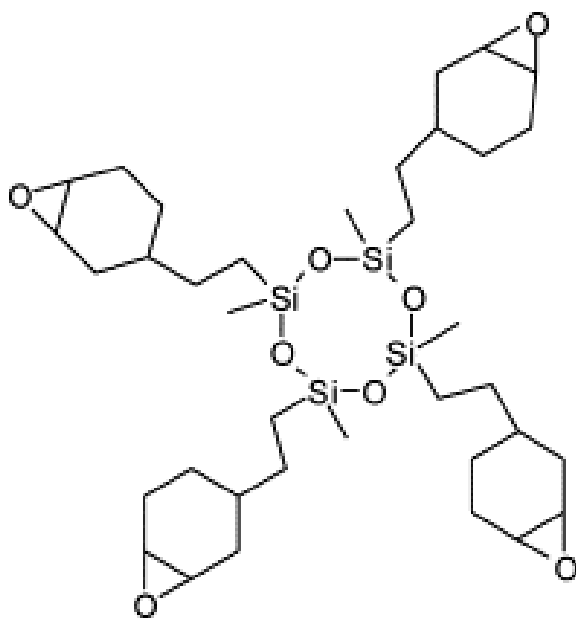


Figure 7: Silorane monomer .⁽⁶⁾

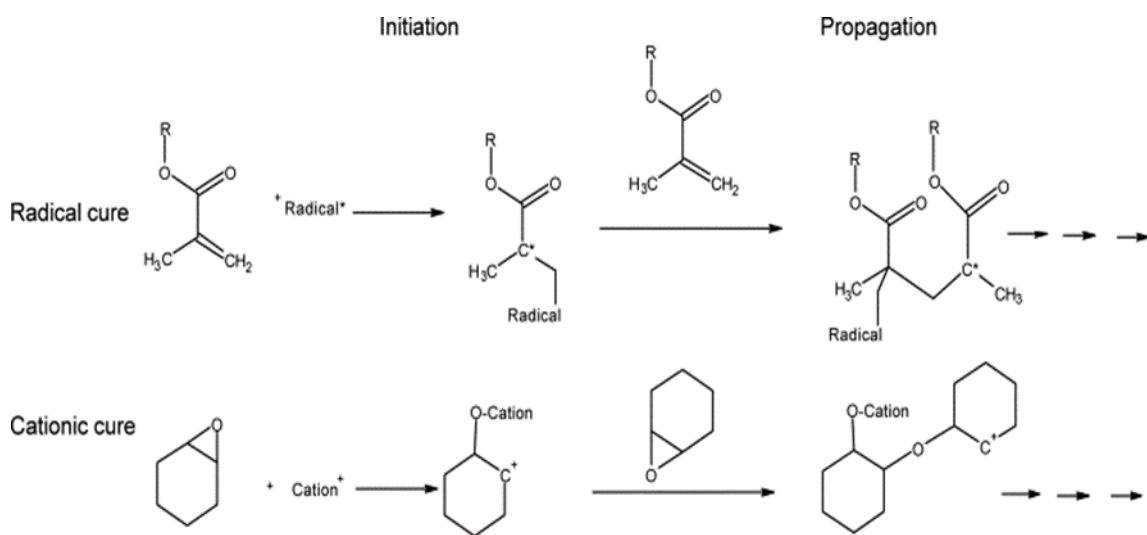


Figure 8: Polymerization of radical curing methacrylates and cationic curing ring opening epoxies. As shown, methacrylates are cured by radical intermediates and oxiranes polymerize via cationic intermediates. ⁽⁶⁾

Silorane-based composites are extremely hydrophobic, like other silicon containing monomers, in which siloxane (one of chemical building block of silorane composite) makes Silorane composites more stable for use in the oral environment as compared to conventional methacrylate.^(9,82,84) The advantage of the hydrophobicity of this restorative material, is that it leads to lower absorption of pigments present in the diet, and may reduce the potential for the adhesion of oral biofilms.^(79,81)

Additionally, Siloranes have been shown to have good storage stability in various media, and compared to conventional composites, they are less susceptible to changes if stored in ethanol.^(79,84) On the other hand, compounds containing oxirane in their chemical building blocks are usually known to be reactive with water,⁽¹⁰⁾ this in turn raising the questions on the stability of silorane-based composites in the oral environment. Eick *et al*, stated that siloxane makes the oxirane groups unreachable to water attack.⁽¹⁰⁾ This is supported by studies by Panahandeh *et al*, Kaleem *et al*, who reported that the hydrophobicity nature of silorane composite, preserved its integrity and mechanical properties to a greater extent, even if stored in water for a year compared to methacrylate-based composites.^(81, 83)

Apart from the predominant radical polymerization initiation in conventional methacrylate-based composites, the cationic ring-opening polymerization associated with the oxirane groups within Silorane is insensitive to oxygen. Tezvergil-Mutluay *et al*,⁽⁸⁵⁾ has suggested that no OIL exists on the surface of the freshly cured sample, this explained why cationic polymerization is called “living polymerization”⁽¹²⁾ as there is no inhibition or deactivation of polymerization initializing radicals.⁽¹³⁾ Contrary to expectations, Shawkat *et al* stated that Silorane exhibited an oxygen inhibition layer, this layer being significantly less than that of the methacrylate-based materials.⁽³⁷⁾

Silorane-based composite with less than 1% polymerization shrinkage,⁽¹³⁾ provokes the question whether it is possible to insert the Silorane composite with bulk insertion technique or whether an incremental insertion technique is still necessary. For Filtek Silorane, the incremental insertion technique is clearly applicable, the reason being that a depth of curing of maximally 2.5mm of composite resin, which makes incremental technique, is essential in cases of deep cavity,⁽¹³⁾ and necessary adjustments of the composite fillings by adding new composite resin.⁽⁸⁵⁾

2.9. *In-vitro* assessment of the shear bond strength of resin-resin interface of silorane composite resin:

Bonding property is one of silorane-based composite properties which needs improvement. Absence and reduction of Oxygen inhibition layer of silorane composites, along with the cationic polymerization, questions the adequacy of bond strength in between the incremental layers of these composites.⁽⁸⁵⁾

This has motivated scientists to examine the bonding of incremental layers of Silorane composite. For the evaluation of the composite to composite bond in the layering technique, the shear bond strength test appears to be the more widely used test, perhaps owing to its relative ease of application.⁽⁸⁶⁾ However, results based on experiments employing this test may be influenced by the modulus of elasticity of the material under investigation.

Available data from laboratory studies, shows that the shear bond strength is dependent on both the insertion technique and type of composite. Shawkat *et al*,⁽³⁷⁾ reported that incremental bond strength is not dependent on OIL since no differences in bond strength were observed between air and nitrogen atmospheres for any material following immediate placement. The same finding was issued by Al Musa *et al*, who concluded that Silorane-based resin composite incremental bond strength is comparable to that of methacrylate based

composite and is not affected by the absence of the oxygen-inhibited layer.⁽⁸⁷⁾ Furthermore, a study by Tavanger *et al*, demonstrated that there was no statistically significant difference between bulk and incremental techniques. It was also reported that when using a silorane-based composite system, the configuration of cavity design and polymerization process via light-curing technique, were as effective on bond stability as when a dimetacrylate-based composite system was used.⁽⁸⁸⁾ On the other hand, both Mutluay *et al*, and Vangelov L *et al*, concluded that the silorane-based composite showed lower shear bond strength between its successive layers than the conventional dimetacrylate composite.^(85,89)

The mechanical properties, including shear bond strength of both silorane and conventionally used methacrylate-based composites, have been extensively studied,⁽⁸¹⁾ however, literature is scarce regarding the effect of water storage on the mechanical properties of silorane-based composites.

Because of the lack of studies in this area, the current study conducted to evaluate the shear bond strength of low shrinkage silorane-based composite resin (composite-composite) with different insertion techniques (layering and bulk) stored in two different conditions (wet and dry) and comparing them with the conventional methacrylate based composite resin.

Aim of the study

Aim of the study

The aim of this study is to:

1. Determine the effect of insertion techniques, either layering (incremental) or bulk on shear bond strength of silorane composite and compare with other two established in literature methacrylate based composites resin.
2. Determine whether storing the composite resin in dry or wet conditions will affect the shear bond strength of the specimens.

Null hypothesis:

1. The shear bond strength of resin –resin interface of silorane-based composite when inserted with layering technique, is lower than one with methacrylate-based composite because of the absence and reduction of oxygen inhibition layer.
2. In cationic polymerization where the photo initiation radicals are insensitive to oxygen, the insertion with bulk technique is the option of choice.
3. Due to hydrophobicity of Silorane molecules, it is expected to preserve mechanical properties after storage in an aqueous environment.

Chapter III

Materials and Method

3.1. Experimental Design Overview:

This experiment was designed to evaluate the effect of two different insertion techniques (bulk and layering) and storage conditions (wet and dry), on the shear bond strength of low-shrinkage (silorane; cationic based monomer) composite, and compare it with two types of conventional methacrylate-based composites (Nanofilled and Hybride).

A total of 108 cylindrical composite samples were fabricated and sorted into three main groups according to the type of dental composite resin material used. 36 samples for each type of composite resin were used, and each group was further divided into two subgroups according to technique of insertion. 18 samples were inserted in bulk and 18 samples were inserted in layers for each material group. Each of the six subgroups was further divided into 12 divisions (nine each), according to the condition of storage for period of one week either dry or wet storage condition (Table 1).

3.2. Experimental materials used:

The materials used in this study are described in Table 2, the selected shade was A2.

Table 1: The groups and subgroups of experimental samples according to materials, insertion technique and used storage condition.

<i>Insertion technique</i>	Bulk		Layers	
	Storage condition		Storage condition	
<i>Materials used</i>	Dry	Wet	Dry	Wet
Silorane based resin	9	9	9	9
Nanofilled based resin	9	9	9	9
Hybride based resin	9	9	9	9

Table 2: Materials used in this study.

Materials type	Manufacture	Shade	Organic matrix	Inorganic matrix
FiltekP90-Silorane (Microhybride)	3M-ESPE	A2	Siloxane Oxirane.	53_73wt% Silanized quartz; yttrium fluoride (size of mean: 0.47 μ m).
Filtek-Supreme (Nanofilled)	3M-ESPE	A2	Bis-GMA UDMA TEGDMA	78.5wt% zirconia/silica particles with size of 5-20nm.
Tetric EvoCeram (Hybrid)	Ivoclar Vivadent	A2	Bis-GMA UDMA	76 wt. % Ba glass, silicate, SiO ₂ , mixed oxide with size 40nm-3000nm

3.3. Samples fabrication and design:

A total of 108 cylindrical composite samples were prepared in accordance with manufacture's recommendations. For each of the three composite materials tested, a custom-made metal cylindrical mould (Figure 10) was used to fabricate a customised cylindrical acrylic block, where a circular retentive cavity (4 mm diameter and 3 mm depth) was made by using Stainless steel cylindrical rod (Figure 13). A custom-made cylindrical over-matrix was made from Teflon with an internal diameter of 4 mm and a thickness of 2 mm used to build the composite resin cylinder. This was applied and adapted over the acrylic blocks with the aid of a specially constructed copper ring, in which the middle mould for composite became 5mm depth in total (Figure11-13). These measurements are close to what used by other researchers ^(36,88,91,92) in accordance to ISO4049:2000.⁽⁹²⁾

Samples were grouped into three main categories, according to the type of composite resin used resulted in 36 samples per group. Each group was then subdivided into two groups, according to techniques of insertion, 18 samples of each group inserted in bulk and 18 samples inserted in layers. Each group was further subdivided into two subgroups according to the storage condition i.e. one subgroup of nine samples were stored dry, and nine samples were stored in distilled water at 37°C for one week in the incubator (MLW, BST 5020, Germany) (Figure 15). In accordance with manufacturer's instructions and by fixing the Teflon over-matrix on the acrylic cylindrical blocks, the bulk groups of each composite type, resin was inserted and packed in one increment measured 5mm in depth and 4 mm in diameter. The increment was carefully placed and condensed with a clean plastic filling spatula in order to avoid contamination and/or incorporation of voids. This was followed by light-curing using a dental LED curing light (Figure 14) (Tulip series; wave length 420- 480,

luminous Intensity 1200 m W/cm²), applying the curing unit tip directly and centring on the sample surfaces for 40 seconds.

For the groups inserted with layering technique, the composite inserted in two increments, the first increment was inserted in the mould of acrylic block measuring 3mm in depth and 4mm in diameter, then light-cured for 20 seconds. The second increment was packed in Teflon over-matrix with 2 mm depth (Figure 11) then covered and compressed with a glass slab in order to obtain a smooth surface, after light-cured for 20 seconds followed the manufacturer's instructions, the samples were carefully removed after two minutes and inspected for defects or any resin flash. All 108 composite resin samples were stored for one week in two conditions at 37 °C ±1, in which one group was stored in containers with distilled water and the other stored in dry containers, all containers placed in incubator (Figure 15) (MLW, BST5020, Germany) for the whole period of storage.

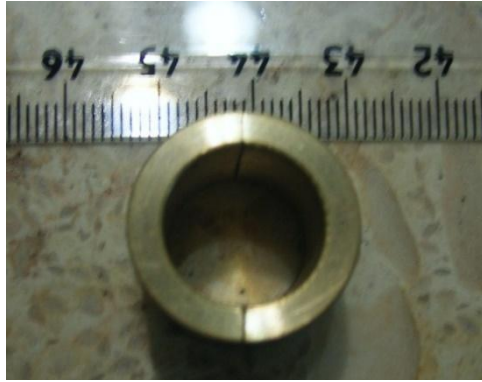


Figure 9: Metal cylindrical mould for acrylic block



Figure 10: Over Teflon matrix internal measurement.

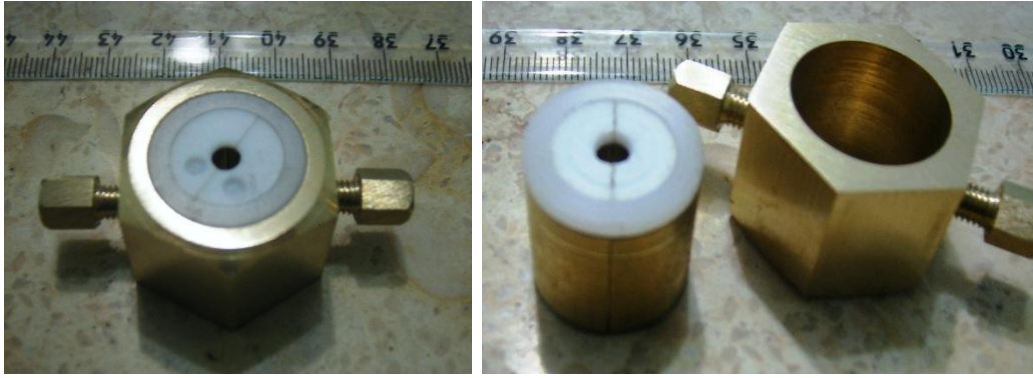


Figure 11: All components of the mould, including the over Teflon matrix and specially constructed copper ring to aid adaptation.

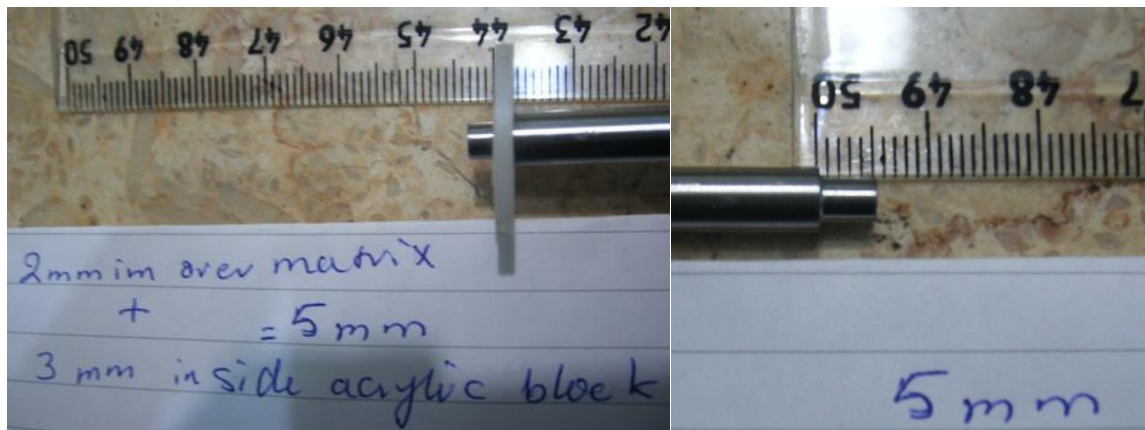


Figure 12: Stainless steel cylindrical rod to make a mould inside an acrylic block.



Figure 13: LED curing device: digital LED curing light, wavelength 420-480 nm, and lamination intensity 1200 mW/cm.



Figure 14: Incubator: MLW, BST5020, Germany.

3.4. Shear bond strength test:

After one week storage period in two conditions at $37\text{ }^{\circ}\text{C} \pm 1$, all 108 samples were placed in a custom-made cylindrical holder to be adapted in the universal testing machine (Comten industries, Inc. St .Petersburg, Florida, USA, Model No .942D10-20.) (Figure 16 and 17). In accordance to ASTM D5379 specification for the shear test of composite,⁽⁹³⁾ a chisel-shaped shearing rod centrally positioned on the specimen parallel to the interface between the composite portion submersed in acrylic block and the portion over the level of block surface (Figure 16). The load cell carrying 30 KN, was applied with a crosshead speed of 0.5 mm/min until fracture occurred. The testing machine recorded the load at fracture in kilograms. The load at fracture was converted to Newton and divided by the cross sectional area of the composite cylindrical samples (12.56 mm^2) to be converted into Megapascals (MPs) by using the following formula:

Shear bond strength in (MPs) =

Fracture force in kilograms $\times 9.81 = \text{N} / \text{Cross sectional area mm}^2 (12.56\text{ mm}^2)$.

Gravitational constant = 9.81.

Where N is the load at failure in Newton, mm^2 is the specimen cross-sectional surface area and MPa is the load at shear failure in Megapascals. After testing, the mode of failure was visually identified and recorded as either cohesive, adhesive or a mixed pattern of failure.

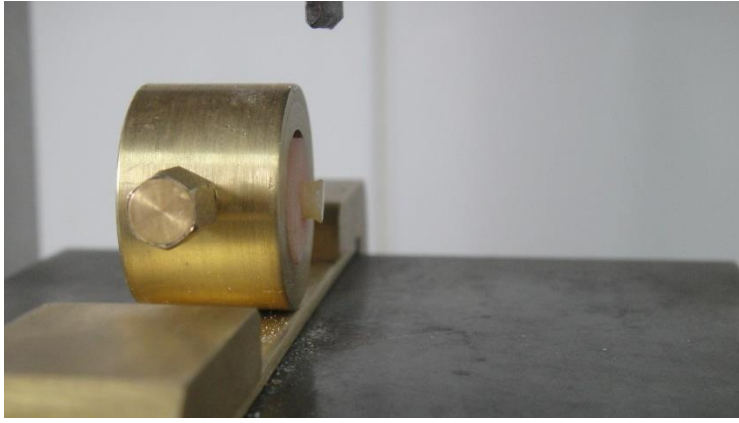


Figure 15: Sample in custom-made cylindrical holder.



Figure 16: Universal testing machine Comten industries, Inc. St .Petersburg, Florida, USA, Model No .942D10-20.

3.5. Light Microscope:

After the shear tests, in attempt to evaluate the fractured surfaces texture created by shear test (mode of failure assessment), the tested samples were visually evaluated using a light microscope with a magnification of 20 and 40. Fracture surfaces were classified as either adhesive, cohesive or mixed (Figure 18).

3.6. Statistical Analysis:

The data of shear bond strength values for all groups were subjected to statistical analyses using analysis of variance (ANOVA) and Tukey's post hoc method was used (followed supervisor's recommendation) to compare the differences among groups of materials at the ($p < 0.05$) significant level by SPSS software version 21.

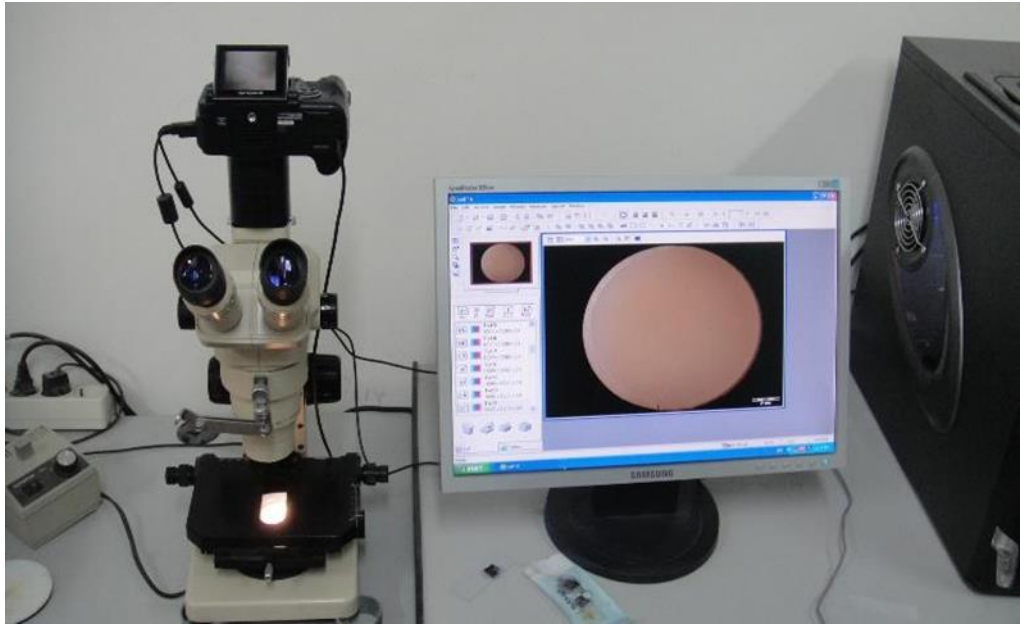


Figure 17: Light microscope to evaluate the fracture surfaces mode.

Chapter IV

Results.

4.1. Shear bond strength results of tested materials:

The results of shear bond strength for the 108 samples evaluated are presented in Table 3 and (Figure 19). It shows the mean and standard deviation of shear bond strength test with the p - values between the three materials tested with different insertions techniques (bulk and layering) in different storage conditions (dry and wet). Used one way ANOVA (Table 4), with illustration in table 5 a, b, c and d, revealed significant differences in the mean SBS results of Silorane, Nanofilled, and Hybride-based resin composites materials (Filtek90, Filtek Supreme and Tetric Ceram respectively) with different insertion techniques and storage conditions.

For the three composite resins where a bulk insertion technique was used and stored at dry condition, there was a significant statistical difference ($p < 0.05$) between the mean SBS of Nanofilled resin composite (36.45MPa) and the other two materials. This was higher than the mean SBS of Silorane (26.45MPa) and the mean SBS of Hybrid (27.51 MPa) (table 5a), while with the same insertion technique (bulk) used, at wet storage condition, there was no significant statistical differences ($p > 0.05$) between the mean SBS of Silorane, Nanofilled and Hybride based resin composite (26.78, 27.06 and 27.02 MPa) respectively (Table 5b).

For layering technique at dry storage conditions, the mean SBS value of Nanofilled was significantly reduced (21.35 MPa) ($p < 0.05$) compared with Silorane and Hybride, were both statistically exhibited no significant difference (27.50 MPa and 27.29 MPa respectively). (Table 5c). On the other hand, the mean SBS results of composite resins group using layering insertions technique in wet storage conditions, showed that there was no significant statistical difference between the mean SBS of both Silorane and Nanofilled resin (21.90 and 22.48 MPa respectively) and both of them were significantly lower than the mean SBS values of Hybride resin (28.21 MPa) ($p < 0.05$) (Table 5d).

Table 3: The mean and standard deviations of SBS of different materials with different insertion techniques and storage conditions.

Type of materials	Insertion technique			
	Bulk		Layering	
	Storage condition		Storage condition	
	Dry	Wet	Dry	Wet
Silorane	26.45±3.380 a	26.78±1.829	27.50±2.399 c	21.90±3.447 e
Nanofilled	36.45±4.871 b	27.06±2.079	21.35±4.896 d	22.48±2.491 e
Hybride	27.51±2.510 a	27.02±1.899	27.29±2.685 c	28.21±4.551 f
Significant ($P < 0.05$)	0.000	0.947	0.01	0.002

a, b, c, d, e, f. Different superscript lower-case letters in each column indicate statistically significant difference between different composite resins ($p < 0.05$).

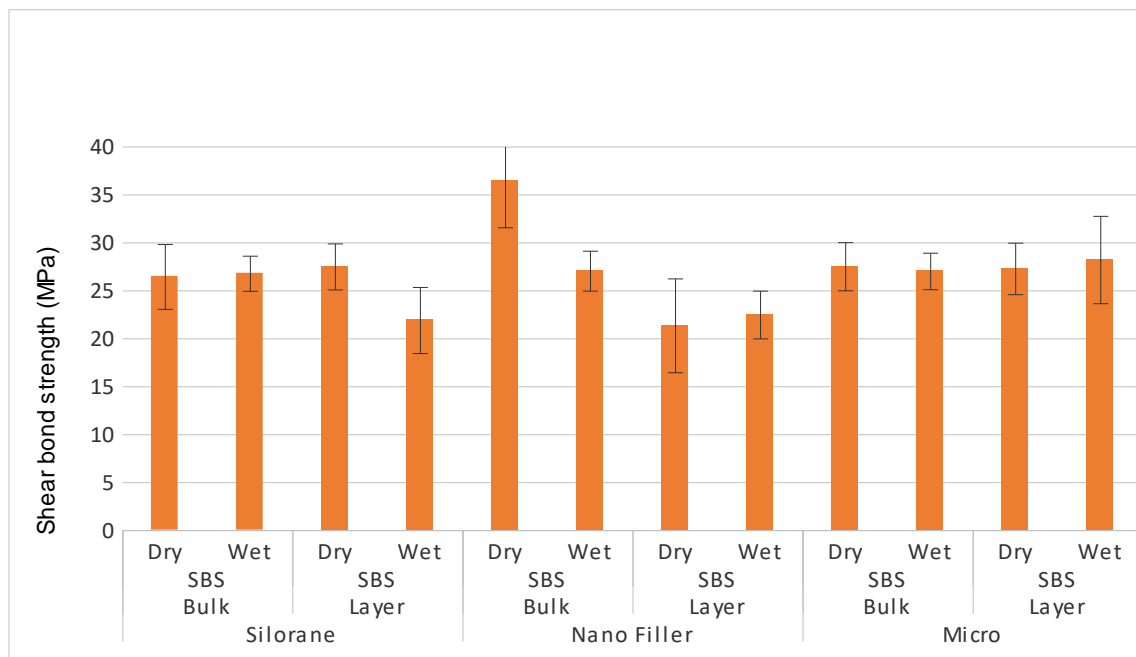


Figure 18: The means and standard deviation of shear bond strengths of different materials. Vertical lines represent standard deviations.

Table 4: A one-way ANOVA highlighting any significant differences in SBS of dental composite materials tested with varying insertion techniques in either wet or dry conditions ($p=0.05$).

Condition	Sum of Squares			df	Mean Square	F	Sig.
Dry	Bulk	Between Groups	542.815	2	271.408	19.640	.000*
		Within Groups	331.661	2 4	13.819		
		Total	874.477	2 6			
	Layer	Between Groups	219.010	2	109.505	8.894	.001*
		Within Groups	295.499	2 4	12.312		
		Total	514.509	2 6			
Wet	Bulk	Between Groups	.410	2	.205	.055	.947
		Within Groups	90.208	2 4	3.759		
		Total	90.618	2 6			
	Layer	Between Groups	219.128	2	109.564	8.471	.002*
		Within Groups	310.424	2 4	12.934		
		Total	529.552	2 6			

*. The significant mean difference at the 0.05 level.

Homogeneous Subsets' mean SBS values (MPa).

Table 5a: Bulk insertion technique /at dry storage conditions.

Material	N	Subset for alpha = 0.05	
		1	2
Silorane	9	26.45	
Hybride	9	27.51	
NanoFilled	9		36.45
Sig.		.819	1.000

Table 5 b: Bulk insertion technique /at wet storage conditions.

Material	N	Subset for alpha = 0.05
		1
Silorane	9	26.78
Hybride	9	27.02
NanoFilled	9	27.06
Sig.		.950

Table 5c: used layer insertion technique /at dry storage condition.

Material	N	Subset for alpha = 0.05	
		1	2
NanoFilled	9	21.35	
Hybride	9		27.29
Silorane	9		27.50
Sig.		1.000	.991

Table 5 d: used layer insertion technique /at wet storage condition.

Material	N	Subset for alpha = 0.05	
		1	2
Silorane	9	21.90	
NanoFilled	9	22.48	
Hybride	9		28.21
Sig.		.939	1.000

*: Significant at $p \leq 0.05$, Means with different number (1, 2) are statistically significantly different according to Tukey's test.

4.2. Effect of insertion technique on shear bond strength:

Table (6a) shows the comparison between the mean SBS values (in MPa) of the two insertion techniques (bulk and layer) regardless of other variables (material tested and storage condition). They showed that there is no significant statistical difference in SBS values when two insertion techniques are used with Silorane in dry conditions ($p>0.05$). Conversely, there is a significant statistical difference between SBS values of Silorane insertion in two techniques when stored in wet conditions ($P<0.05$).

On the other hand, the Nanofilled composite showed that there was a significant difference in SBS values when two insertion techniques are used in both storage condition ($P<0.05$). In contrast, there was no statistical difference between the mean SBS values of Hybrid dental composite resin when both insertion technique used in either wet or dry storage conditions.

Table 6 a: T-test: Comparison between two insertion technique within same material and storage condition variables.

Materials	Storage condition	Insertion technique	Mean SBS	P- value
Silorane	Dry	Bulk	26.45	0.463
		Layer	27.50	
	Wet	Bulk	26.78	0.003*
		Layer	21.90	
Nanofilled	Dry	Bulk	36.45	0.000*
		Layer	21.35	
	Wet	Bulk	27.60	0.001*
		Layer	22.48	
Hybride	Dry	Bulk	27.51	0.855
		Layer	27.29	
	Wet	Bulk	27.2	0.483
		Layer	28.21	

*= there is significant different $p < 0.05$

4.3. Effect of storage condition on SBS of composite resin:

Table (6b) illustrates the comparison between the mean SBS values (in MPa) of the different storage conditions (wet or dry) regardless of other variables (within same composite resin and insertion condition). They showed that when Silorane is inserted in bulk, there was no significant difference between the mean SBS whether the samples are stored in wet or dry conditions ($P>0.05$). While there was a significant difference when Silorane is inserted in layer ($P<0.05$). A totally reverse finding to Silorane was obtained with Nanofilled dental composite, in which there was a statistical difference between the two storage conditions when the Nano filler samples were inserted in bulk technique ($p<0.05$). On the other hand, there was no significant statistical difference if the Nanofilled sample is inserted in layer technique ($p>0.05$). In the case of Hybride, the mean SBS values were entirely similar with both insertion technique used at either wet or dry storage conditions ($P>0.05$).

Table 6 b: T-test: Comparison between two storage conditions using the same material and insertion technique variables.

Materials	Insertion technique	Storage condition	Mean SBS	<i>P</i> - value
Silorane	Bulk	Dry	26.45	0.805
		Wet	26.78	
	Layer	Dry	27.50	0.001*
		Wet	21.90	
NanoFilled	Bulk	Dry	36.45	0.000*
		Wet	27.06	
	Layer	Dry	21.35	0.550
		Wet	22.48	
Hybride	Bulk	Dry	27.51	0.644
		Wet	27.02	
	Layer	Dry	27.29	0.607
		Wet	28.21	

*= there is significant different $p < 0.05$.

4.4. Microscopic evaluation of fractured surfaces:

The fractured surfaces of the sheared specimens were visually evaluated using a light microscope with a magnification of 20 and 40, Fractures were classified as adhesive, cohesive and mixed mode of failure (Figure 23, 24 and 25).

4.5. Results of mode of failure:

The mode of failure results for all groups (n=9/group) are presented as percentages in Tables 7 a, b, c and (Figures 20, 21 and 22) according to the composite resin tested, insertion technique and storage condition used respectively.

4.5.1. Mode of failure regarding composite resin tested:

Table 7a and Figure 20, regarding the material tested (n=36/composite resin type), Nanofilled resin failed with the highest percentage rate of mixed failure (63.9%) and the lowest adhesive failure (2.8%), also possessing a similar cohesive failure with Hybride (both 22.2%). For Silorane, the highest percentage of failure mode observed is mixed mode of failure (58.3%) which was close to the Hybride's mixed mode of failure (52.8%), and the lowest percentage was the cohesive failure (8.3%).

Table 7a: Descriptive percentages of the failure modes of the sheared composite samples with consideration to types of material used.

Mode of failure	materials used		
	Silorane	Nano Filler	Micro
Mixed	58.3%	63.9%	52.8%
Adhesion	33.3%	2.8%	13.9%
Failure	0.0%	11.1%	11.1%
Cohesion	8.3%	22.2%	22.2%

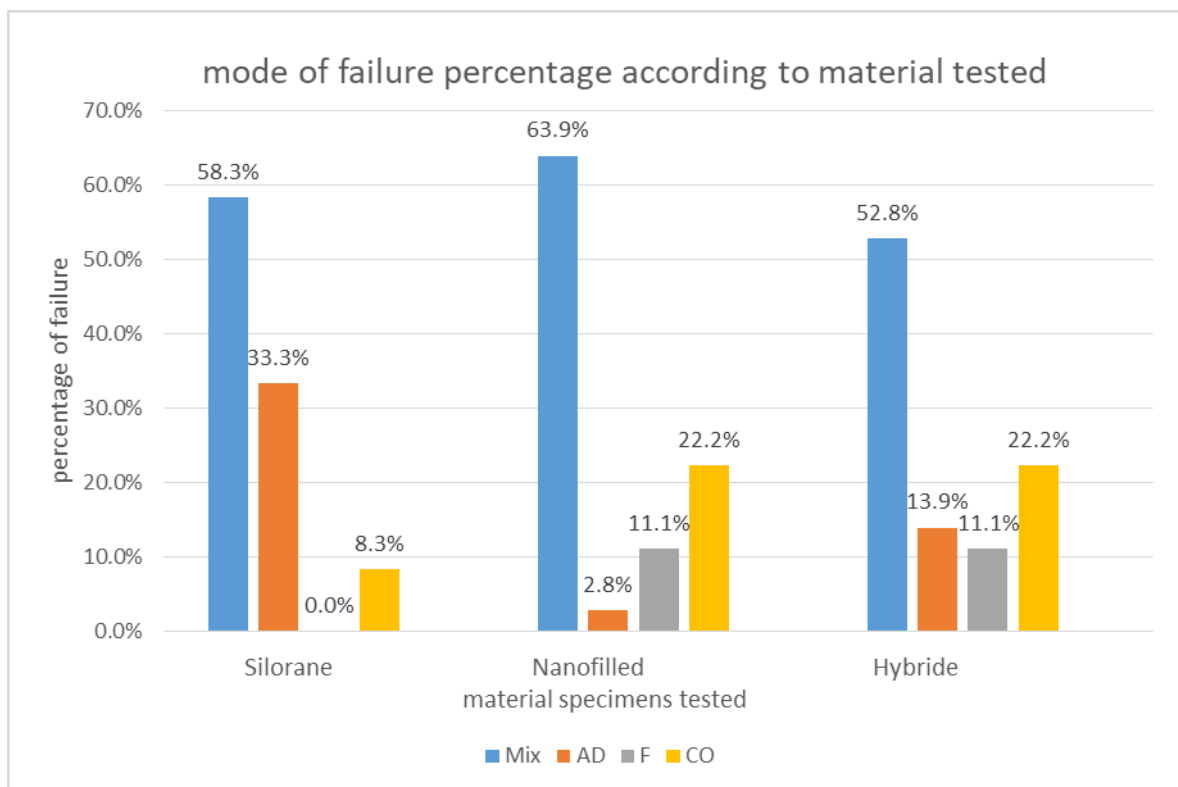


Figure 19: Diagrammatic representation of the failure modes of the sheared samples for all the tested groups according to the materials tested.

4.5.2. Mode of failure regarding insertion technique:

Table 7b and Figure 21, showed and illustrated the percentage of the failure modes in relation to the insertion technique used (n=54/bulk/layer). Sheared samples inserted in bulk technique had the highest mixed mode of failure percentage (75.90 %) and the lowest percentage was observed with adhesive mode (1.8%). Within the sample group inserted in layer, the highest mode of failure was mixed (40.70%).

Table 7b: Descriptive percentage of the failure modes of the sheared composite samples in consideration to insertion technique.

Mode of failure	insertion technique	
	Bulk	Layer
Mixed	75.90%	40.70%
Adhesion	1.80%	31.40%
Failure	3.70%	11.10%
Cohesion	18.50%	16.70%

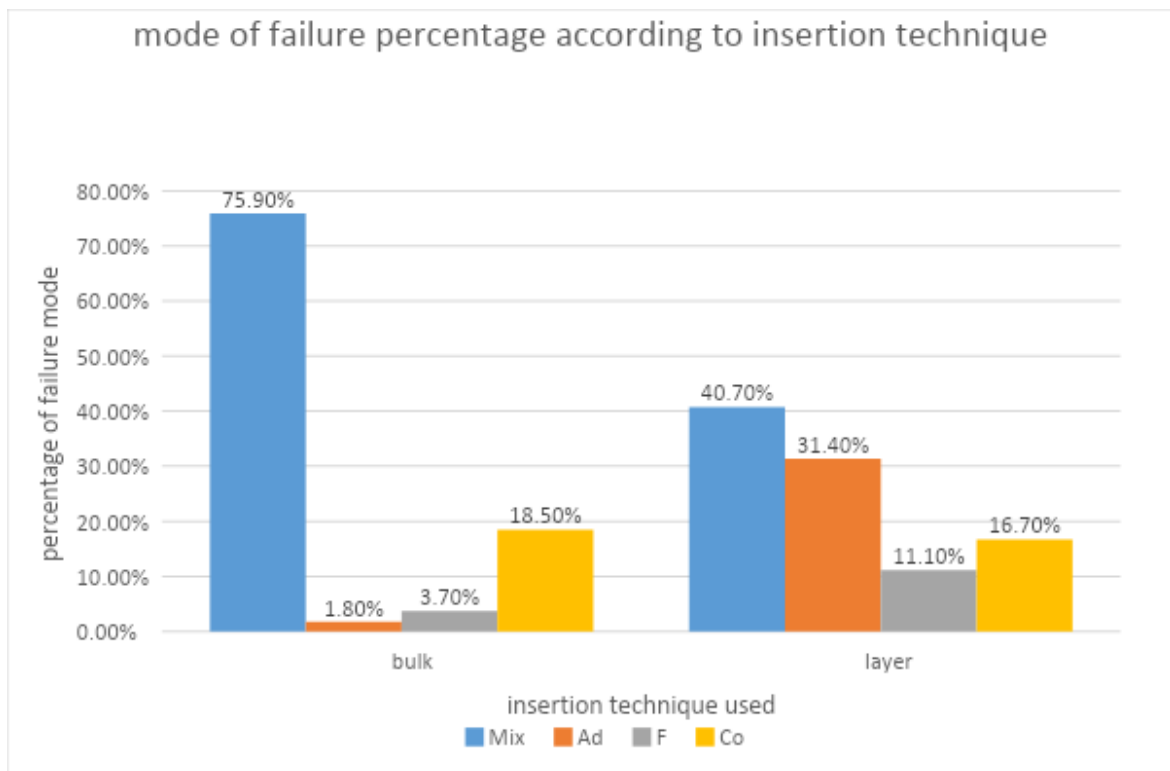


Figure 20: Diagrammatic representation of the failure modes of the sheared samples for all the tested groups according to the insertion technique.

4.5.3. Mode of failure regarding storage condition:

After one week storing period in either dry or wet conditions (n=54/dry/wet), as illustrated in Table 7c and Figure 22, the samples stored in dry showed 51.90% mixed failure but similar adhesive and cohesive percentage of failure (18.5% each), while the samples stored in wet showed 64.80% mixed failure but close adhesive and cohesive failure percentages (14.8%, 16.7%) respectively.

Table 7c: Descriptive percentage of the failure modes of the sheared composite samples in consideration to storage condition.

Mode of failure	Condition of storage	
	Dry	Wet
Mixed	51.90%	64.80%
Adhesion	18.50%	14.80%
Failure	11.10%	3.70%
Cohesion	18.50%	16.70%

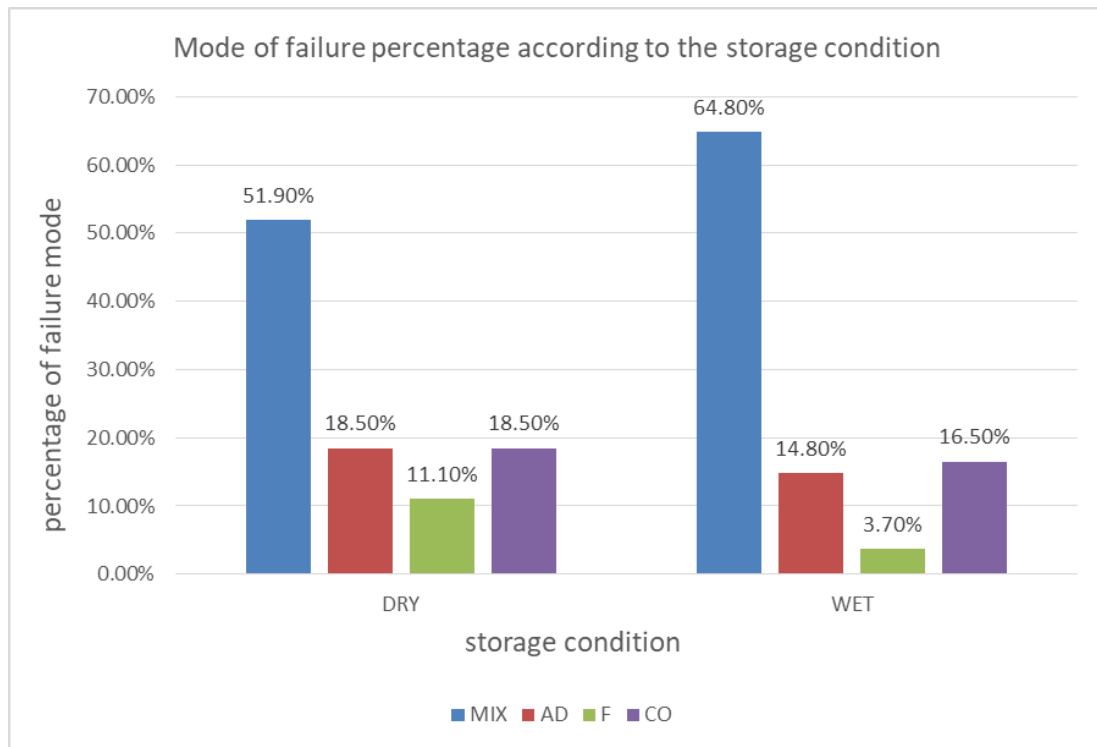


Figure 21: Diagrammatic representation of the failure modes of the sheared samples for all the tested groups according to the storage conditions.

4.6. Selected Stereomicroscope images of light microscope evaluation of the samples fracture surfaces:

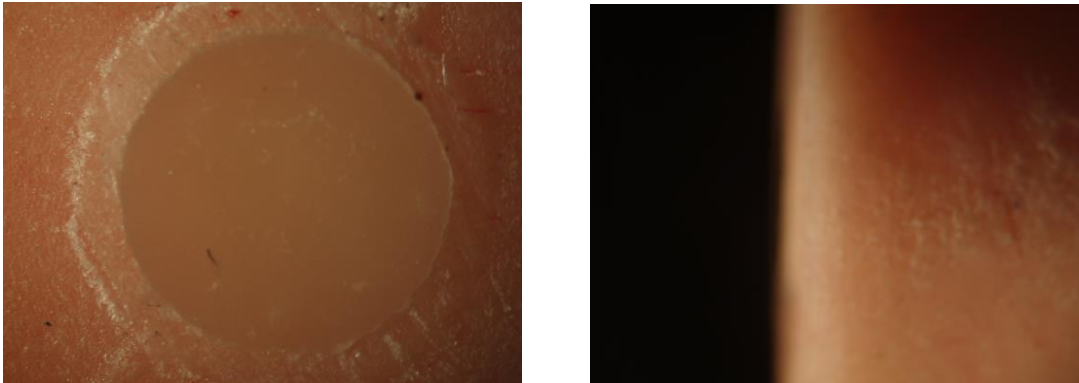


Figure 22: Stereomicroscope images of Silorane resin with layer insertion and dry condition (horizontal #20, vertical # 40 view) showed adhesive mode of failure.

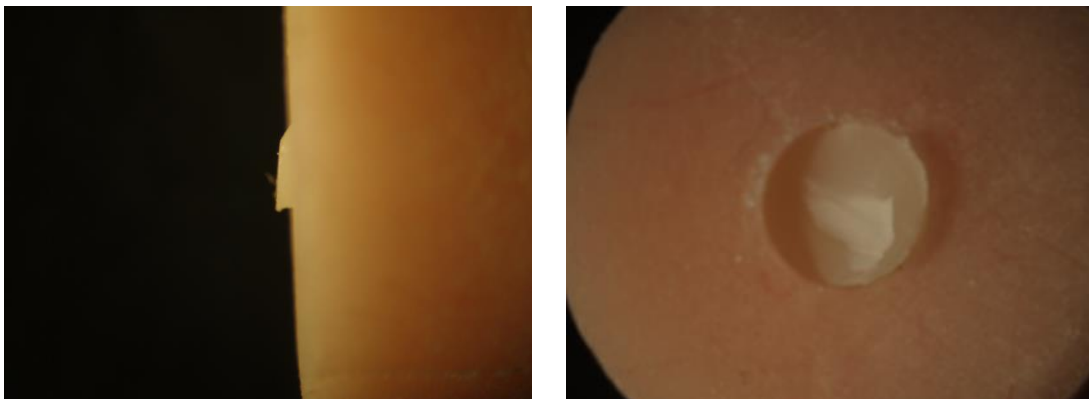


Figure 23: Stereomicroscope images of Nanofilled resin with layer insertion and dry condition (horizontal #20, vertical #20 view) showed a mixed mode of failure.

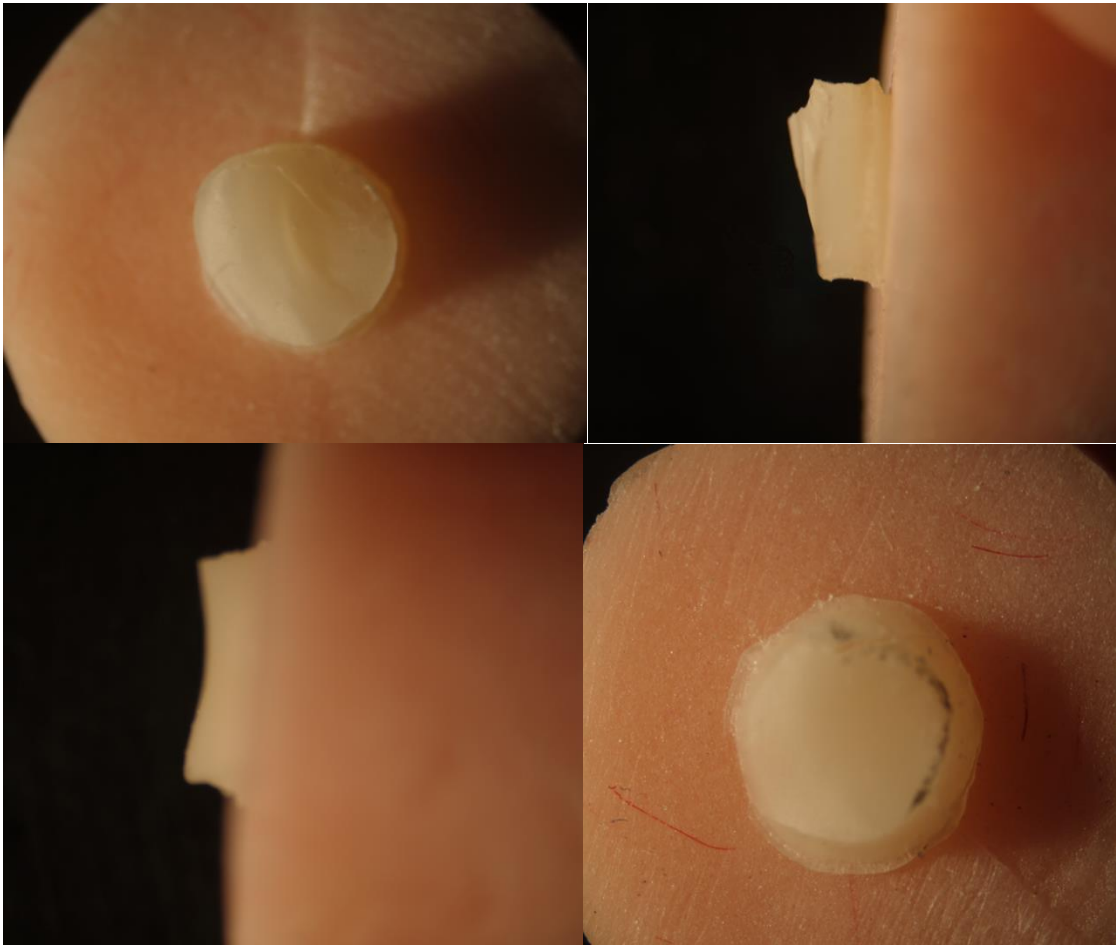


Figure 24: Stereomicroscope images of Hybrid resin specimens with bulk insertion and dry conditions (horizontal #20, vertical #20 view), showed cohesive mode of failure.

Chapter V

Discussion

Resin composite has become the material of choice in restorative dentistry, especially due to its aesthetic properties. Composites are essentially made of three basic components: resin-based organic matrix, inorganic filler (dispersed phase). and organic-inorganic bonding agent, Silane coupling agent.⁽²⁰⁾

Organic matrix is made of monomers, which, due to polymerization, bonds into polymers and forms a three-dimensional network, which is filled with fillers. In this way, the physical and mechanical properties of the network are improved.^(2,57)

The polymerization begins as the resin enters the pre-gel stage, during which the organic matrix is in viscous plastic form, allowing it to “flow”. At this stage the monomers can still move or flow into new positions within the organic matrix. The polymerization process continues, and the composite hardens and homogenizes into a solid body. The point at which any, and all, movement is no longer possible is referred to as the gel point, and denotes the transition from pre-gel to post-gel stage.⁽⁹⁴⁾ The material is in a stiff elastic state, but is still contracting, this shrinkage causes stress to occur. Gelation can be seen as the moment in which molecules within the material can no longer compensate the shrinkage. Total material shrinkage is determined by the pre-gel stage, during which the material can still be controlled.^(57,91)

Many efforts have been made in order to reduce polymerization shrinkage, either by changing material formulations (organic and inorganic phase) or by using different clinical techniques.^(13,78)

This study was conducted to investigate the effect of an insertion technique, either layering or bulk on shear bond strength of low polymerization shrinkage resin composite (Filtek Silorane), and compare it with Methacrylate (Nanofilled and Hybride) resin based composite after a storage period of one week, either in dry or wet conditions.

In vitro shear bond strengths are useful and essential for predicting the performance of silorane-based composite in comparison to conventional methacrylate-based resin. Sau *et al*,⁽⁹⁵⁾ reported that shear bond strength was the test of choice because it provided the most appropriate measure of the maximum stress applied at the bonding interface between the resin increments. In addition, restorations when used in the anterior and posterior regions are commonly subjected to shear forces during mastication.^(90,95) Furthermore, shear tests include ease of specimen preparation and simple test protocol.⁽⁹⁶⁾

Studies in publication reported that, the greatest change in mechanical properties of composites occurred within the first seven days after exposure to an aqueous environment.^(95,96) For this reason, the specimens in this study were stored in an incubator for one week before shear test, in which nine specimens of each material inserted either with layer or bulk insertion technique were stored in distilled water and the other nine specimens were stored dry, both at $37\pm 1^{\circ}\text{C}$. Distilled water simulates the wet oral environment provided by saliva and water.⁽⁸¹⁾ As for the water absorption and water solubility of dental RBC materials, they depend on a host of factors: chemistry of the monomer resins, the extent of polymerization of the polymer matrix,⁽⁷⁹⁾ filler particle size, shape,⁽⁸³⁾ and distribution, and the interfacial properties between the filler and resin matrix.^(53,96)

In this study, for three different materials inserted with bulk technique, there was significant statistical differences ($p < 0.05$) between the mean SBS of Nanofilled resin composite (36.45MPa) and the other two materials. The mean SBS of Nanofilled resin was higher than the mean SBS of Silorane (26.45MPa) and the mean SBS of Hybrid resin (27.51 MPa). Yet with the same insertion technique (bulk) used, at wet storage conditions, there was no significant statistical differences ($p > 0.05$) between the means SBS of Silorane, Nanofilled and Hybride resin composite (26.78, 27.06 and 27.02 MPa) respectively.

The highest difference in shear bond strength within same materials after one week of storage in dry and wet conditions was seen in the Nanofilled resin composite specimens. The SBS of Nanofilled specimens stored in wet conditions (26.78 MPa) was approximately 27% lower than that of specimens stored in dry conditions (36.45 MPa). The lowest significant change was seen in Filtek silorane (2%) and Hybride resin (2%), but both Silorane (26.45-26.78 MPa) and Hybride based resin (27.51-27.02 MPa) showed no significant difference between each other in either dry or wet conditions ($p>0.05$).

In this study, the differences in shear bond strength could be explained by the variations in the chemistry of monomer and size, shape, type and amount of filler particles present in the compositions of the tested materials (Table 2) which largely decides the mechanical properties of the restoration material.^(84,95)

Regarding Nanofilled (Filtek Supreme) resin composite which contains zirconia particles, it showed the highest significant reduction when stored in a wet environment. These results are explained by the findings of a study undertaken by Asopa *et al*,⁽¹⁰⁰⁾ who examined the effect of incorporating ZrO_2 with different percentages on water absorption of resin. Their results showed a statistical increase in water absorption as the percentage of ZrO_2 increase, due to the fact that water molecules were able to penetrate between polymer chains.

Furthermore, the interface between the particle and polymer was water sensitive because of high surface energy of the particle related to the polymer and the permeability of the polymer which allows water to reach the interface, this in turn results in more water absorption, in addition to TEGDMA-based resin-composites releasing higher quantities of monomers into aqueous environments when compared to Bis-GMA and UDMA-based materials.⁽¹⁰¹⁾ All of these reasons may contribute to effecting the mechanical properties of the Nanofilled resin composite when stored in wet conditions.^(97,100)

On the other hand, in the case of Silorane, because the hydrophobicity of siloxane molecule, the silorane-based resins were expected to show superior properties in aqueous environments compared to methacrylate-based composites. Panahandeh *et al*, and Kaleem *et al*, in their studies reported that storage conditions followed by the type of composite were the most important factors affecting the mechanical properties of composites. In addition, Filtek P90 silorane-based composite more effectively preserved its integrity and mechanical properties after one week of storage in wet and dry conditions. ^(81,83)

The same findings were observed by Eick *et al*, who concluded that the oxirane functionality in siloranes was stable in all the aqueous biological fluids tested. This lack of solubility of the siloranes is due to the hydrophobic siloxane which makes the oxirane unreachable by the water. ⁽¹⁰⁾

Furthermore, Palin *et al*,⁽⁷⁹⁾ in their study assessed the effect of water absorption and solubility on mechanical properties of an experimental oxirane and silorane RBC, their conclusion came in accordance with the fact that the stability of silorane RBC in aqueous environment is made by the siloxane component, in which a significant increase in water absorption and solubility of an experimental oxirane RBC, was manifested as a significant decrease in bi-axial flexure strength following water immersion. In contrast, the experimental silorane RBC, exhibited decreased water absorption and solubility, which improves hydrolytic stability and higher bi-axial flexure strength compared with conventional methacrylate based materials. ⁽⁷⁹⁾

These results came in accordance with our findings for the silorane-based composite inserted with bulk technique, the conditioning in water did not lead to a significantly different SBS values (26.45 MPa) when compared with the specimens stored in dry conditions (26.78MPa).

In this case, the testable hypothesis that due to hydrophobicity of silorane resin will be expected to show preservation of mechanical properties after being stored in an aqueous environment, was accepted with the specimens inserted in bulk technique.

One of the approaches to minimize the effects of curing shrinkage, is the insertion of resin composite in increments. Silorane still has shrinkage to some extent, especially in the deep cavities,⁽⁶⁷⁾ where incremental insertion techniques are still the option of choice. Additionally, adjustments of the composite fillings by adding new composite resin, require good adhesive properties for the substrate composite.⁽¹⁰²⁾

In the present study, three dental composite specimens were inserted with incremental technique (first increment 3mm and the over layer 2mm), stored dry and wet for one week to investigate the interfacial bonding properties of Silorane-based composite, and compared it with other two Methacrylate-based resins. In addition, they were compared with the specimens made from the same three types of composite resin inserted with bulk insertion technique (discussed earlier in this chapter).

Many studies concluded that, the interfacial bond strengths between resin composite layers can potentially be affected by several factors, such as surface monomer reactivity, material viscosity, intermediary bonding materials, type of curing procedure and the oxygen inhibition layer.^(10,34,37,103)

In the current study, Silorane and Hybride based resin's mean SBS values (27.50 MPa and 27.29MPa respectively) when inserted with layering technique at dry storage condition, exhibited statistically no significant difference to specimens inserted in bulk and stored either dry or wet (26.45 MPa and 27.51 MPa for dry specimens of Silorane resin and 26.78 MPa, 27.02 MPa for wet specimens of Hybride based resin respectively) ($p>0.05$).

For Nanofilled resin, mean SBS value was significantly reduced (21.35 MPa) than the specimens inserted in bulk and stored either dry or wet (36.45, 27.06 MPa) ($p < 0.05$). This reduction of SBS values could be explained by the findings of Shawkat *et al*, who concluded that the increasing filler load may lead to complicating the effects of resin viscosity and oxygen diffusion.⁽³⁷⁾

Several studies have shown that an oxygen-inhibited layer of free radical polymerized methacrylate resins was crucial for enhanced bond strength between the composite layers by means of remaining unreacted acrylate groups forming chemical covalent bonds with an interpenetrating network.^(33,34,36,37,39,40)

The ring-opening polymerization of the Silorane molecule is cationic polymerization reaction where the oxygen inhibition layer is reduced or does not exist on the surface of the composite after polymerization in surrounding atmosphere.^(37,85)

In current study, the mean SBS values of Silorane specimens inserted in layers and stored dry, were insignificantly different from Silorane specimens inserted with bulk technique and stored either dry or wet. This shows that the absence or decrease of an oxygen inhibited layer has no effect on the resultant SBS of Silorane consecutive layers, especially with dry stored specimens. This created agreement with the findings of Shawkat *et al*,⁽³⁷⁾ and Al Musa *et al*.⁽⁸⁷⁾ The former's study compared the incremental bond strength of Silorane composite in oxygen and nitrogen atmospheres, they found no significant difference between the two groups, concluding that the absence of an oxygen-inhibited layer did not affect the shear bond strength between two successive layers of silorane-based composites.⁽³⁷⁾ While study made by Al Musa *et al*, found that the bond strength between immediately placed incremental layers of the silorane composite was not different from that of the methacrylate-based composite⁽⁸⁷⁾.

As result, the testable hypothesis that due to the absence and reduction of an oxygen inhibition layer, the shear bond strength of resin –resin interface of silorane composite when inserted in layers is lower than that with methacrylate composite which was rejected with specimens stored dry.

On the other hand, regarding the storage condition used in this study, the Silorane composite specimens showed the highest significant difference between the composite- composite interfacial SBS values of specimens stored dry (27.50 MPa) and specimens stored wet (21.90 MPa) for one week. This came in agreement with the findings of Shawkat *et al*, Musa *et al*, and Tezvergil-Mutluay *et al*, regarding the effect of storage conditions on bonding between two successive layers of Silorane composite. They concluded that storing wet, negatively affects the resin composites by causing water absorption, chemical degradation, and leaching out of some of the constituents of the material. ^(37,87,85)

Furthermore, because the cationic ring-opening polymerization reaction is insensitive to oxygen, the incremental bond strength is dependent on the chemical reactivity of the material, which decreases over time. The chemical reactivity of silorane-based composite materials seems to undergo a significant drop when stored wet, this contributed to what stated earlier in the literature review of this study that, compounds containing oxirane in their chemical building blocks are usually known to be reactive with water,⁽¹⁰⁾ this in turn could cause a low incremental bond strength in the Silorane based composite resin.^(67,87)

Moreover, Tezvergil-Mutluay *et al*, concluded in their study that, the cationic initiators are generally prone to inactivation in the presence of water which might affect the bonding and mechanical properties in the oral cavity. In their study, the shear bond strengths decreased from 26.7MPa to 22.4MPa within 5 minutes which also suggests a potential of even greater deterioration of bond

strengths after a longer period of storage.⁽⁸⁵⁾ In addition, the presence of water can disrupt the polymerization since its nucleophilicity allows it to compete with monomer for the oxonium ions.⁽⁸³⁾

It was hypothesized that Silorane resin, when stored wet, would retain its mechanical properties because of its hydrophobicity in an aqueous environment. However, within the limitations of the current study, this hypothesis was not accepted with specimens inserted in layers, as the statistical evaluation showed a significant reduction of mean SBS values of Silorane specimens at wet storage conditions ($p < 0.05$).

Apart from recording lower SBS values (21.90 MPa) after one week storing wet for Silorane resin inserted in layers, the null hypothesis that insertion with bulk technique in the case of Silorane based resin, is the option of choice, this is owing to the photo initiation radicals in cationic polymerization are insensitive to oxygen is partially accepted. Silorane-based resin specimens inserted in bulk, resulted in the mean SBS values that were comparable to those obtained with methacrylate-based resin (Hybrid, TEC) with both insertion techniques and storage conditions, and with the mean SBS values of Nanofilled resin with bulk insertion and wet conditions.

Moreover, SBS values of Silorane-based resin specimens inserted in bulk at all storage conditions, were similar to those obtained with Silorane resin specimens inserted in layers at dry storage condition. These findings support the clinical reliability of insertion of the Silorane resin (Feltik Silorane) in bulk with a cavity deeper than 2 mm depth.

Furthermore, study made by Tavenger *et al*, presented results came in accordance with our findings, they reported that there is no significant statistical difference between one layer/bulk and double-layer/incremental techniques. They also reported that when using silorane-based composite system, the

configuration of the cavity design and polymerization process via light-curing technique, were as effective on bond stability as when a dimethacrylate-based composite system was used.⁽⁸⁸⁾

Moreover, in a study completed by Bijelic-Donova *et al*, the oxygen inhibition layer was optically observed for both dimethacrylate and silorane-based composite. They reported that the polymerization of silorane-based composite involves two stages: an initial free-radical phase and a cationic phase. The radical concentration for silorane is significantly reduced (but not completely eliminated), which consequently leads to the formation of a remarkably thinner oxygen inhibition layer on the surface of the silorane based composite than observed with methacrylate-based resin.⁽¹⁰⁴⁾

Furthermore, Silorane specimens showed the highest present of adhesive failure (33.3%) and lowest present of cohesion failure (8.3%) among tested composite resins. At the same time, Silorane's percentage of mixed mode failure (58.3%) was slightly higher than Hybride resin (52.8%) and slightly lower than Nanofilled resin (63.9%).

As known that, the bond strength of methacrylate based resin is directly influenced by their mechanical properties, which in turn, depends on the filler content, filler particle size and material chemistry.⁽¹⁰⁵⁾

In the present study, the mean SBS of the two MBCs, and Nanofilled and Hybride composite resins inserted with incremental technique and stored for one week in wet conditions (22.48, 28.21 MPa respectively), showed an insignificant minor increase in mean SBS values from those specimens stored in dry conditions for the same period (21.35, 27.29 MPa respectively) with similar cohesive failure percentage (22.2 %).

Moreover, as the main differences in their composition lies in the type of filler particles used, Filtek Supreme (Nanofilled) composed of Nanoscale zirconia

and silica particles, some of which are aggregated in 'Nanoclusters', these aggregated Nanofillers in the matrix may increase crack propagation under applied load, slippage may exist within the nanoclusters. The surface area for the interaction between nanofillers and matrix decreases and fractures in the aggregate sites initiated.⁽¹⁰⁶⁾

On the other hand, Tetric EvoCeram (Hybride) contains barium glass, ytterbium trifluoride, mixed oxide and silicon dioxide filler particles. These differences in the types of filler particles in the two materials did seem to have a significant effect on the shear bond strength values.⁽⁹⁷⁾

Furthermore, from the results of this study, the insignificant difference of the effect of storage time (one week) and conditions on shear bond strength of methacrylate resin groups inserted in layers, may be due to the fact that one week is not enough time to determine the durability of the attained bond strength and further studies should be made with long-term data of bond testing.

It is worth mentioning that the fractured surfaces were examined under a light microscope with a magnification of 20 and 40 to classify the failure mode into adhesive, cohesive and mixed failures. The adhesive failure was the fracture at the interface between the over acrylic block part (within Teflon over matrix) of composite specimens and the submersible in acrylic block part of composite. Where cohesive failure occurs either in the resin composite submersible in the acrylic block resin composite, or in the over acrylic block part. Mixed failure includes both adhesive and cohesive failures.

Chapter VI

Conclusion and further studies.

6.1. Conclusion:

Within the limitations of the current *in-vitro* investigation, a number of conclusions can be reached:

- Regardless of the presence or absence of an oxygen inhibited layer, Silorane- based resin composite inserted in layers and stored dry showed interlayer SBS values comparable to that Hybride based resin (TEC) inserted in layers at both storage conditions. As for Silorane specimens stored wet, SBS values showed an insignificant difference to that obtained with Nanofilled-based resin inserted in layer at both condition.
- For Silorane-based resin specimens inserted in bulk at both storage conditions, SBS values showed an insignificant difference to those obtained with Hybrid based resin (TEC) inserted in bulk at both storage conditions and Nanofilled based resin (Feltik supreme) inserted in bulk and stored wet.
- SBS values of Silorane-based resin specimens inserted in bulk in all storage conditions were insignificantly different to those obtained with Silorane resin specimens inserted in layers at dry storage condition and higher than the results obtained with specimens inserted in layers and stored wet. This finding supports the clinical reliability of insertion of the Silorane resin (Feltik Silorane) in bulk with a cavity deeper than 2 mm depth.
- Storage conditions insignificantly affected the SBS of Silorane-based resin specimens inserted in bulk, and adversely affected the SBS of Silorane-based resin specimens inserted in layers.
- The highest and the lowest mean shear bond strength values were recorded with Nanofilled resin composite specimens.
- Storage conditions and insertion techniques affected the SBS of Methacrylate (Hybride) based resin insignificantly.

6.2. Further studies:

- Despite specimens aging in distilled water can offer an approximate realistic *in vitro* scenario, the findings may not exactly mimic the intraoral behavior of composite restorations. This is because the *in vitro* results of shear bond strength testing do not always correlate to the clinical performance and longevity of the same materials. This may also be due to the large variety of clinical factors that influence material longevity and the difficulty in replicating all of them in vitro. Should an *in vivo* study be conducted, a randomized clinical trial may be a more effective method of evaluating the effectiveness of different insertion technique.
- The insignificant difference in the effect of the storage time (one week) on shear bond strength results of some tested composite groups, may be due to the fact that one week is not enough time to determine the durability of the attained bond strength. Thus further shear bond strength studies should be carried out with increased storage time, as a longer storage period may result in greater statistical significance.

Reference

References:

1. Bowen. Properties of a silica-reinforced polymer for dental restorations. J Am Dent Assoc. 1963;66(1):57–64.
2. SÖDERHOLM K-J, Mariotti A. BIS-GMA-based resins in dentistry: are they safe? J Am Dent Assoc. 1999;130(2):201–9.
3. AlShaafi MM. Factors affecting polymerization of resin-based composites: A literature review. Saudi Dent J. 2017;29(2):48–58.
4. Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review. Dent Mater. 2005;21(10):962–70.
5. Zakir M, Al Kheraif AAA, Asif M, *et al.* A comparison of the mechanical properties of a modified silorane based dental composite with those of commercially available composite material. Dent Mater. 2013;29(4):e53–9.
6. Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. Dent Mater. 2005;21(1):68–74.
7. Deliperi S, Bardwell DN. An alternative method to reduce polymerization shrinkage in direct posterior composite restorations. J Am Dent Assoc. 2002;133(10):1387–98.
8. Pfeifer CSC, Braga RR, Cardoso PEC. Influence of cavity dimensions, insertion technique and adhesive system on microleakage of Class V restorations. J Am Dent Assoc. 2006;137(2):197–202.
9. Gauthier MA, Stangel I, Ellis TH, *et al.* Oxygen inhibition in dental resins. J Dent Res. 2005;84(8):725–9.

10. Eick JD, Smith RE, Pinzino CS, *et al.* Stability of silorane dental monomers in aqueous systems. *J Dent.* 2006;34(6):405–10.
11. Ilie N, Hickel R. Silorane-based dental composite: behavior and abilities. *Dent Mater J.* 2006;25(3):445–54.
12. Ilie N, Jelen E, Clementino-Luedemann T, Hickel R. Low-shrinkage composite for dental application. *Dent Mater J.* 2007;26(2):149–55.
13. Yamazaki PC V, Bedran-Russo AKB, Pereira PNR, *et al.* Microleakage evaluation of a new low-shrinkage composite restorative material. *Oper Dent.* 2006;31(6):670–6.
14. Burke FJT, Crisp RJ, James A, *et al.* Two year clinical evaluation of a low-shrink resin composite material in UK general dental practices. *Dent Mater.* 2011;27(7):622–30.
15. Baum L, Phillips RW, Lund MR. *Textbook of operative dentistry.* 2nd ed. edited by Saunders;1985:204-210.
16. Sakaguchi RL, Powers JM. *Craig's restorative dental materials.* 12th ed. edited by Mosby Elsevier;2006:190-199.
17. Van Noort R. *Introduction to Dental Materials.* 3rd ed. edited by Mosby Elsevier; 2007:99-105.
18. Zimmerli B, Strub M, Jeger F, *et al.* Composite materials: composition, properties and clinical applications. A literature review. *Schweizer Monatsschrift für Zahnmedizin= Rev Mens suisse d'odonto-stomatologie= Riv Mens Svizz di Odontol e Stomatol.* 2010;120(11):972–86.
19. García AH, Lozano MAM, Vila JC, *et al.* Composite resins. A review of the materials and clinical indications. *Med Oral Patol Oral Cir Bucal.* 2006;11(2):E215-220.

20. Anusavice KJ. Phillips' science of dental materials. 7th ed. edited by Mosby Elsevier; 2003:167-168, 406-407.
21. Gajewski VES, Pfeifer CS, Fróes-Salgado NRG, *et al.* Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. *Braz Dent J.* 2012;23(5):508–14.
22. Al-Ahdal K, Silikas N, Watts DC. Rheological properties of resin composites according to variations in composition and temperature. *Dent Mater.* 2014;30(5):517–24.
23. Talib R. Dental composites: a review. *J Nihon Univ Sch Dent.* 1993;35(3):161–70.
24. Mitra SB. Nanoparticles for dental materials: synthesis, analysis, and applications. In: *Emerging Nanotechnologies in Dentistry.* Elsevier. 2018;2(2):17–39.
25. Sturdevant CM, Roberson TM, Heyman HO, *et al.* The art science of operative dentistry 3rd ed. edited by St Louis, Toronto, Pricet CV Mosby Co. 1995;504–12.
26. Patodiya A, Hegde MN. Dental composites: past, present and future. *Natl J Commun Med.* 2012;3(4):754–6.
27. Antonucci JM, Dickens SH, Fowler BO, *et al.* Chemistry of silanes: interfaces in dental polymers and composites. *J Res Natl Inst Stand Technol.* 2005;110(5):541-558.
28. Ferracane JL. Resin composite—state of the art. *Dent Mater.* 2011;27(1):29–38.
29. Singh S, Rajkumar B, Gupta V, Bhatt A. Current photo-initiators in dental materials. *Int J Appl Dent Sci.* 2017;3(1):17–20.

30. Ghivari S, Chandak M, Manvar N. Role of oxygen inhibited layer on shear bond strength of composites. *J Conserv Dent JCD*. 2010;13(1):39.
31. McCabe JF. *Applied dental materials*. 7th ed. edited by Blackwell Scientific Publications;1992:145-147.
32. Habib ANA, Waly GH. The degree of conversion and class II cavity microleakage of different bulk fill composites placed with different restorative techniques. *Futur Dent J*. 2018;4(2):231–8.
33. Yatabe M, Seki H, Shirasu N, *et al*. Effect of the reducing agent on the oxygen-inhibited layer of the cross-linked reline material. *J Oral Rehabil*. 2001;28(2):180–5.
34. Dall'Oca S, Papacchini F, Goracci C, *et al*. Effect of oxygen inhibition on composite repair strength over time. *J Biomed Mater Res Part B Appl Biomater An Off J Soc Biomater Japanese Soc Biomater Aust Soc Biomater Korean Soc Biomater*. 2007;81(2):493–8.
35. Eliades GC, Caputo AA. The strength of layering technique in visible light-cured composites. *J Prosthet Dent*. 1989;61(1):31–8.
36. Sehgal A, Rao YM, Joshua M, *et al*. Evaluation of the effects of the oxygen-inhibited layer on shear bond strength of two resin composites. *J Conserv Dent JCD*. 2008;11(4):159.
37. Shawkat ES, Shortall AC, Addison O, *et al*. Oxygen inhibition and incremental layer bond strengths of resin composites. *Dent Mater*. 2009;25(11):1338–46.
38. Bayou S, Mouzali M, Lecamp L, *et al*. Photoinitiated polymerization of a dental formulation: 1. Influence of photoinitiating system, temperature and luminous intensity. *J Fundam Appl Sci*. 2017;9(2):685–95.

39. Rueggeberg FA, Margeson DH. The effect of oxygen inhibition on an unfilled/filled composite system. *J Dent Res.* 1990;69(10):1652–8.
40. Suh BI. Oxygen-inhibited layer in adhesion dentistry. *J Esthet Restor Dent.* 2004;16(5):316–23.
41. Finger WJ, Lee K-S, Podszun W. Monomers with low oxygen inhibition as enamel/dentin adhesives. *Dent Mater.* 1996;12(4):256–61.
42. Al Hmedat SJA-Z, Jaber ZA. Comparison of Shear Bond Strength of Composite to Composite Bond in Increment Technique (An in vitro study). *of.* 2017;3(5):1-6.
43. Ilie N, Hickel R. Resin composite restorative materials. *Aust Dent J.* 2011;56(1):59–66.
44. Ferracane JL. Current trends in dental composites. *Crit Rev Oral Biol Med.* 1995;6(4):302–18.
45. Lang BR, Jaarda M, WANG R. Filler particle size and composite resin classification systems. *J Oral Rehabil.* 1992;19(6):569–84.
46. Kim RJ-Y, Kim Y-J, Choi N-S, *et al.* Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites. *J Dent.* 2015;43(4):430–9.
47. Kuijs RH, Fennis WMM, Kreulen CM, *et al.* Does layering minimize shrinkage stresses in composite restorations? *J Dent Res.* 2003;82(12):967–71.
48. Sakaguchi RL, Peters M, Nelson SR, *et al.* Effects of polymerization contraction in composite restorations. *J Dent.* 1992;20(3):178–82.

49. de Melo Monteiro GQ, Montes MAJR, Rolim TV, *et al.* Alternative methods for determining shrinkage in restorative resin composites. *Dent Mater.* 2011;27(8):e176–85.
50. Cadenaro M, Biasotto M, Scuor N, *et al.* Assessment of polymerization contraction stress of three composite resins. *Dent Mater.* 2008;24(5):681–5.
51. Giachetti L, Scaminaci Russo D, Bambi C, *et al.* A review of polymerization shrinkage stress: current techniques for posterior direct resin restorations. *J Contemp Dent Pr.* 2006;7(4):79–88.
52. Condon JR, Ferracane JL. Assessing the effect of composite formulation on polymerization stress. *J Am Dent Assoc.* 2000;131(4):497–503.
53. Loguercio AD, Reis A, Ballester RY. Polymerization shrinkage: effects of constraint and filling technique in composite restorations. *Dent Mater.* 2004;20(3):236–43.
54. Davidson CL, Feilzer AJ. Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives. *J Dent.* 1997;25(6):435–40.
55. Stansbury JW, Trujillo-Lemon M, Lu H, *et al.* Conversion-dependent shrinkage stress and strain in dental resins and composites. *Dent Mater.* 2005;21(1):56–67.
56. Christensen RP, Palmer TM, Ploeger BJ, *et al.* Resin polymerization problems--are they caused by resin curing lights, resin formulations, or both? *Compend Contin Educ Dent NJ 1995) Suppl.* 1999;(25):S42-54.
57. Cakir D, Sergent R, Burgess JO. Polymerization shrinkage—A clinical review. *Insid Dent.* 2007;3(8):3–10.

58. AlQahtani MQ, Michaud PL, Price RB, *et al.* Effect of high irradiance on depth of cure of a conventional and a bulk fill resin-based composite. *Oper Dent.* 2015;40(6):662–72.
59. Tarle Z, Knezevic A, Demoli N, *et al.* Comparison of composite curing parameters: effects of light source and curing mode on conversion, temperature rise and polymerization shrinkage. *Oper Dent.* 2006;31(2):219–26.
60. Schneider LFJ, Pfeifer CS, Ferracane JL, *et al.* Curing efficiency of dental resin composites formulated with camphorquinone or trimethylbenzoyl-diphenyl-phosphine oxide. *Dent Mater.* 2012;28(4):392–7.
61. Meereis CTW, Münchow EA, da Rosa WL de O, *et al.* Polymerization shrinkage stress of resin-based dental materials: a systematic review and meta-analyses of composition strategies. *J Mech Behav Biomed Mater.* 2018;82:268–81.
62. Park J, Chang J, Ferracane J, *et al.* How should composite be layered to reduce shrinkage stress: incremental or bulk filling? *Dent Mater.* 2008;24(11):1501–5.
63. Donly KJ, Jensen ME. Posterior composite polymerization shrinkage in primary teeth: an in vitro comparison of three techniques. *Pediatr Dent.* 1986;8(3):209–12.
64. Rueggeberg FA, Ergle JW, Mettenburg DJ. Polymerization depths of contemporary light-curing units using microhardness. *J Esthet Restor Dent.* 2000;12(6):340–9.
65. Karacolak G, Boyacioglu H, Ferracane JL, *et al.* Influence of increment thickness on radiant energy and microhardness of bulk-fill resin composites. *Dent Mater J.* 2018;37(2):206–13.

66. Abbas G, Fleming GJP, Harrington E, *et al.* Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments. *J Dent.* 2003;31(6):437–44.
67. Peutzfeldt A, Asmussen E. Determinants of in vitro gap formation of resin composites. *J Dent.* 2004;32(2):109–15.
68. Ferracane JL. Placing dental composites—a stressful experience. *Oper Dent.* 2008;33(3):247–57.
69. Gonzalez-Lopez S, Lucena-Martin C, de Haro-Gasquet F, *et al.* Influence of different composite restoration techniques on cuspal deflection: an in vitro study. *Oper Dent.* 2004;29(6):656–60.
70. Kwon Y, Ferracane J, Lee I-B. Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites. *Dent Mater.* 2012;28(7):801–9.
71. Opdam NJM, Roeters FJM, Feilzer AJ, *et al.* Marginal integrity and postoperative sensitivity in Class 2 resin composite restorations in vivo. *J Dent.* 1998;26(7):555–62.
72. Nikolaenko SA, Lohbauer U, Roggendorf M, *et al.* Influence of c-factor and layering technique on microtensile bond strength to dentin. *Dent Mater.* 2004;20(6):579–85.
73. Jafarpour S, El-Badrawy W, Jazi HS, *et al.* Effect of composite insertion technique on cuspal deflection using an in vitro simulation model. *Oper Dent.* 2012;37(3):299–305.
74. Ferracane JL, Hilton TJ. Polymerization stress—is it clinically meaningful? *Dent Mater.* 2016;32(1):1–10.

75. Pereira RA, Araujo PA de, Castañeda-Espinosa JC, *et al.* Comparative analysis of the shrinkage stress of composite resins. *J Appl Oral Sci.* 2008;16(1):30–4.
76. Son S, Roh H-M, Hur B, Kwon Y-H, *et al.* The effect of resin thickness on polymerization characteristics of silorane-based composite resin. *Restor Dent Endod.* 2014;39(4):310–8.
77. Nicholson JW. Polyacid-modified composite resins (“compomers”) and their use in clinical dentistry. *Dent Mater.* 2007;23(5):615–22.
78. Guggenberger R, Weinmann W. Exploring beyond methacrylates. *Am J Dent.* 2000;13(Spec No):82D-84D.
79. Palin WM, Fleming GJP, Burke FJT, *et al.* The influence of short and medium-term water immersion on the hydrolytic stability of novel low-shrink dental composites. *Dent Mater.* 2005;21(9):852–63.
80. Duarte Jr S, Botta AC, Phark J-H, *et al.* Selected mechanical and physical properties and clinical application of a new low-shrinkage composite restoration. *Quintessence Int (Berl).* 2009; 40(8):631-8
81. Panahandeh N, Torabzadeh H, Naderi H, *et al.* Effect of water storage on flexural strength of silorane and methacrylate-based composite resins. *Restor Dent Endod.* 2017;42(4):309–15.
82. Maia RR, Reis RS, Moro AF V, *et al.* Properties evaluation of silorane, low-shrinkage, non-flowable and flowable resin-based composites in dentistry. *PeerJ.* 2015;3:e864.
83. Kaleem M, Satterthwaite JD, Watts DC. Effect of filler size and morphology on viscoelastic stability of resin-composites under dynamic loading. *J Mater Sci Mater Med.* 2012;23(3):623–7.

84. Ilie N, Hickel R. Macro-, micro-and nano-mechanical investigations on silorane and methacrylate-based composites. *Dent Mater.* 2009;25(6):810–9.
85. Tezvergil-Mutluay A, Lassila LVJ, Vallittu PK. Incremental layers bonding of silorane composite: the initial bonding properties. *J Dent.* 2008;36(7):560–3.
86. Hickel R, Brühaver K, Ilie N. Repair of restorations—criteria for decision making and clinical recommendations. *Dent Mater.* 2013;29(1):28–50.
87. Musa AH Al, Nahedh HNA Al. Incremental layer shear bond strength of low-shrinkage resin composites under different bonding conditions. *Oper Dent.* 2014;39(6):603–11.
88. Tavangar M, Davaloo RT, Darabi F, *et al.* A comparative evaluation of microleakage of two low-shrinkage composites with a conventional resin composite: an in vitro assessment. *J Dent.* 2016;17(1):55-61.
89. Vangelov L, Markova K, Miteva T. Application of Filtek Silorane-initial observations and prospective clinical trial for 12 months. *J IMAB.* 2010;16(4):58–62.
90. Rinastiti M, Özcan M, Siswomihardjo W, *et al.* Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. *J Dent.* 2010;38(1):29–38.
91. Price RB, Doyle G, Murphy D. Effects of composite thickness on the shear bond strength to dentin. *JOURNAL-CANADIAN Dent Assoc.* 2000;66(1):35–9.
92. Standard I. ISO 4049 polymer based filling, restorative and luting materials. *Int Organ Stand.* 2000;1:27.

93. ASTM-D5379. D5379: Standard test method for shear properties of composite materials by the V-notched beam method. Norm ASTM C273-00 Shear Prop Sandw Core Mater. 2000.
94. Gao B-T, Lin H, Zheng G, *et al.* Comparison between a silorane-based composite and methacrylate-based composites: shrinkage characteristics, thermal properties, gel point and vitrification point. *Dent Mater J.* 2012;31(1):76-85.
95. Sau CW, Oh GSY, Koh H, *et al.* Shear bond strength of repaired composite resins using a hybrid composite resin. *Oper Dent.* 1999;24:156–61.
96. McDonough WG, Antonucci JM, He J, *et al.* A microshear test to measure bond strengths of dentin–polymer interfaces. *Biomaterials.* 2002;23(17):3603–8.
97. Yesilyurt C, Yoldas O, Altintas SH, *et al.* Effects of food-simulating liquids on the mechanical properties of a silorane-based dental composite. *Dent Mater J.* 2009;28(3):362–7.
98. Cesar PF, Júnior WGM, Braga RR. Influence of shade and storage time on the flexural strength, flexural modulus, and hardness of composites used for indirect restorations. *J Prosthet Dent.* 2001;86(3):289–96.
99. Ferracane JL. Elution of leachable components from composites. *J Oral Rehabil.* 1994;21(4):441–52.
100. Asopa V, Suresh S, Khandelwal M, *et al.* A comparative evaluation of properties of zirconia reinforced high impact acrylic resin with that of high impact acrylic resin. *Saudi J Dent Res.* 2015;6(2):146–51.
101. Moharamzadeh K, Van Noort R, Brook IM, *et al.* HPLC analysis of components released from dental composites with different resin

- compositions using different extraction media. *J Mater Sci Mater Med.* 2007;18(1):133–7.
102. Patras M, Naka O, Doukoudakis S, *et al.* Management of provisional restorations' deficiencies: a literature review. *J Esthet Restor Dent.* 2012;24(1):26–38.
103. Papacchini F, Dall Oca S, Chieffi N, *et al.* Composite-to-composite microtensile bond strength in the repair of a microfilled hybrid resin: effect of surface treatment and oxygen inhibition. *J Adhes Dent.* 2007;9(1):25-31.
104. Bijelic-Donova J, Garoushi S, Lassila LVJ, *et al.* Oxygen inhibition layer of composite resins: effects of layer thickness and surface layer treatment on the interlayer bond strength. *Eur J Oral Sci.* 2015;123(1):53–60.
105. Ribeiro JCR, Gomes PN, Moysés MR, *et al.* Shear strength evaluation of composite–composite resin associations. *J Dent.* 2008;36(5):326–30.
106. Kundie F, Azhari CH, Muchtar A, *et al.* Effects of filler size on the mechanical properties of polymer-filled dental composites: a review of recent developments. *J Phys Sci.* 2018;29(1):141–65.

Appendices

Appendix 1

Raw data of shear bond strength from 108 composite samples:

Materials tested	Insertion technique	load at fracture (kg)	SBS (MPs)	Storage condition	Mode of failure
Filtek Silorane	Bulk	36.5	28.5	Wet	Mixed
		35.7	27.8		Mixed
		35.6	27.8		Mixed
		37.0	28.8		Cohesive
		32.6	25.4		Mixed
		35.4	27.6		Mixed
		32.0	24.9		Mixed
		33.8	26.3		Mixed
		30.0	23.43		Cohesive
		32.0	24.99	Dry	Mixed
		34.4	26.86		Mixed
		27.2	21.24		Mixed
		36.7	28.66		Adhesive
		40.5	31.63		Cohesive
		38.0	29.67		Mixed
		28.2	22.02		Mixed
		33.5	26.16		Mixed
		34.4	26.86		Mixed
	Layering	22.9	17.88	wet	Adhesive
		35.4	25.13		Mixed
		24.5	19.13		Adhesive
		25.0	19.52		Adhesive

		28.3	22.10		Adhesive
		37.2	29.05		Mixed
		29.3	22.85		Mixed
		27.1	21.16		Mixed
		26.0	20.30		Adhesive
		30.5	23.82	Dry	Mixed
		36.4	28.42		Adhesive
		35.2	27.49		Adhesive
		33.7	26.31		Mixed
		34.8	27.17		Mixed
		36.0	28.11		Adhesive
		31.5	24.6		Adhesive
		39.4	30.77		Adhesive
		39.4	30.77		Adhesive
Filtek Supreme Nanofilled	Bulk	32.5	25.38	Wet	Mixed
		40.4	31.55		Mixed
		36.0	28.11		Mixed
		32.0	24.99		Mixed
		36.4	28.42		Mixed
		34.8	27.17		Mixed
		32.6	25.48		Mixed
		33.0	25.77		Mixed
		34.1	26.63		Mixed
		40.0	31.24	Dry	Mixed
		34.0	26.55		F
		51.6	40.29		Mixed
		50.5	39.44		Mixed
		47.7	37.25		Mixed
		52.5	41.00		Cohesive

		44.2	34.50		F
		48.0	37.48		Mixed
		51.6	40.29		F
	Layering	29.5	23.03	Wet	Mixed
		26.5	20.69		Mixed
		33.5	26.16		Cohesive
		24.9	19.44		Mixed
		31.7	24.75		Cohesive
		28.1	21.94		Mixed
		32.6	25.46		Cohesive
		25.5	19.91		Mixed
		26.8	20.93		Mixed
		30.9	24.13	Dry	Mixed
		27.6	21.55		Mixed
		30.8	24.05		Cohesive
		23.0	25.77		F
		23.9	18.66		Mixed
		31.0	24.21		Adhesive
		35.5	27.72		Cohesive
		14.1	11.01		Cohesive
		29.3	22.88		F
Tetric EvoCeram Hydride	Bulk	37.7	29.44	Wet	Cohesive
		34.8	27.17		Mixed
		36.3	28.35		Mixed
		30.0	23.43		Mixed
		36.7	28.66		Cohesive
		34.3	26.78		Mixed
		32.9	25.69		Mixed
		32.6	25.46		Mixed

		36.1	28.19		Mixed
		35.0	27.33	Dry	Mixed
		38.0	29.67		Cohesive
		36.2	28.27		Mixed
		36.8	28.74		Mixed
		33.9	26.47		Mixed
		41.0	32.02		Cohesive
		33.2	25.92		Mixed
		33.0	25.77		Mixed
		30.0	23.43		Cohesive
	Layering	40.0	31.24	Wet	Adhesive
		34.3	26.78		Mixed
		33.0	25.77		Mixed
		45.0	35.14		Adhesive
		25.0	19.52		F
		32.7	25.53		F
		41.2	32.17		Cohesive
		36.0	28.11		Adhesive
		38.0	29.67		Cohesive
		31.7	24.75	Dry	Mixed
		38.0	29.67		adhesive
		35.9	28.03		F
		34.0	26.55		F
		32.1	25.07		Mixed
		29.0	22.64		Mixed
		38.1	29.75		Adhesive
		39.0	30.45		Cohesive
		36.7	28.66		Mixed

F = Failure and dislodged from the acrylic block after fracture.

Appendix 2:

الجمهورية العربية السورية اللجنة الشعبية الاشتراكية الصحية الوطني
إدارة اللجنة الشعبية للصحة والبيئة
بنغازي

مكتب الأمين

الرقم: 6.1424/6.1424
التاريخ: / /
الموافق: 2010 / 9 / 22

الأخ / مدير عام العيادة المركزية للأسنان .
بغداد

بالإشارة إلى رسالة الأخ / أمين اللجنة الشعبية كلية طب و جراحة الفم و الأسنان بنغازي
ذات رقم أشراري " 811 / ش المؤرخة بتاريخ 15 . 9 . 2010 ف بشأن منح الأخت / د . أسماء هلال العبيدي
إجازة وذلك لتمكينها من السفر إلى جمهورية مصر لغرض الدراسة ...
وبناءً على تأشيرة الأخ / الأمين ... نفيديكم بعدم التمساعة من منح المعطية الإجازة المطلوبة
وتأجيل التعادك معها إلى « أن رجوعها بعد إتمام البحث في جمهورية مصر العربية .
للتفضل بالاستسلام و اتخاذ ما يلزم من إجراء

رئيس اللجنة الشعبية للصحة والبيئة
بنغازي

مرعى علي بن هلال
رئيس وحدة شؤون اللجنة الشعبية للصحة والبيئة

شعبية بنغازي

الأخ / أ. د. أمين اللجنة الشعبية للصحة والبيئة بنغازي .
الأخ / مدير شؤون الإدارية و العلاقات
ملف الشؤون العامة
خ.م. بن هلال
قسم العلاقات العامة

هاتف : 2241121 - 2230371
بريد مصور : 2224294

Appendix 3:

قرش جنيه
١٢١٥

جامعة الإسكندرية
كلية طب الأسنان
وحدة اختبار مواد الأسنان

إيصال رقم
٢١٣

إسم الطبيب: أ. د. هلال هبيرة

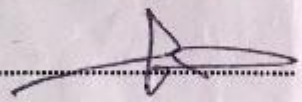
مبلغ وقدره: الف وثمانمائة وثمانون جنيهاً فقط

قيمة: ١١٨ كذا ليس عليه ١٠٨ كذا لا ١٠٨

١٣٥ ^{CD} ٣٥ * ١٠٨

١٢١٥
توقيع المستلم

التاريخ ١٧ / ١١ / ٢٠١٩



Arabic summary

دراسة تأثير طرق وضع الحشوات الرتنج منخفضة الانكماش (السيلوران) على معامل قوة الالتصاق

مقدمة من الطالب : أسماء هلال امراجع عبدالمولي

تحت اشراف : أ. د . احمد عبدالله الحجازي

الملخص

الهدف: تقييم قوة رابطة القص لمركب الراتنج منخفض الانكماش (السيلوران) باستخدام طرق ادراج (طبقات وطبقة واحدة) وتخزين (رطب وجاف) مختلفة, ومقارنتها مع مركبات الراتنج التقليدية (ميثاكريلات).

المواد والطرق: تم إعداد 108 عينات باستخدام قالب اسطواني مستخدم لاختبارات قوة رابطة القص، تم تصنيعها باستخدام ثلاث مواد راتنج مركبة؛منخفض الانكماش (سيلوران) واثنان من الميثاكريلات (تترك ايفو سيرام والسبريم)، مقسمة الى 12مجموعة وفقا لتقنية الإدراج وطرق التخزين (جاف او رطب) عند درجة حرارة + 37 درجة مئوية لمدة اسبوع واحد (ن=9/ مجموعة). تم قياس قوة ارتباط القص بسرعة 5مم/دقيقة. قدرت قيمة بيانات قوة القص بالميقاباسكال وقدمت جميعها لتحليل التباين أحادي الاتجاه و اختبار التوكي بحيث $p=0.05$. تم تقييم الأسطح المكسورة لعينات الاختيار بصريا باستخدام المجهر الضوئي بتكبير 20 و 40 مرة.تم تصنيف الأسطح اما لاصقة او متماسكة او مختلطة.

النتائج: أظهرت عينات السوبريم التي أدرجت مرة واحده وخزنت جافة اعلى قيم لقوة القص (36.45)، بينما كانت عينات السيلوران والتترك ايفوسيرام اقل في جميع مجموعاتها (26.45,26.78 و 27.50 ميقاباسكال للسيلوران و 27.29,27.02,27.51,و28.21 ميقاباسكال لعينات التترك ايفو سيرام)، باستثناء عينات السيلوران المدرجة في طبقات والتي انخفضت بدرجة ملحوظة نتيجة تخزينها فالماء(21.90) والتي كانت قريبة الى حد ما من عينات السوبريم المدرجة في طبقات والمخزنة جافة والتي أظهرت ادنى قوة رابطة قص.

الخلاصة: لم تؤثر تقنية الإدراج وكذلك حالة التخزين بشكل كبير على قوة رابطة القص لعينات السيلوران مقارنة مع عينات مركب الميثاكريلات (تترك ايفوسيرام) ،باستثناء العينات المدرجة فى طبقات والمخزنة فى الماء ، كان هناك انخفاض ملحوظ فى قيم قوة رابطة القص.



دراسة تأثير طرق وضع الحشوات الرتنج منخفضة الانكماش (السيلوران) على معامل قوة الالتصاق.

قدمت من :

أسماء هلال امراجع عبدالمولى العبيدي

تحت إشراف :

أ.د. احمد عبدالله الحجازي

قدمت هذه الرسالة استكمالاً لمتطلبات الحصول على درجة الماجستير في تخصص

خواص مواد الأسنان

كلية طب وجراحة الفم و الأسنان

جامعة بنغازي

أغسطس 2019