

Physical Properties of Dental Resin Nanocomposites

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Abstract

Objectives: (1) To evaluate and compare the flexural strength, flexural modulus, compressive strength, surface roughness, and gloss for two nanohybrid composites (Herculite XRV Ultra, and Tetric Evo Ceram) and two microhybrid composites (Filtek Z250, and Venus). (2) To evaluate the differences in surface roughness and gloss between the materials. (3) To evaluate the effect of polishing and toothbrushing abrasive on surface roughness and gloss. (4) To assess the relationship between 2D surface roughness and 3D surface roughness parameters.

Materials and methods: Compressive strength, Flexural strength, and Flexural Modulus of each material were determined using a universal testing machine (each test $n=10$). Flexural strength and flexural modulus were evaluated by using a three-point bending test. The results were statistically analyzed, with $p \leq 0.05$. Surface roughness and gloss of each material were evaluated by a 2D contact stylus profilometer: before polishing, after polishing, and after abrasive toothbrushing. Three surface roughness parameter were used in this study (R_a , R_t , R_{sm}). The results were analysed to evaluate the effect of polishing and toothbrushing abrasive by t-test for paired data and Oneway (ANOVA) test, followed by Tukey's *post hoc* test, for multiple comparisons. 3D surface roughness measurements (S_a , S_t , S_{ds}) were recorded using 3D non contact stylus profilometer. 3D surface roughness (S_a) and 2D surface roughness (R_a) for all tested materials were plotted to show the relationship between S_a and R_a after polishing and after brushing.

Results: For the mechanical tests, Filtek Z250 exhibited the highest flexural strength (136.71 MPa), flexural modulus (7.9 GPa), and compressive strength (405.33 MPa) among all materials tested, while Tetric Evo Ceram showed the lowest mean values for compressive strength (237.74 MPa) and flexural strength (83.75 MPa).

For Surface roughness after polishing, R_a , R_t , and R_{sm} of the materials did not differ significantly from each other, whereas after brushing there were significant differences between the materials. Tetric Evo Ceram exhibited the rougher surface after brushing. The gloss results showed significant differences in gloss between the materials tested after polishing and after brushing. Herculite XRV exhibited the highest surface gloss after polishing (74.85) and was the best at retaining the gloss after brushing, whereas Venus recorded the lowest result for gloss after polishing (52.28). However, Filtek Z250 had the lowest gloss retention after brushing. Also the results showed a positive correlation between 3D surface roughness (S_a) and 2D surface roughness (R_a) for all materials tested after polishing: $r^2=0.9$ and after brushing: $r^2=1$.

Conclusion: While nanohybrid composites may have certain advantage, such as higher gloss, over microhybrid composite resin, their mechanical properties were not superior to those of the microhybrid composites which were tested.

Declaration

I declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.

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2011

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To Memory of My Father

Chapter 1

Introduction

1. Introduction:

Over the last 40 years, the routine use of dental amalgam is gradually decreasing due to poor aesthetics of amalgam especially for anterior restoration, mercury toxicity, and environmental consideration arising from mercury disposal, potential dental fracture, secondary caries, and marginal leakage. Dental composites are among the synthetic resins used as adhesives or restorative material in dentistry and now represent general alternation to dental amalgam. However, composites have limited uses because of low durability and strength {Leinfelder *et al.* 1980; Abell *et al.* 1983; Lacy, 1987; Jordan and Suzuki 1991}.

Longevity and survival studies in posterior teeth continue to show that amalgam has better track record than composites. The survival rate over all for composites in permanent teeth after 7 years was 67.4% compared with 94.5% for amalgam restoration {Bernardo, *et al.* 2007}. Also flexural strength ranges from 80 to 120 MPa for dental composites which can fulfil the needs of small restoration but cannot survive large stress bearing restoration while dental amalgams have flexural strength over 400 MPa {Corbin and Kohn, 1994; Berry, *et al.* 1994}.

Many attempts have been undertaken to improve the clinical performance of dental resin composites since their development {Moszner and Salz 2001}. Investigations on the filler content centred on particle size, loading, silanization {Ikejima, *et al.* 2003} and formation of new particles {Ruddell, *et al.* 2002; Xu, *et al.* 2002}, While examination on the resin matrix is mainly focused on the formation of new monomers {Taylor, *et al.* 1998; Chung, *et al.* 2002; Atai, *et al.* 2004}. Such analyses are of great significance as the physical properties of dental composites rely greatly on the particle size and filler volume. The hardness, compressive strength, elastic modulus and flexural strength increase while the polymerization shrinkage decrease as filler volume fraction increase {Ikejima, *et al.* 2003}.

In the last few years the nanotechnology has played as an important role in improving the clinical performance of dental resin composites. Nanotechnology deals with chemical and physical methods to produce nanoscale operational materials which ranging in size from 0.1 to 100 nanometers {Kirk, *et al.* 1991}. Nowadays, nanotechnology has become the most considerably stimulated discipline in technology {Beun, *et al.* 2007}. Nanomaterial includes nanoparticles, nanocluster, nanocrystals, nanotubes, nanofiber, nanowire, nanorod, etc. Until now, numerous top-down and

bottom-up manufacturing approaches are available to synthesize nanomaterial {Beun, *et al*, 2007}. The intense interest in using nanomaterial's come from the idea that they may be used to manipulate the structure of materials to provide dramatic improvement in mechanical, physical, chemical, and optical properties {Terry, 2004}. With the use of nanomaterials striking enhancements in optical, chemical, physical and mechanical properties may be attained {Terry, 2004}. A large amount of examinations is being dedicated to the development of nanocomposites of distinct kinds for several programs, involving high performance coatings, biomedical systems, structural materials, catalysts, photonics and electronics.

In dentistry, posterior restoration class I and II need composites that show high mechanical properties, whereas anterior restoration require composites with superior aesthetics. The resin composite that offers all the requirements of both posterior and anterior restoration has not emerged yet. Nanotechnology has great impact on restorative dentistry by offering refinements to already resin based composite system {Terry, 2004; Beun, *et al*, 2007}. and the novel nanocomposite expected to be useful for all posterior and anterior restorative applications.

1.1 Historical background

As early as 1871, dental technology produced the first tooth coloured material, silicate cement {Federick, 1987}. Although silicate cement had the advantage of high fluoride release, the high solubility in oral fluids affects the longevity of restoration. Silicate cement remained in widespread use until direct filling methyl methacrylate resins were introduced in to United States in 1947 and came in general use by 1949. These resins had low solubility which enabled them to overcome the silicate cement, but their shrinkage and marginal leakage was high {Schulein, 2005}.

In 1955 the introduction of acid etch technique by Michael Buonocore has improved the quality of composite resins restoration by improving the adhesion of acrylic resins to surface of enamel by orthophosphoric acid {Buonocore, 1955}.

The use of acrylic resins and silicate cements came to an end within about two years after the introduction of composite resins {Minguez, *et al*, 2003}. In 1962 Bowen introduced BisGMA monomer in attempt to improve the physical properties of acrylic resins. However, this monomer is extremely viscous due to the presence of hydrogen

bonding. BisGMA forms the basis of present day composite resins as it limited shrinkage and fracture resistance {García, 2006}.

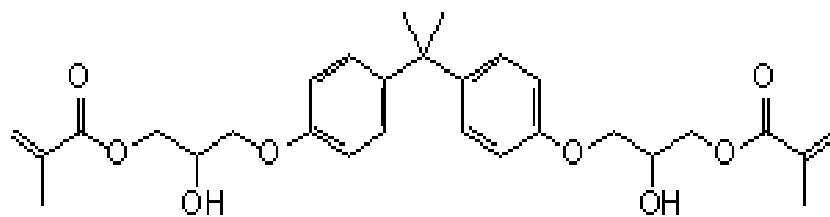
A notable event in evolution of resin based composites was the introduction of Ultra violet system in 1973 and by this system the anterior teeth fracture could be restored quite aesthetically and conveniently {García, 2006}. This system remained popular for many of years until 1978 when the visible light curing composite resins offered the advantages of no UV hazards, faster sitting, and better colour stability. Microfilled resin came in to use around 1977 provided filling with high polishable and stain resistant when used in conjunction with the acid etch technique {Schulein, 2005}. By 1980, hybrid and microhybrid resin were used extensively in cosmetic procedures. Resin based composites have continued to evolve to offer improved physical properties and esthetics and nowadays nanocomposites resin have come in to widespread use for the restoration of cavities in the posterior teeth.

1.2 Composition of dental resin composite

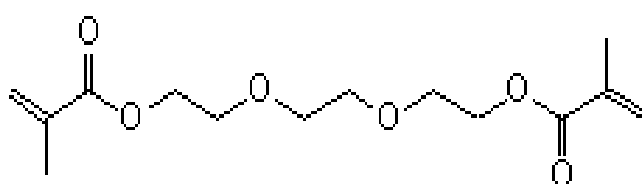
Dental resin composites basically derived from three chemical different materials: organic matrix or organic phase, filler or disperse phase, and an organosilane which is used as a coupling agent to bond the filler particles to the organic matrix {Kahler, *et al*, 2008}.

1.2.1 Matrix organic phase

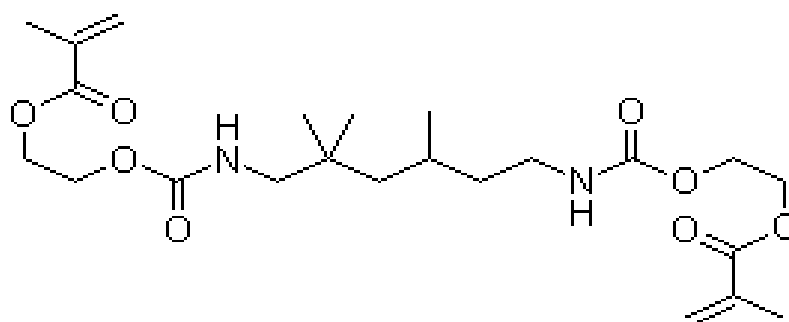
The organic matrix is generally dimethacrylate monomer (Fig 1.1) such as BisGMA, glycol dimethacrylate, urethane dimethacrylate (UDMA), triethyleneglycol dimethacrylate (TEGDMA), urethane dimethacrylateethoxylated bisphenol-A-dimethacrylate (Bis-EMA), decanediol dimethacrylate (D₃MA). In present day the most commonly used as organic phase (monomer) is BisGMA {Chen, 2010}. The advantages of using BisGMA over other monomers are less shrinkage, higher modulus and reduce toxicity due to its lower volatility and diffusivity into tissue. Although, BisGMA possesses high strength and hardness, the drawback of this monomer is its high viscosity, because of hydrogen bonding interaction that occur between hydroxyl groups, which limits the incorporation of inorganic fillers and hence a low degree of conversion {Korichi, *et al*, 2009}. Thus BisGMA diluted with other low-viscosity monomer such as trimethyleneglycoldimethacrylate (TEGDMA) {Chen, *et al*, 2010}.



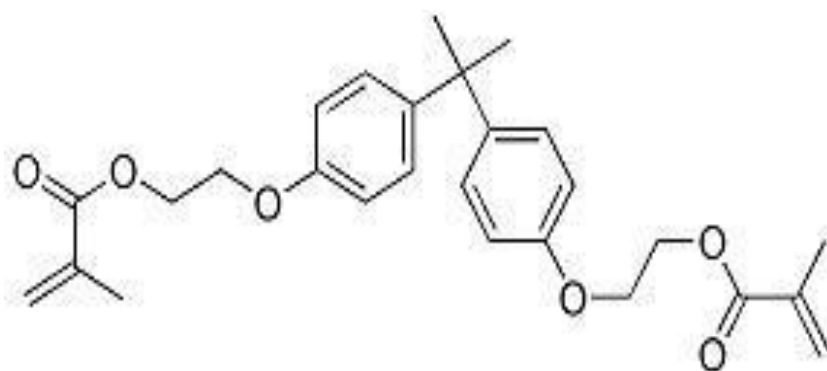
BisGMA



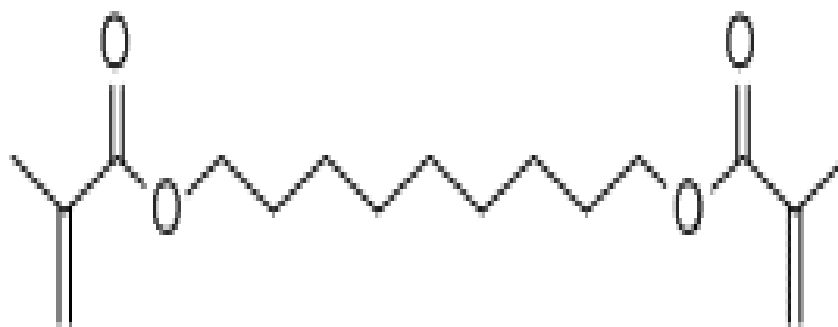
TEGDMA



UDMA



Bis-EMA



D₃MA

Fig 1.1 The chemical formula of the most commercial dental monomer used in dental composites

1.2.2 Filler disperse phase

The most current composites are filled with silicate particles based on oxides of barium, strontium, Zinc, aluminium or zirconium. The concentration rate of filler is generally 70%-80% by Weight. The particle filler size is in the range from 0.04 to 85 μm {Tanimoto, *et al*, 2006}. The primary purpose of the filler particles is to increase the strength of composite and to decrease the amount of matrix material, resulting in increased hardness, decrease wear and reduction in polymerization shrinkage {Azzopardi, *et al*, 2009}. The filler content, filler size, morphology, and the distribution of filler particles influence the physical and mechanical properties of composite resin and many studies reported the relation between filler and flexural strength, compressive strength, diametral tensile strength, shear punch strength, fracture toughness, hardness, wear, shrinkage stress and thermal expansion {Kahler, 2008; Lu, *et al*, 2006; Turssi, *et al*, 2005}.

1.2.3 Silane coupling agents

To obtain good mechanical properties in dental composites, strong covalent bond between inorganic fillers and the organic matrix is essential Bonding of the two phases is achieved by coating the fillers with a silane coupling agent that has functional groups to link the filler and the matrix chemically. A typical coupling agent is 3-methacryloxypropyltrimethoxysilane (MPTS) (Fig 1.2) One end of the molecule can be bonded to the hydroxyl groups of silica particles, and the other end is capable of copolymerizing into the polymer matrix {Chen, 2010}.

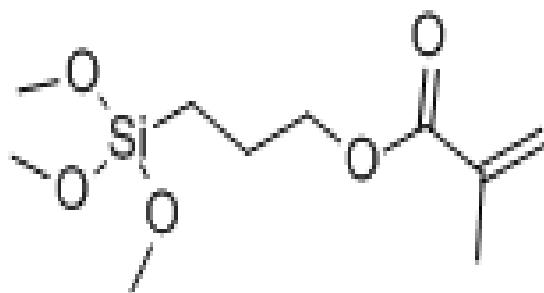


Fig 1.2, MPTS chemical formula

1.2.4 Other Ingredients {Azzopardi,*et al*, 2009}:

1.2.4.1 Polymerization Initiators: for chemically-activated resin composites, benzoyl peroxide and tertiary amines which serve as the source of free radicals.

For light-activated resin composites, a diketone photoactivator is used, such as camphoroquinone.

1.2.4.2 Polymerization Inhibitors: Because dimethacrylate monomers can polymerize spontaneously under normal storage conditions, inhibitors such as the monomethyl ether hydroquinone are added to solve this problem.

1.2.4.3 Ultraviolet Radiation Absorbers: These are added to improve colour stability by absorbing electromagnetic radiation that can cause discoloration. The most commonly used absorber is 2-hydroxy-4-methoxy benzophenone.

1.3 Classification of dental resin composites

The composite resins have been classified in different ways, depending on their composition. The most popular classification and still valid is Lutz and Philips (1983) classification which is based on the particle size of inorganic filler {Lutz, and Philips, 1983}. According to this classification composite resins are divided into three main groups: macrofilled, microfilled, and hybrid composite.

Also Willems *et al.* published a more detailed classification based on a number of parameters, the percentage of inorganic filler, the size of main particles, surface roughness and compressive strength {Willems, *et al*, 1992} (Table 1.1).

A simpler classification system has been described by Bayne et al in 1994 {Bayne, *et al*, 1994}. The three popular types of composites in this system are shown in Table 1.2.

Composite Type	Filler
Densified composites - Midway-filled - Ultrafine - Fine - CoMPact-filled >60% by volume - Ultrafine - Fine	< 60% by volume Particles < 3 μm Particle > 3 μm > 60% by volume Particles < 3 μm Particles > 3 μm
Microfine composites - Homogeneous - Heterogeneous	Average particle size = 0,04 μm
Miscellaneous composites	Blends of densified and microfine composites
Traditional composites	Equivalent to what are termed macrofill composites in other classifications
Fiber-reinforced composites	Industrial-use composites

Table 1.1 Classification of composites (Willems, *et al*, 1993)

Type of composites	Average of particle size
Microfills	0.01-0.1 μm
Minifills	0.1-1.0 μm
Midifills	1.0-10.0 μm

Table 1.2: Classification of composites resin according to Bayne *et al* in 1994

According to Lutz and Philips (1983) dental composites can be classified as the following:

1.3.1 Macrofilled resin composites

Macrofilled resin composites had inorganic particle filler with a size of 10-40 μm and their disadvantages are poor finishing and relatively high wear. The most used fillers in these composites are 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. Barium and strontium glass particles are radiopaque, but are less stable than quartz {Lindberg, 2005}.

1.3.2 Microfilled resin composites

Microfilled Resin composites were introduced to satisfy the need for polishable composite. These materials contain a very fine particle size of colloidal silica that ranges between 0.01-0.05 μm . Nevertheless, the very large surface area of the particles considerably limits the volume of filler that can be incorporated. Compared to macrofilled resin composites, the microfilled have lower mechanical properties due to the large volume of resin {Lindberg, 2005}.

1.3.3 Hybrid resin composites

Hybrid resin composites were introduced to solve the mechanical and the shrinkage problems. 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 . Hybrid composites are therefore ideal for use as universal composites.

1.3.4 Modern hybrid composites

Modern hybrid composites contain reduced submicron fillers. These composites are supposed to combine the advantages of macrofilled and microfilled composites, but they do not have the final finish and translucency of microfilled resin composites. {Lindberg, 2005}

1.3.5 Nano-composites

Nano-composites are recent development on the market. They contain filler particles with sizes less than 10 nm (0.01 μm) and are claimed to provide increased aesthetics, strength and durability {Lindberg, 2005}.

Nanocomposites are available as nanohybrid types which contain milled glass fillers and discrete nanoparticles (40–50 nm) and as nanofill types, containing both nano-sized filler particles, called nanomers and agglomerations of these particles described as “nanoclusters” {Mitra, *et al*, 2003}. The nanoclusters provide a distinct reinforcing mechanism compared with the microfill or nanohybrid systems resulting in significant improvements to the strength and reliability {Curtis, *et al*, 2009}.

Composite type	Filler size (µm)	Filler material
Macrofilled	10-40	Quartz or glass
Microfilled	0.01-0.1	Colloidal silica
Hybrid	15-20 and 0.01-0.05	Glass and colloidal silica
Modern Hybrid	0.5-1 and 0.01-0.05	Glass, Zirconia and colloidal silica
Nanofiller	< 0.01 (10 nm)	Silica or Zirconia

Table 1.3 Filler sizes and materials in dental composite materials

Composites also can be classified according to their method of activation

- (1) Chemically activated composites.
- (2) Light activated composites.
- (3) Heat-cured composites.
- (4) Dual-cured composites.

1.4 Properties of dental composites resin

1.4.1 Handling and stickiness;

The handling properties of dental resin composite play an important part in the clinical use.

Resin based composites have many different handling characters, such as packability, flow, thixotropy, viscosity and shape stability. While many refinements have been made in resin based composites two handling properties have not been achieved until now: non stickiness and fluid inject-ability {Bayne, et al, 1998}.

Lee et al in 2006 found that there were connection between the viscosity of the material and stickiness, the more viscous a material is, the less sticky is it. Also they found that the viscosity of the material is exponential increase with the increase of the percentage of filler volume. In addition they found the viscosity of composites decreased in correspondence to high temperature {Lee, et al, 2006}.

Another problem with dental composite related to stickiness of composites which are porosities and voids in restorations. The risk of voids and porosities increases when the material sticks to filling instruments as the air will entrap {Opdam, *et al*, 1996}.

In study conducted by Ertl et al to evaluate the stickiness of dental resin composite material they found that different resin composites material differ significantly in stickiness. Also stickiness increases with increase temperature and resin composite stick better to dentin than to steel or bonded dentin {Ertl, et al 2010}.

1.4.2 Polymerization shrinkage;

Full polymerization of material is determined by the degree of conversion of monomer into polymers. There are many factors that affect the polymerization process and degree of conversion which are:

- Curing time: it depends on; resin shade, light intensity, box depth, resin thickness, curing through tooth structure and composites filling.
- Temperature: at room temperature composites cure more completely and rapidly.
- Thickness of the resins: Maximum thickness 1-2 mm.
- Type of filler: curing of microfine composites is more difficult than heavily loaded composites.
- Distance between light and resins: Optimum distance less than 1 mm, with light positioned 90 degrees from the composites surface.
- Polymerization shrinkage which depends on the amount of organic phase {Chen, 2006}.

The polymerization shrinkage leads to gaps at the interface between resin and tooth causing leakage, food, fluid, saliva, residue and microorganism trapped in the gap leading to decay of teeth and damage of the enamel which is the major problem in current restorative and aesthetic dentistry{Chen, 2006}. The potential effect of polymerization shrinkage is summarized as the following:

- Enamel cracks.
- Marginal degradation.
- De-bonding of tooth-composite interface.
- Postoperative sensitivity.

The factors that can affect the shrinkage are the molecular weight of the monomer system, inorganic filler content, and the degree of conversion of the monomer system {Kleverlaan, and Feilzer, 2005}. Composites polymerization always involves a degree of shrinkage, depending on its monomer for this, to reduce this undesirable effect dental industry tested a great variety of the monomers including spiroorthocarbonate (SOCs) which expand {Millich, *et al*, 1998}, epoxy-polyol system combinations, which illustrate 40-50% less shrinkage in vitro than tradition systems {Tilbrook, *et al*, 2000}, or the use of high molecular weight molecules such as multiethylene glycol dimethacrylate and co-polymers which manage to achieve 90-100 conversion by reducing C=C bonds. Also Ormocers have demonstrated their ability to decrease

curing shrinkage. {Manhart, *et al*, 2000} Nevertheless the main dental composites still concentrate of traditional systems, mostly adding a BisGMA/TEEDMA monomer or BisGMA/VEDMA/TEGDMA combinations.

1.4.3 Wear behaviour

Among other factor, life expectancy of resin composites depends on the wear, which mostly occurs as result of occlusion, chewing, tooth brushing, or parafunctional activity, and might result in functional or aesthetic problems. Wear might occur through different process: abrasion, adhesion, fatigue and corrosion effects which act in various combinations depending upon materials properties {Braem, *et al*, 1998}.

High wear resistance is a desirable property for composite resins. The majority of studies has focused on reinforcing phase of composite considering the size of filler particle and surface treatment {Ruddell, *et al*, 2002; Lim, *et al* 2002; Turssi, *et al*, 2005} the use of porous fillers {Luo, *et al*, 1998}, silica-fused whiskers {Xu, *et al*, 2004}, and fibres {Callaghan, *et al*, 2006} to improve the wear resistance of dental composites.

Nanofilled dental composites (nanocomposites) are theoretically purported to have increased wear and fatigue resistance compared with microfill composites. Studies related to the effect of filler size on wear exhibited that a finer particle size for the composite results in less interparticle spacing, more protection of softer resin matrix, and less filler pulling all of which lead to enhanced wear resistance of the material {Kim, *et al*, 2006; Xing, and Li, 2004; Turssi, 2005}

The raised surface area of the sub-micron filler particles is also conceived to improve the attachment at the filler/matrix interface. The conventional microfil composites may comprise particles on the same size order as the new nanofill composites, while the former also normally comprises prepolymerized resin fillers (PPRF) ground to comparatively large sizes to increase overall filler loading. The adhesion between the resin matrix and the PPRFs has been exhibited to be slighter than ideal, and this may lead to increased wear in conditions of heavy loading. Consequently, it has been implied that the material elimination from the surface of nanofilled composites would be less than from composites filled with micrometer-sized particles or PPRFs {Mitra, *et al*, 2003}.

Yap *et al* investigated wear resistance of nanofill and ormocer composites and reported that wear resistance of nanofill and ormocer was comparable or superior to conventional hybrid composite {Yap, *et al*, 2004}.

However, Mitra, *et al* in 2003 found that the wear resistance of dental nanocomposite is equivalent to those of several hybrid composite {Mitra, *et al*, 2003}. Also Yesil *et al* 2008 reported that nanocomposite did not significantly improve wear resistance or the amount of opposing cusp wear when compared with traditional composites tested {Yesil, *et al*, 2008}. This finding supports Turssi, *et al* finding which reported that the new nano-structured dental composites are not likely to provide improved wear and fatigue performance over the traditional microfill composites {Turssi, *et al*, 2006}.

Over all little evidence is available that suggests that nanostructure materials show improvement over other available traditional composites {Dresch, *et al*, 2006; Turssi, *et al*, 2005}.

1.4.5 Surface roughness and gloss

The surface properties of restorative material are critical for their success, since they mediate the interaction of restorative materials with oral environment.

Surface roughness is a widely studied mechanical property. It has a chief effect on plaque accumulation, aesthetic impressions, gingival irritation {Bollen, *et al* 1997; Ono, *et al*, 2007} discoloration of restorations {Morgan, 2004}, secondary caries and wear of adjoining and opposing teeth. Moreover, smooth surface certifies sufferer ease and assists oral hygiene {Jefferies, 2007}.

Surface gloss is another factor playing an important role on aesthetic of composite resins. Gloss is a desirable characteristic for restorative materials to mimic the appearance of the enamel. More over glossy and smooth surface decrease coefficient of friction and subsequently this may reduce the wear rate. Many studies revealed that Mylar strip (poly ester matrix film) provides the smoothest and glossiest composite surface without subsequent finishing or polishing. However the use of this strip is limited because of the complex of tooth anatomy and diverse restorative {Attar, 2007; Baseren, 2004; Endo, *et al*, 2010}. Moreover such a surface has higher resin content and will reduce the wear resistance of the restoration over time {Anusavice, 2003; Morgan,

2004}. Therefore finishing and polishing of composite materials after placement are inevitable procedures that will enhance aesthetic, early wear resistance and marginal integrity and colour stability {Anusavice, 2003; Morgan, 2004; Lu, *et al*, 2005}.

Currently wide variety of finishing and polishing instruments are available such as multifluted tungsten carbide finishing burs, hard bonded-surface coated ceramic diamond rotary instruments, impregnated rubber or silicone discs and wheels and silicon carbides coated or aluminium oxide-coated abrasive discs, Among them aluminium oxid graded abrasive flexible disks were reported to produce the best result {Jefferies, 2007; Jung, *et al*, 2007; Jefferies, 1998}.

The final polishing result relies on the filler size, shape, and loading in resin composite. The larger the filler particles, the rougher the surface is after polishing {Yap, *et al*, 1997; da Costa, *et al*, 2010; Marghalani, 2010; Endo, 2010; Ryba, *et al*, 2002}.

Treatment of different resin composites with polishing and brushing led to varying degrees of surface roughness, depending upon the polishing systems and materials used {Costa, *et al*, 2007; Ergücü, andTürkün 2007; Marghalani, 2010}.

Brushing caused increased roughness on all surfaces, and the effects of polishing and brushing were reduced when nanofill resin composites containing nanoclusters were applied. Senawongse and Pongprueksa found that the nanofill resin composites with high filler loadings, best withstand the polishing and brushing processes. With comparable physical and mechanical properties as microhybrid resin composites, these nanofill resin composites have the potential to become good universal composite resin materials {Senawongse, and Pongprueksa, 2007}.

In another study conducted by Janus to investigate surface roughness of three nanocomposites polished with two different polishing systems, Nanocomposite which contains only nanofillers, showed the best results when associated to Sof-Lex polishing discs {Janus, 2010}.

1.5 The effect of composites composition on their properties:

The properties and hence the performance of composite resin, are dependent on three basic component of the material. Some properties are mainly related to the filler and coupling agent, other properties mainly stem from the resin matrix. The first property group includes stiffness, strength, wear resistance and coefficient of thermal expansion,

whereas colour stability and viscosity can be found in the second group. The third group depends on both filler and matrix such as polymerization shrinkage and water sorption.

1.5.1 The effect of fillers

The development of resin based composite system highlights a gradual decrease in filler particle size, there has been a significant increasing the use of nanofilled or nano-hybrid resin based composite in recent years {Curtis, *et al*, 2008}. Decreasing filler particle sizes is model of better dispersion and increased interfacial area between matrix and filler. This can be translated into increased flexural strength, surface microhardness, and polishability of the finished restoration {Saunders, *et al*, 2009; Beun, *et al*, 2007; Lohbauer, 2006}. In addition to the effect of particle size, morphology, and constituents of filler particles employed in dental RBCS have been identified to influence the properties of resultant material such as compressive strength, fracture toughness, and wear resistance {Kim, *et al*, 2002; Yap and Teoh 2003; Curtis, *et al*, 2008; Tanimoto, *et al*, 2006; Mitra, *et al*, 2003; Rodrigues, *et al*, 2007}.

Kim *et al* 2002 investigated the effect of filler morphology on filler loading, and evaluated the effect of filler morphology and loading on flexural strength and flexural modules. The composites were classified into four categories: prepolymerized, irregular-shaped, both prepolymerized and irregular-shaped and round particles. Filler loading was influenced by filler morphology. The flexural strength and modules were influenced by both filler morphology and filler loading. The composites with highest filler by volume exhibited the highest flexural strength, flexural modules and hardness {Kim, *et al*, 2002}. Also Yap and Teoh 2003 compared different types of composites, and they found that microfine composite with the lowest filler content have the lowest flexural strength and modules {Yap and Teoh 2003}.

Tanimoto and his colleague's conducted a study aimed to investigate the influence of particle size of filler particle on flexural properties of composite resins by computational approach. According to the results the flexural strength of composite resins decreased with increasing filler particle size. This investigation was limited to silica fillers ranging from 3.3 to 15.5 μm , which is considerably above the maximum particle size range of nanohybrids or nanofills {Tanimoto, *et al*, 2006}. However, a study by Beun, and his colleagues compared the physical properties of nanofilled, universal hybrid, and micro filled composites. Nanofilled showed the higher elastic modulus, while all materials tested exhibited similar flexural strength; microfills show the poorest physical

properties over all. The nanofilled resin in this study significantly showed mechanical properties at least as good as those of universal hybrids composite {Beun, *et al*, 2007}.

Mitra and colleagues in their study used bottom-up manufacturing design to produce nanocomposite contained a combination of nanomeric and nanocluster nanofillers. They reported that compressive strength and dimetral tensile strengths and fracture resistance of nanocomposites were equivalent to or higher than those of other commercial composites that they tested. The nanocomposites also showed better polish retention than hybrids and microhybrids tested after extended brushing periods {Mitra,*et al*, 2003}. The combination use of nanomeric particles and nanoclusters is reported to have distinct physical and mechanical properties compared with those of microhybrid composite {Curtis, *et al*, 2008}. Curtis *et al* 2009 found that water uptake and mechanical properties of composites were influenced by the size and morphology of the reinforcing particulate phase. They conducted a study to determine the influences of nanosized filler particales and agglomerates of nanocluster in resin based composites materials on their flexural strength. Composites containing different filler particle types and morphologies were used. A general linear model analytical of variance highlighted a reduction in flexural strength. However individual composite materials were response differently. Composite resin with nanocluster demonstrated distinctive and unique pattern of response. The conclusion of this study was that the nanocluster provided distinct reinforcing mechanism compared with the microhybrid, microfill or nanohybrid composites resulting in significant improvement to the composite strength and reliability {Curtis, *et al*, 2009}.

Rodrigues *et al* 2007 conducted a study to investigate whether the filler composition of resin composites influences their flexural strength and modulus of elasticity through a three-point bending test. Universal nanofilled composite, universal hybrid composites and a microfine composite were used. The results show significant association between the mechanical properties evaluated and the filler weight content. The microfine composite presented the lowest filler weight and the lowest mechanical properties. Statistically different flexural strength and modulus of elasticity results were observed among the universal hybrid composites. The nanofilled composite presented intermediary results. Thus the filler content significantly affect the flexural strength and modulus of elasticity of the composites tested {Rodrigues, *et al*, 2007}.

1.5.2 The effect of coupling agent:

In the composite, two major phases, a resin matrix and filler phase they are chemically bonded together by an interfacial coupling agent. Although the filler matrix interphase is the least abundant phase of composite material it can have significant effect on physico-mechanical properties of composites {Wilson, *et al*, 2005; Wilson, *et al*, 2006; Wilson, *et al*, 2007}.

Effective coupling between resin matrix and filler has been reported to protect the filler surface against fracture {Mohsen, and Craig, 1995} and also to improve distribution of stress transmission from the flexible resin matrix to stiffer and stronger inorganic filler particles {Calais, and Soderholm 1988}. The effect of the interface on physico-mechanical properties are more prominent in nanocomposites compared with conventional composites contain microsize filler, since nanofiller have extremely high surface area to volume ratio and require a high degree of silanization than large particulate filler {Wilson, *et al*, 2005; Wilson, *et al*, 2006}. In dental composites γ -methacryloxypropyltrimethoxy silane [γ -MPS] is the most widely used silane, which is expected to copolymerize with the methacrylic polymer matrix and chemically bond to the filler surface. Generally, it is considered that the amount of the silane coupling agent adsorbed on the filler and mechanical properties of the composite material show close association {Nishiyama, *et al*, 1991}. To be efficacious the amount of silane adsorbed on the filler surface must be optimal. Incomplete silane coverage of the filler surface causes it to insufficient bonding with the resin leading to intensified viscosity, non-uniform filler dispersion and inferior mechanical properties of the accompanying composites {Mohsen and Craig, 1995}. Silane can also contribute to degradation in mechanical properties if present in excess of an optimal amount {Ishida, and Koenig 1978}. Choice of the optimal amount of silane for dental resin composites is usually founded on a consideration of their physical properties with little or no concern to their hydrolytic stability. The amount of silane that is favorable for mechanical properties may not be advantageous to the perspective of their resistance to hydrolysis in the aqueous oral environment {Karmaker, *et al*, 2007}.

Sideridou and Karabela 2009, found that the amount of silane used for silanization of silica particles affect the orientation of silane molecules relative to silica surface. This seems to affect the dynamic mechanical properties of composites {Sideridou, and Karabela, 2009}.

1.5.3 The effect of the monomer

The monomer BisGMA has been used as an important dental base monomer since it was invented in early 1960's. BisGMA is very viscous due to –OH groups. To improve the handling properties, and increase crosslinking, and increase hardness a low-molecular-weight (low-viscosity) monomers like triethyleneglycol dimethacrylate [TEGDMA] and ethyleneglycol dimethacrylate [EGDMA] were added to thin the resin. In BisGMA/ TEGDMA dental resin system. The role of BisGMA is to limit the photopolymerization induced volumetric shrinkage and to enhance resin reactivity, while TEGDMA increases vinyl double bond conversion {Vasudeva, 2009}. Several monomers developed with the goal of minimizing polymerization shrinkage and its associated stress. While these monomers were promising, problems balancing mechanical properties, water sorption, curing time, solubility still exist {Klee, *et al*, 1999; Miyazaki, *et al*, 1994; Nuyken, *et al*, 1996; Stansbury, 1992}.

In recent years the development of dental composites has focused on the use of organic–inorganic hybrid nanocomposite such as polyhedral oligomeric silsesquioxanes (POSS). POSS monomer is represented by the empirical formula $(\text{RSiO}_{1.5})_n$ with an inorganic silica-like core $(\text{SiO}_{1.5})$ surrounded by organic corner groups R. It has been shown that the performance of POSS modified polymers is generally attractive. They have the organic characteristics such as good processability, durability and low expense, in addition it containing outstanding inorganic implementations in mechanics, thermodynamics, anti-oxidation, etc {Musselman, 2003; Mark and Lee 1995; Jang, *et al* 2001}. At present day, it is a very important way to obtain functional and good performance materials altered by POSS.

Sellinger and Laine {Sellinger and Laine, 1996} first referred the feasibility of POSS being used in dental restorative materials. In a later study, the research group of Culbertson and co-workers {Gao, *et al*, 2001} assessed the techniques of POSS embodied with neat resins (without filler). The outcomes indicated that miscibility between the POSS constituent and the matrix, particularly the diluents, performed a highly significant part in developing the properties of the invented thermosets. Novel polymeric dental restorative composites were investigated {Fong, *et al* 2005}, in which POSS-MA was utilized to partially (or completely) replace BisGMA founded on the research of Culbertson and co-workers' group {Gao, *et al*, 2001}. The outcomes showed that mechanical properties of the composites could be better by only a small amount of POSS-MA substitution of BisGMA in the resin systems. Nevertheless, it was

a disappointment that there was no different enhancement in such consequential properties as compressive strength, hardness and toughness.

Dodiuk-Kenig et al. 2006 inferred that the kind of the grafted functional group of the caged silica was the leading factor in nanotailoring of improved dental composites and adhesives {Dodiuk-Kenig, 2006}. It revealed that the acrylated POSS enhanced the mechanical properties of dental composites and adhesives but the properties decreased by octaphenyl grafted POSS. Much other research works {Li et al. 2008; Cho, *et al*, 2005; Dell'erba and Williams 2007; Wu, *et al*, 2010} have been done on the constructional design of the POSS molecule, synthesizing the process of modified resins, as well as properties characterization. However, we have to confess that substantially complex methods of synthesis and rigorous conditions of reaction are required for most new POSS monomers. Hence, this acutely limits the progression and implementation of POSS in dental restorations.

1.6 Improvement of dental resin composites

1.6.1 Reinforced Fillers

Nanofibers can be used as reinforcement of dental composites applications {Fong, 2004; Tian, *et al*, 2007}. In study conducted by Tian et al. 2007, electorspun nylon 6 nanocomposite nanofibers containing highly aligned fibrillar silicate single crystals were added in BisGMA/TEGDA, for reinforcement of dental composite. The results revealed that the small mass fractions of nanofiber impregnation improved the mechanical properties substantially, while large mass fraction of nanofiber impregnation resulted in less desired mechanical properties {Tian, *et al*, 2007}.

1.6.2 TiO₂ Nanoparticles

In order to improve mechanical properties of composites the surface of TiO₂ nanoparticules (<20nm) was modified with organosilane allytriethoxysilane (ATES) {Xia, *et al*, 2008}.

The modification by organosilane ATES influences the dispersion and linkage of TiO₂ nanoparticules within a resin matrix and adding the modified nanoparticules improves

the microhardness and flexural strength of dental resin based composites {Xia, *et al*, 2008}.

1.6.3 Caries-prevention Fillers

Secondary caries and restoration fracture remain the two main problems in restorative dentistry {Sarrett, 2005}. Although composites are generally satisfactory for small restorations, they are not recommended for large, stress bearing restorations {Sakaguchi, 2005}. Secondary caries is the most frequently reason for the replacement of existing restorations {Mjör, *et al*, 2000}. There is evidence that sustained release of fluoride ions (F^+) could be a substantial benefit for a dental restoration, because the fluoride could enrich neighbouring enamel or dentine to combat secondary caries {Hsu, *et al*, 1998}.

To address the two problems of secondary caries and restoration fracture, the nanoparticles and reinforcing whiskers were combined to develop stress-bearing, caries-inhibiting composites. These nanocomposites released supersaturating levels of calcium (Ca^+) and phosphate (PO_4^+) ions requisite for remineralization to occur, while possessing mechanical properties that matched commercial stress-bearing, non-releasing composites {Xu, *et al* 2006; Xu, *et al* 2007a, Xu, *et al* 2007b}.

1.6.3.1 DCPA-whiskers

In the study of DCPA nanoparticles, 2 types of fillers were used: DCPA particles and nano-silica fused whiskers. It was found that decreasing the DCPA particle size decreased the composite's strength, while whisker reinforcement more than doubled the composite's strength and significantly increased the elastic modulus. The investigators also found that silanization of the DCPA particles increased the composite's strength, but decreased the Ca^+ and PO_4^+ release. The use of unsilanized nano DCPA together with whisker reinforcement appeared to be the best method to produce a composite with high strength and Ca and PO_4 release {Xu, *et al*, 2007a}.

Xu and colleuogess in another study found that increasing the DCPA partical surface area significantly increased the Ca^+ and PO_4^+ release, and composites with nano DCPA exhibited the highest release {Xu, *et al*, 2007a}.

Nanocomposites which contain CaF_2 and DCPA, can release F^+ , Ca^+ , and PO_4^+ ions for precipitation of fluoroapatite and inhibition of caries, it were also formulated with good mechanical properties {Xu, *et al*, 2008}.

Study by Xu et al. (2008) illustrated that the combination of calcium fluoride (CaF_2) nanoparticles and reinforcing whisker fillers and nanocomposites improved fluoride release than that of traditional and resin-modified glass-ionomer materials. The strength and elastic modulus of the nanocomposite also matched those of commercial stress-bearing, non-releasing composites {Xu, *et al*, 2008}.

1.6.3.2 TTCP Whiskers:

Another calcium ion releasing composites with fine tetracalcium phosphate TTCP and nanosilica-fused whiskers have been developed in order to improve esthetics since the whisker-reinforced $\text{Ca}_3(\text{PO}_4)_2$ composites were relatively opaque.

Xu, et al. (2010), found that TTCP-whisker composite has strengths about two-fold those of the TTCP composites without whiskers and was suggested that whiskers may have the potential to provide the required combination of load-bearing and caries-inhibiting capabilities {Xu, *et al*, 2010}.

1.6.3.3 NACP Nanocomposite:

Xu, 2011 found that nanocomposites which contain amorphous calcium phosphate nanoparticle greatly increased the ion release at a cariogenic pH4 (NACP).

The new NACP nanocomposite had strength matched or exceeded a commercial composite {Xu, 2011}

1.7 Aims of the study:

The aims of the study were

a- To investigate the mechanical properties of nanohybrid composite materials and compare it with the mechanical properties of microhybrid composite. For this the following were evaluated;

- Flexural strength.
- Flexural modulus.
- Compressive strength.

b- To investigate the differences in surface roughness and gloss between the materials.

c- To investigate the effect of brushing and polishing on the surface roughness and gloss.

For (b and c) the following were evaluated;

- Surface roughness (Ra) of the materials before polishing procedure.
- Surface roughness (Ra) of the materials after polishing procedure.
- Surface roughness (Ra) of the materials after tooth brushing abrasion.

- Surface gloss of the materials before polishing procedure.
- Surface gloss of the materials after polishing procedure.
- Surface gloss of the materials after tooth brushing abrasion.

d- To assess the relationship between 2D surface roughness measurements and 3D surface roughness measurements.

1.8 Hypotheses:

Mechanical properties

1-significantly different flexural strength mean values would be found between the materials tested.

2 -significantly different flexural modulus mean values would be found between the materials tested.

3-significantly different compressive strength mean values would be found between the materials tested.

Surface roughness

1-polishing and toothbrushing abrasive effects on the surface roughness of the materials tested.

2-Signifacantly different surface roughness mean values would be found between the materials tested.

3-Significant relationship would be found between 2D surface roughness mean values and 3D surface roughness mean values.

Surface gloss

1-polishing and toothbrushing abrasive effects on the gloss of the materials tested.

2-signifacantly different gloss mean values would be found between the materials tested.

Chapter 2

Materials and Methods

2. Materials and Methods:

2.1 Materials:

Four materials were used in this study two nanohybrid composite and two microhybrid composite. The materials used in this investigation listed in Table 2.1.

Herculite XRV Ultra (Kerr, Italy, dentin shade A2, batch no 3089406), Filtek Z250 (3M ESPE, USA, shade A3, batch no 20051217), Tetric Evoceram (Ivoclar vivadent, Liechtenstein, shade A2, batch no L56579), Venus (Heraeus Kulzer, Germany, batch no 010125) and OpticDisc polishing system (Kerr, KerrHawe, Switzerland).

2.2 Methods:

2.2.1 Flexural strength and flexural modulus test:

2.2.1.1 Specimen preparation for flexural strength and flexural modulus tests:

A stainless-steel mould which has escape channel to extrude excess materials and reduce surface defect was used to prepare 10 rectangular specimens (2 x 2 x 25 cm) for each test Fig 2.1. Each provisional material was placed on the mould and covered with a glass slid to which pressure was applied to extrude excess material. The top and the bottom surfaces were then polymerized with the use of three overlapping irradiations of 20s each with the use of a curing light (Optilux 501, SDS Kerr, USA) that emitted 500 mw/ cm² as measured by internal radiometer, calibrated against a flat response power meter.

After the samples were removed from the mould they were examined for any surface defect and irregularities on the surface and edges by naked eyes. Sharp and irregular edges were removed by using abrasive paper of 400 grit and samples with porosities were eliminated. Samples were stored in plastic bag at room temperature for 24 h before testing. All specimen dimensions were measured with a digital metal calliper (Draper expert, U.K); the measurements were taken in three locations for width, thickness, and length.

Table 2.1 List and information about the composites tested according to the manufactures

Materials	Type	Shade	Manufacture	Filler content wt% / vol%	Composition
Herculite XR Ultra	Nanohybrid	A2	Kerr	97	Matrix:Bis GMA, TEGDMA Filler:PPF, barium glass, Silica nanofiller
Tetric Evo ceram	Nanohybrid	A2	Ivoclar- Vivadent	82.5/68	Matrix: BisGMA, UDMA, dimethacrylate, TEGDMA Filler: Fluorosilicate glass, so ₂ microfiller and spherical nanofiller (Barium glass, ytterbium trifluoride)
Filtek Z250	Microhybrid	A3	3M ESPE	84.5/60.0	Matrix:BisGMA,UDMA, TEGDMA Filler: Zirconia, silica
Venus	Micro hybrid	A3	Hereaeus Kulzer	79/61	Matrix:BisGMA, TEGDMA Filler:so ₂ , Ba-Al-B-F-Si glass

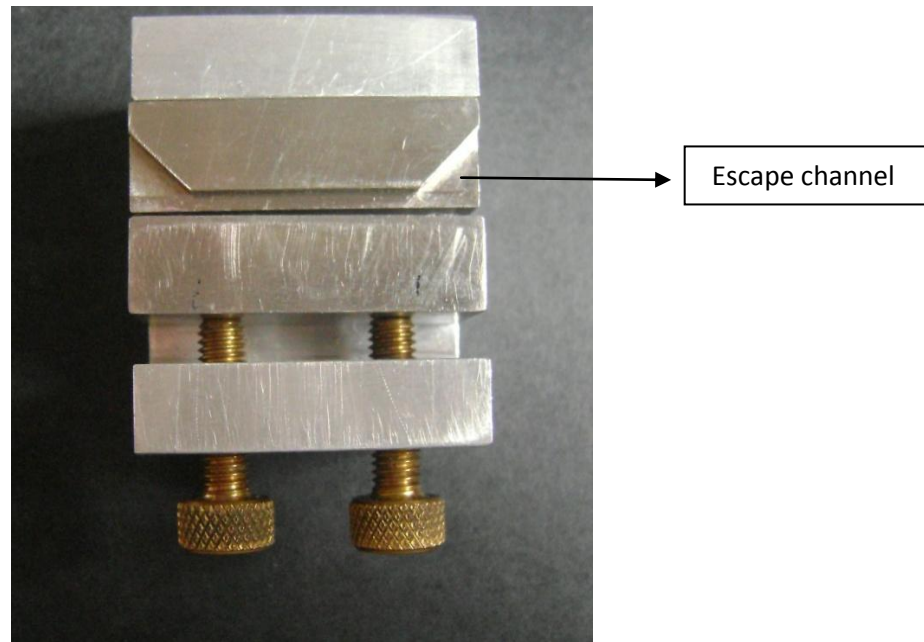


Fig 2.1 The stainless-steel mould used to prepare specimens for flexural strength test.

2.2.1.2 Three point bending test:

Following storage all specimen were subjected to transverse loading to determine the maximum load required for fracture. The measurements were performed using a universal testing machine (Zwick/ Role, Leominster, UK) at cross head speed of 1mm/minute. The specimens were placed on fixtures on universal test machine with 20mm distance between the supports (L) Fig 2.2.

The flexural strength was expressed as maximum flexural load pre-cross-sectional area of specimen (MPa), according to international standards organization (ISO 4049) Fig 2.3.

The values of flexural strength were obtained from three points bending test, in (MPa), based on the following formula:

$$\text{Flexural strength} = \frac{3FL}{2bd^2}$$

Where F is the force load at fracture point (N), L is the length of support span (mm), d thickness (mm), and b the width of specimen (mm).

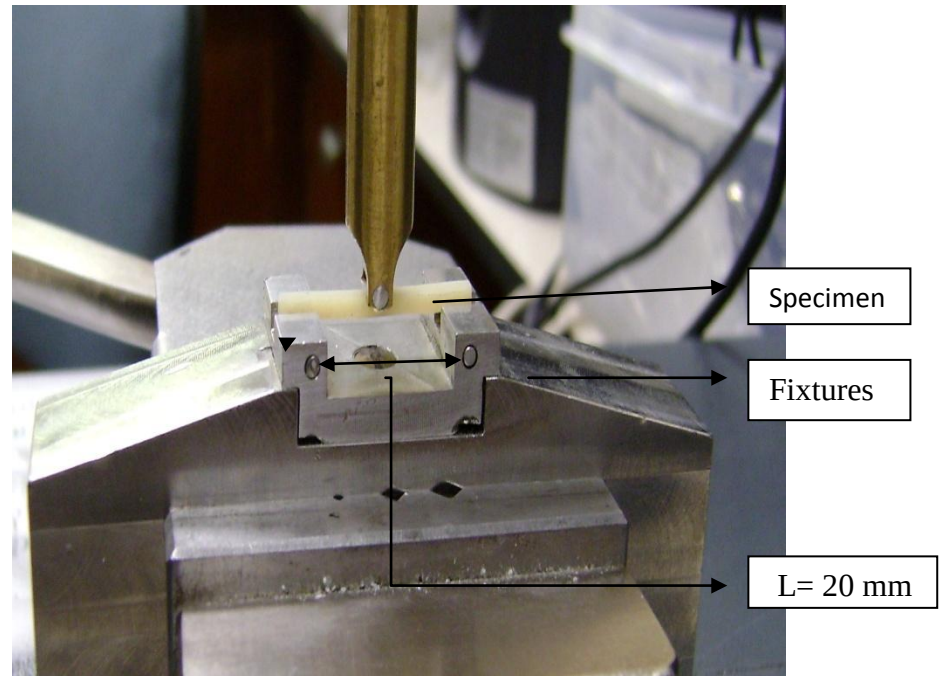


Fig 2.2 flexural strength test

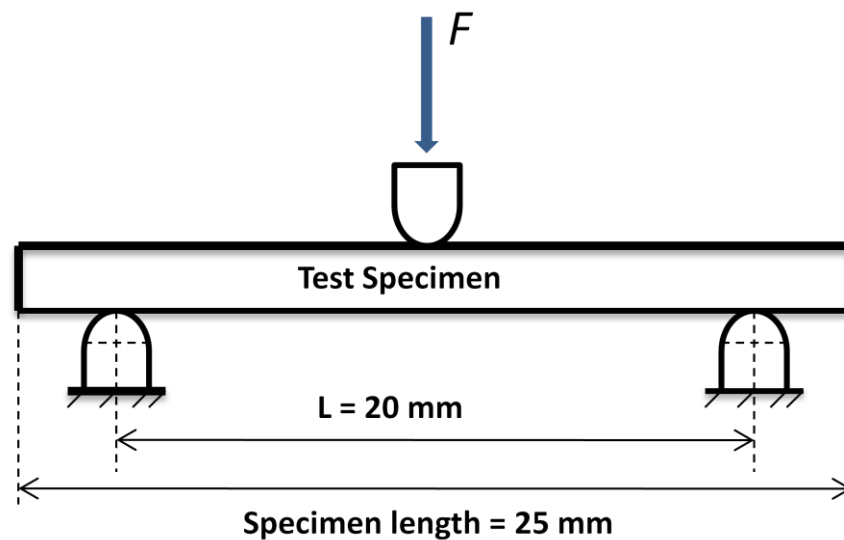


Fig 2.3 Schematic of test setup for ISO 4049 three point bending test

2.2.1.3 Flexural modulus test:

Load/displacement curves which generated by the flexural strength test were used to determine the flexural modulus, the flexural modulus E , in MPa, was determined at the non destructive interval of load/deflection curve using the following formula;

$$\text{Flexural modulus (E)} = \frac{L^3 \times F_1}{4Sbd^3}$$

Where, S is deflection in mm of specimen at load F_1 , b the width, and d the thickness of the specimen in mm, and L , length of support span (mm).

2.2.1.4 Statistical analysis:

One way analysis of variance (ANOVA) test, followed by Tukey`s test, for multiple comparisons between means to determine significant differences was used at significance level set at $p \leq 0.05$.

2.2.2 Compressive strength:

2.2.2.1 Specimen preparation for compressive strength test:

Cylindrical specimens ($n=10$), 4mm diameter x 6mm height were prepared by using split stainless steel mould Fig 2.4. The mould was placed on glass slide and filled with the materials and cover with another class slide, then the material was compressed by hand pressure using two flat surfaces. The top and the bottom end of the specimen was exposed to light cure unite for 60s, and then the specimen was removed from the mould and cured on a longitudinal side for another 40s. A total of 160s light curing was done for each specimen. The specimens were stored in plastic bags at room temperature for 24h.

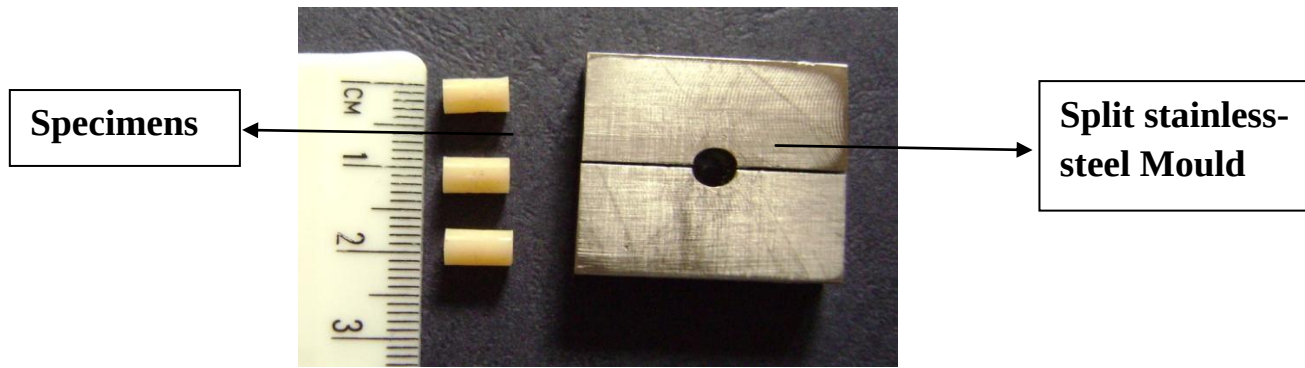


Fig 2.4 split stainless steel mould used to prepare specimens for compressive strength test.

2.2.2.2 Compressive strength test:

Prior to test the diameter of each specimen was measured by digital calliper (Draper expert, UK). The specimen was placed on its end between the plates of universal testing machine (Zwick / Role, Leominster, UK) (Fig 2.5). The compressive load was applied along the long axis of specimen Fig 2.6 at cross-head speed 1.0 mm/min up to failure. The compressive strength was calculated based on peak load and diameter of the specimen by using the following formula; F/A

Where F , the force at failure point and A , cross sectional area.

2.2.2.3 Statistical analysis:

One way analysis of variance (ANOVA) test, followed by Tukey's test, for multiple comparisons between means to determine significant differences was used at significance level set at $p \leq 0.05$.

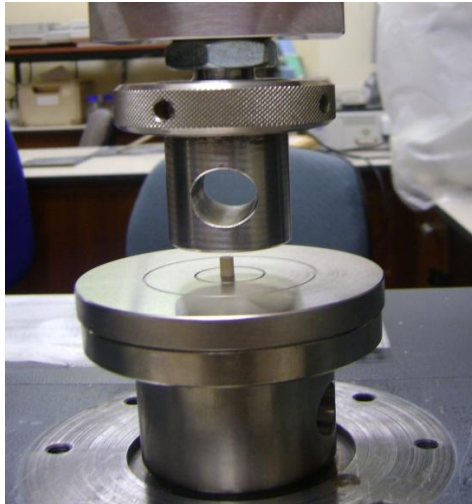


Fig 2.5 compressive stress test

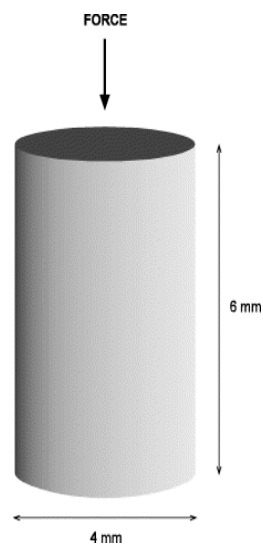
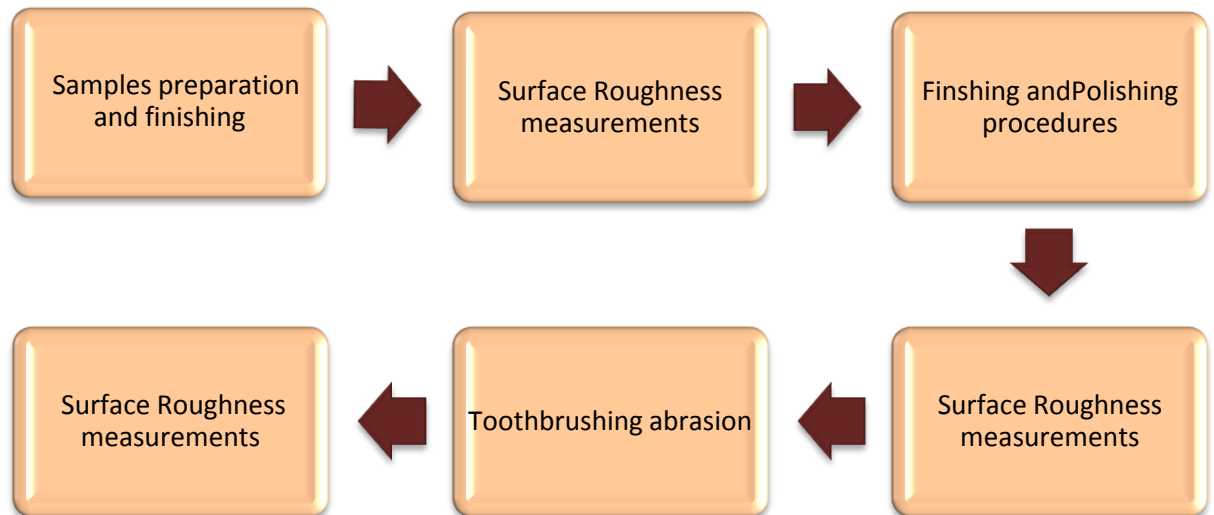


Fig 2.6 schematic diagram of the specimen in the compressive strength test the load was applied along the long axis of specimen.

2.2.3 Surface roughness measurements:

The study was performed as following:



2.2.3.1 Specimen preparation:

Five disc shaped specimens (12 mm diameter, 2mm height) Fig. (2.7) were prepared for each material by using ring shape Teflon mould Fig 2.8. Teflon mould was placed on glass slide and filled with composites material and covered with another glass slide, then the mould was compressed by hand pressure between the two slides to extrude excess material and obtain flat a surface. The disc was light cured for 40 s from each surface with a QTH light curing unit (Optilux 501, Danbury, USA) emitting 500 mw/cm² as measured by the incorporated radiometer, calibrated against a flat response power meter.

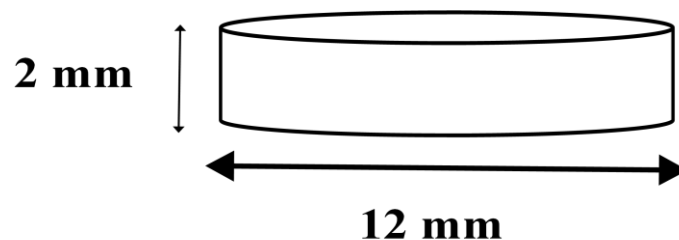


Figure 2.7 Typical specimen (diameter: 12 mm; height: 2 mm).

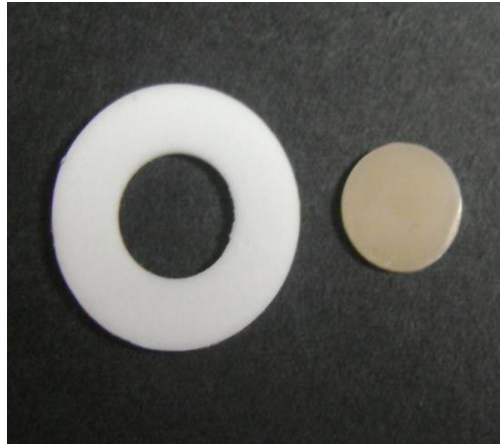


Figure 2.8 Telfon mould used to prepare the specimen for surface roughness and gloss measurements.

2.2.3.2 Surface roughness measurement before finishing and polishing:

Surface roughness for all materials was measured after treating the specimens with abrasive paper grit size 400 in order to standardise the base line of all materials.

2.2.3.3 Surface roughness measurements:

A contact stylus profilometer (Taylor Hobson Precision Instrument, Taylor Hobson Ltd, Leicester, England) was used to measure the surface roughness Fig 2.9.

The diamond stylus has radius of 5 μm , tip angle of 90° and transversed at a contact speed 1.00 mm/s across the surface with force 6 mN. For each specimen six line scans were performed, three perpendicular direction and three horizontal direction. The cut off length was .25 mm and the measuring length 2 mm.

For each scan R_a , R_t , R_{sm} were recorded, representing different surface texture.

R_a : is the most commonly used parameter in surface texture analysis and define as the arithmetic mean of the absolute departures of the roughness profile from the mean line.

R_t : the vertical height between the highest and lowest point of the profile within the evaluation length.

R_{Sm} : used to describe the average spacing of profile elements (peaks and valleys) measured along the measurement direction.

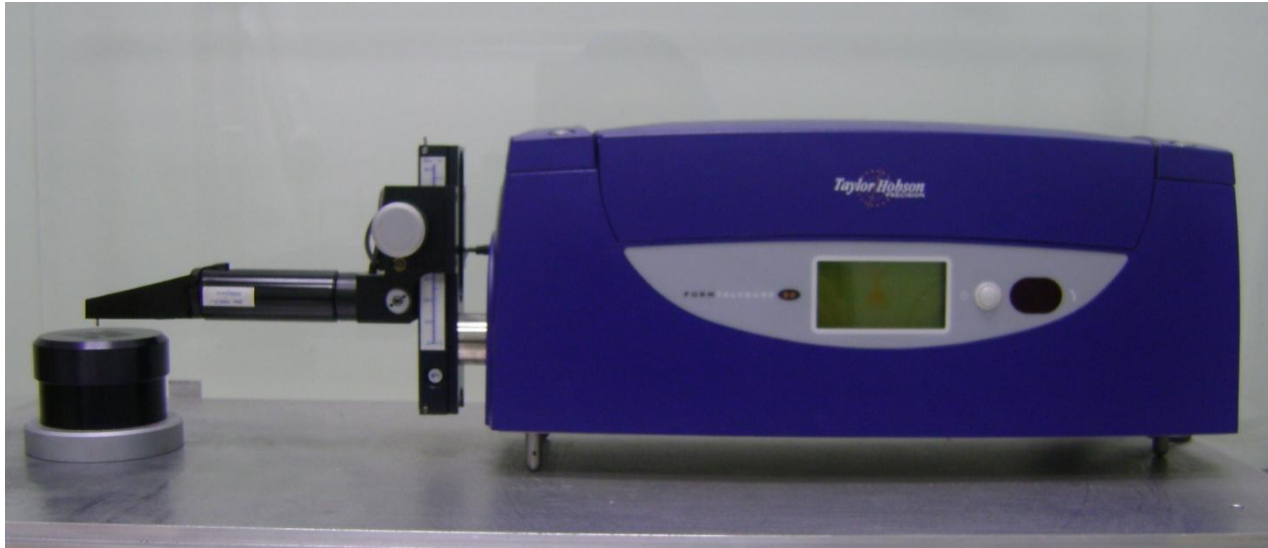


Figure 2.9 A contact stylus profilometer (Taylor Hobson Precision Instrument)

2.2.3.4 Finishing and polishing

The samples were finished and polished using OptiDisc system (Kerr, KerrHawe, Switzerland) Fig 2.10. For each sample a finish disc, fine polish disc, and high gloss disc were used. The time was 30s for each disc to standardize the time for all samples. After polishing, the specimens were placed in an ultrasonic water bath (Elma ultrasonic T 310, singen, Germany) for five minutes to remove any potentially remaining debris. Surface roughness measurement (Ra, Rt, Rsm) was recorded immediately after the finishing and polishing procedures.



Figure 2.10 OpticDisc polishing system

2.2.3.5 Surface roughness measurements after finishing and polishing:

Surface roughness measurement was taken immediately after finishing and polishing procedures, Ra, Rt, Rsm was recorded for each sample.

2.2.3.6 Simulation of toothbrushing:

Specimens brushing were developed in a custom made toothbrush simulating machine from the University of Manchester Biomaterial's laboratory which is shown in Fig 2.11. The simulator was equipped with four independent stations in which to place the specimens and four separated toothbrush holder, which were driven by motor. A toothbrush head with soft bristles (Oral-B 40 indicator, regular, Oral-B Laboratories, London,UK) was used and fixed to the holder with impression material.

The specimens were placed in the station and toothbrushes were placed in parallel to the specimens, with the bristles in contact with the specimens. The movement of crankshaft sets the arms in motion with active part of the brush in a unique direction and in two movements, forward and backward. To record the movement number there is a counter fixed in the base of the machine, the brushing speed was 78 cycles per minutes giving a total of 312 tooth brush stroke per minutes.

The slurry was prepared by mixing water with dentifrice (Colgate Total, Manchester, UK) in ratio (2:1). The slurry was filled into the station of the brushing machine and was replaced for every new sample.

Before the brushing test began, the testing machine was adjusted to apply 2.5 N vertical loads on the specimen during horizontal movement of the brush throughout the test. All specimens were brushed with strokes 32,000 times, as measured with an incorporated meter. After abrasion, specimens were removed from the machine, rinsed with tap water, cleaned in an ultrasonic water bath (Elma ultrasonic T 310, singen, Germany) for two minutes, and gently dried. All roughness measurements were then repeated.

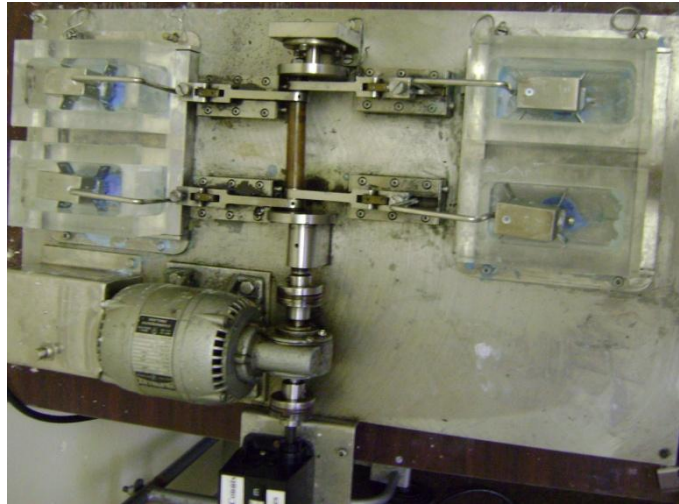


Figure 2.11 Simulating toothbrushing machine.

2.2.3.7 Statistical analysis:

T-test for paired data was used to test the effect of polishing and toothbrushing abrasive on the surface roughness of tested materials

One way analysis of variance (ANOVA) test, followed by Tukey's post-hoc test, for multiple comparisons between surface roughness R_a , R_t , R_{sm} means values to determine significant differences between the materials, after polishing, and after brushing at significant level $P \leq 0.05$.

2.2.4 Measurement of surface gloss:

Surface gloss was measured with Nova-curve glossmeter (Rhopoint, instrumentation LTD Bexhill on sea, England) Fig 2.12. The measurement of this device based on a light beam that strikes the surface at an angle 60° , and measurement area of 2×2 mm.

To perform the measurements, the specimen was placed on the top plate. The specimen was covered with black cover to shield the specimen from external light while the measurement is conducted. Five readings were performed at the centre of each specimen, each time turning the specimen by 90° . The five readings were averaged to obtain a single value for each specimen. The gloss was measured as following;

Before polishing

After polishing

After brushing



Figure 2.12 Gloss-meter (Novo-Curve, Rhopoint, Bexhill-on-Sea, England).

2.2.4.1 Statistical analysis:

T-test for paired data was used to test the effect of polishing and toothbrushing abrasive on the gloss of tested materials.

One way analysis of variance (ANOVA) test, followed by Tukey's post-hoc test, for multiple comparisons between gloss means values to determine significant differences between the materials, after polishing, and after brushing at significant level $P \leq 0.05$.

2.2.5 3D surface roughness measurements:

The same sample used before were used. The 3D surface roughness measurements were recorded by using non-contact stylus surface profilometry TALYSURF CLI 1000 from Talysurf Hobson precision (Leicester, England) with Talysurf CLI soft-ware to control the machine and platinum soft-ware for calculation. The gage used was CLA 3mm and sampling rate of 1000 Hz. The measured surface was 10mm x 3mm at speed of 5mm/s with 100 μm increment, a surface levelling was applied to eliminate sample misalignment. Three surface parameter were measured Sa, St, Sds.

Sa: average absolute deviation of the surface.

Sds: density of summits of the surface. This parameter used to evaluate the density of peaks and pits in the surface.

St : (Peak-Peak Height) the height difference between the highest and lowest pixel in the image

2.2.5.1 3D surface roughness measurements after tooth brushing:

After brushing the specimens were placed in an ultrasonic water bath (Elma ultrasonic T 310, Singen, Germany) for five minutes to remove any potentially remaining debris. The specimens were scanned immediately after the cleaning and drying and The Three surface parameter were recorded (Sa, St, Sds).

2.2.5.2 3D surface roughness measurements after polishing:

The same specimens were polished using OptiDisc system as described in (2.3.4) and cleaned using an ultrasonic water bath for five minutes and then scanned. The Three surface parameter were recorded (Sa, St, Sds).

2.2.5.3 Statistical analysis:

One way analysis of variance (ANOVA) test, followed by Tukey`s post-hoc test, for multiple comparisons between 3D surface roughness parameter (Sa, St, and Sds) mean

values to determine significant differences between the materials, after polishing, and after brushing at significant level $P \leq 0.05$.

2.2.5.4 3D surface roughness (Sa) and 2D surface (Ra) Correlation

The correlation between the 3D surface roughness (Sa) and 2D surface roughness (Ra) for all tested materials is plotted using *Sigmaplot* software (version 9, IL, VSH).

Chapter 3

The results

3. The Results:

3.1 Flexural strength test results:

Mean values and standard deviation of the flexural strength of the materials tested are shown in Fig 3.1 and Table 3.1 the values varied from 136 MPa for Filtek Z250 vs to 83.75 MPa for Tetric Evo Ceram.

One way ANOVA test exhibited significant differences ($P \leq 0.05$) among the tested composite materials for the mean values of the flexural strength. Pair wise multiple comparisons with Tukeys HSD test ($\alpha=0.05$) revealed that Filtek Z250 exhibit significantly high values for flexural strength than for all other materials.

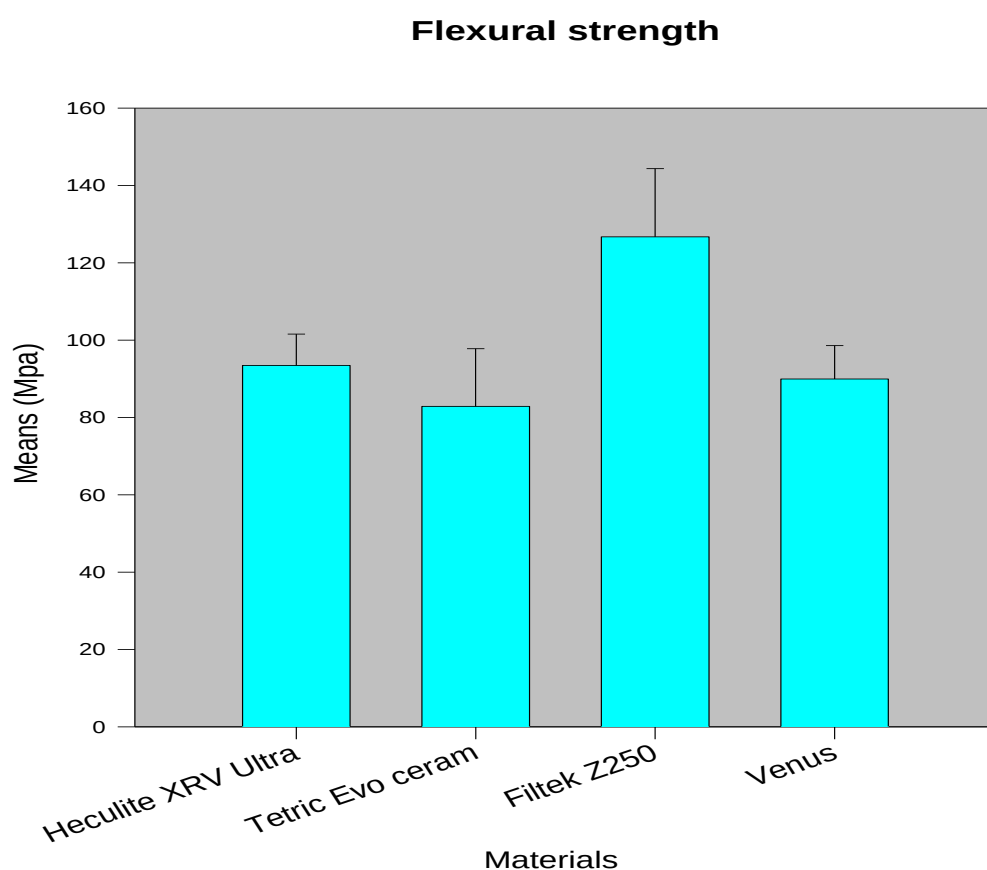


Figure 3.1 Error bar chart illustrates the mean flexural strength and standard deviation of composite materials tested

Materials	CS (S.D.) MPa	FS (S.D.) MPa	FM (S.D.) GPa
Herculite XRV Ultra	300.95 (52.9) a	93.46 (8.9) a	4.96 (0.55) a
Tetric Evo ceram	237.74 (44.9) b	83.75 (8.72) a	5.45 (0.9) a
Filtek Z 250	405.33 (20.28) c	136.71 (17.63) b	7.9 (0.34) b
Venus	330.17 (14.59) a	89.93 (8.65) a	5.63 (1.05) a

Table 3.1 Mean values, standard deviation and significance of compressive strength (CS), flexural strength (FS), and flexural modulus (FM) for composite materials tested.

*The means with the same letter are not significantly different

No statistical differences detected among Herculite XRV Ultra Vs. Venus and Tetric Evo Ceram. Also no significant differences detected between Venus and Tetric Evo Ceram. The comparison and significant between the materials tested and differences of the means showed in Table 3.2.

Comparison	Diff of Means	P	<i>P</i><0.050
Z250 vs. TEC	43.851	0.004	<i>Yes</i>
Z250 vs. XRVU	33.252	0.002	<i>Yes</i>
Z250 vs. V	36.773	0.006	<i>Yes</i>
XRVU vs. TEC	10.599	0.666	<i>No</i>
XRVU vs. V	3.520	0.969	<i>No</i>
V vs. TEC	7.078	0.908	<i>No</i>

Table 3.2, the significant between the materials tested and differences of the means value of flexural strength, Z250=Filtek Z250, TEC=Tetric Evo ceram, XRVU=Herculite XRV Ultra, V=Venus

3.2 Flexural modulus test results:

Mean values and standard deviation of the flexural modulus of the materials tested are shown in Fig 3.2 and Table 3.1. The values varied from 4.96 GPa for Herculite XRV Ultra vs to 7.9 GPa for Filtek Z250.

One way ANOVA test exhibited significant differences ($P \leq 0.05$) among the tested composite materials for the mean values of the flexural modulus. Pair wise multiple comparisons with tukeys HSD test ($\alpha=0.05$) revealed that Filtek Z250 exhibited significantly high values for flexural modulus than for all other materials.

No statistical differences were observed between Venus and Herculite XRV Ultra, Venus and Tetric Evo Ceram. Also there were no significant differences between Tetric Evo Ceram and Herculite XRV Ultra. The comparison and significant between the materials tested and differences of the means showed in Table 3.3.

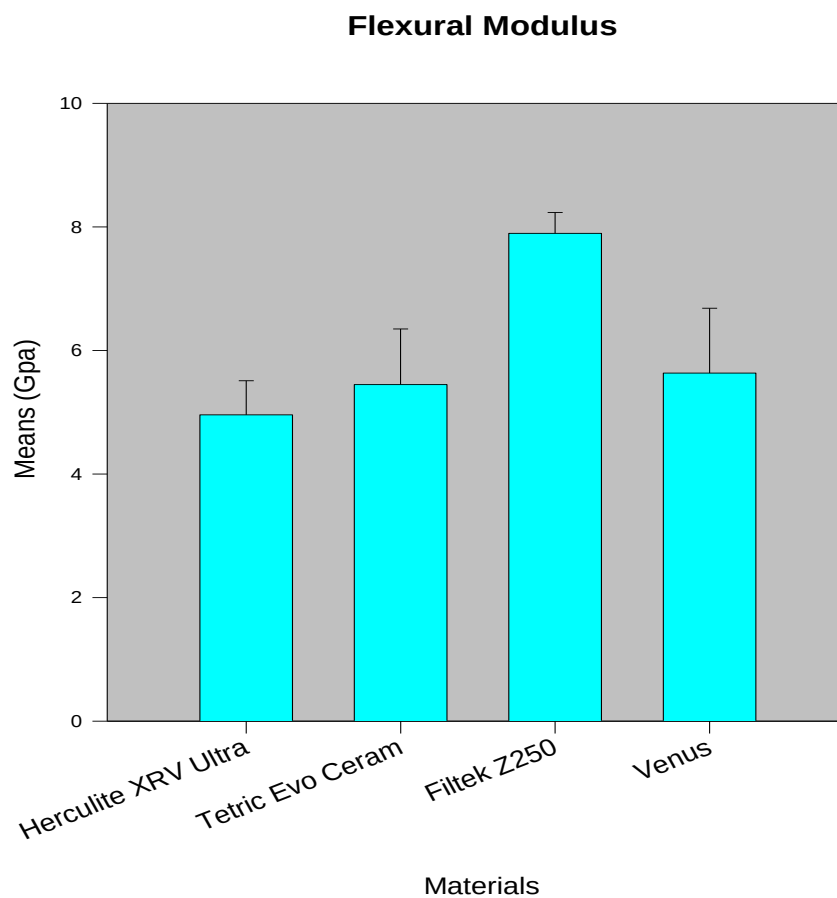


Figure 3.2 Error bar chart illustrates the mean flexural modulus and standard deviation of composite materials tested

Comparison	Diff of Means	P	P<0.050
Z250 vs. XRVU	2.938	<0.001	Yes
Z250 vs. TEC	2.449	<0.001	Yes
Z250 vs. V	2.263	0.003	Yes
V vs. XRVU	0.675	0.491	No
V vs. TEC	0.186	0.984	No
TEC vs. XRVU	0.489	0.660	No

Table 3.3 The significant between the materials tested and differences of the means of flexural modulus. Z250=Filtek Z250, TEC=Tetric Evo ceram, XRVU=Herculite XRV Ultra, V=Venus.

3.3 Compressive strength test results:

Mean values and standard deviation of the compressive strength of the materials tested are shown in Fig 3.3 and Table 3.1. The values varied from 237.74 MPa for Tetric Evo Ceram vs to 405.33 MPa for Filtek Z250.

One way ANOVA test exhibited significant differences ($P \leq 0.05$) among the tested composite materials for the mean values of the flexural strength. Pair wise multiple comparisons with Tukeys HSD test ($\alpha=0.05$) revealed that Filtek Z250 exhibited significantly high values for flexural strength than for all other materials.

No statistical differences were observed between Venus vs. Tetric Evo Ceram and Herculite XRV Ultra vs. Tetric Evo Ceram but there were significant differences between Venus and Tetric Evo Ceram. Tetric Evo Ceram exhibited the lowest values for compressive strength among the tested materials. The comparison and significant between the materials tested and differences of the means are shown in Table 3.4.

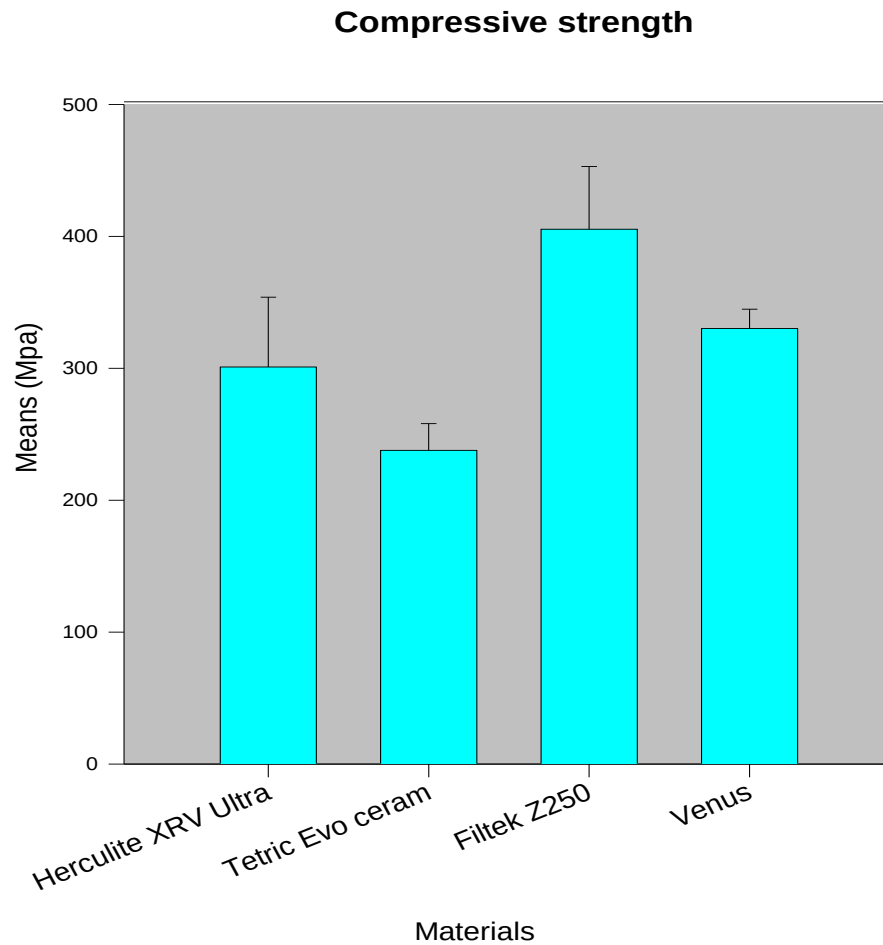


Figure 3.3 Error bar chart illustrates the mean compressive strength and standard deviation of composite materials tested

Comparison	Diff of Means	P	P<0.050
Z250 vs. TEC	167.589	<0.001	Yes
Z250 vs. XRVU	104.377	<0.001	Yes
Z250 vs. V	75.160	0.095	No
V vs. TEC	92.429	0.042	Yes
V vs. XRVU	29.217	0.748	No
XRVU vs. TEC	63.212	0.071	No

Table 3.4, The significant between the materials tested and differences of the means of compressive strength. Z250=Filtek Z250, TEC=Tetric Evo ceram, XRVU=Herculite XRV Ultra, V=Venus

3.4 Surface Roughness measurements results

Surface roughness Ra, Rt, and Rsm mean values (μm) and standard deviations for all tested materials are presented in Table 3.5 also presented in Figure3.4-Figure3.6.

Materials		Ra		Rt		Rsm	
		Mean	Sd	Mean	Sd	Mean	Sd
Herculite	A	0.86	0.56	5.77	1.07	90.91	11.14
	b	0.05	0.02	0.39	0.11	83.64	5.22
	C	0.15	0.05	1.16	0.21	111.85	17.01
Filtek	A	0.60	0.08	4.98	0.59	82.24	6.47
	b	0.05	0.02	0.38	0.13	80.01	17.53
	C	0.10	0.04	0.77	0.33	62.37	5.23
Tetric	A	0.52	0.04	4.80	0.43	90.47	12.92
	b	0.05	0.01	0.43	0.23	82.89	7.50
	C	0.26	0.05	1.47	0.27	86.05	14.78
Venus	A	0.50	0.12	4.03	0.64	82.69	6.14
	b	0.04	0.01	0.43	0.04	81.23	3.72
	C	0.07	0.02	0.59	0.09	78.65	3.57

Table 3.5 Ra, Rt, and Rsm mean values (μm) and standred deviation for Herculite XRV Ultra, Filtek Z250, Tetric Evo Ceram, and Venus.

A-before polishing, **B**-after polishing, **C**-after brushing.

3.4.1 Effect of polishing and toothbrushing abrasive

T-test for paired data exhibited that the polishing and brushing procedures had an effect on the surface roughness of all tested materials. For each material there were significant differences between the following

- Surface roughness Ra before polishing and surface roughness after polishing.
- Surface roughness Ra after polishing and surface roughness after brushing.
- Rt before polishing and Rt after polishing
- Rt after brushing and Rt after brushing

3.4.2 The differences in surface roughness between the materials

One way (ANOVA) with Tukey's post-hoc test ($\alpha=0.05$) did not show any significant differences in Ra, Rt, and Rsm mean values after polishing between the materials tested. Surface roughness of the resin-composites tested had a tendency to decrease with the polishing procedures as shown in Figure 3.4.

Tooth brushing abrasion was responsible for increase the roughness absolute values, of all polished resin-composites, with statistical difference for the mean values of Herculite XRV Ultra, Filtek Z250, Venus, and Tetric Evo Ceram. For Tetric Evo Ceram and Herculite XRV Ultra the effect of tooth brushing abrasion was the higher, with the worst performance for Tetric Evo Ceram Figure 3.4.

Also the results show significant differences for Rt values and Rsm values after brushing. The highest Rt values were for Tetric Evo Ceram and Herculite XRV Ultra and the lowest values were for Venus. Herculite XRV Ultra exhibited the highest Rsm values, while Filtek Z250 the lowest values.

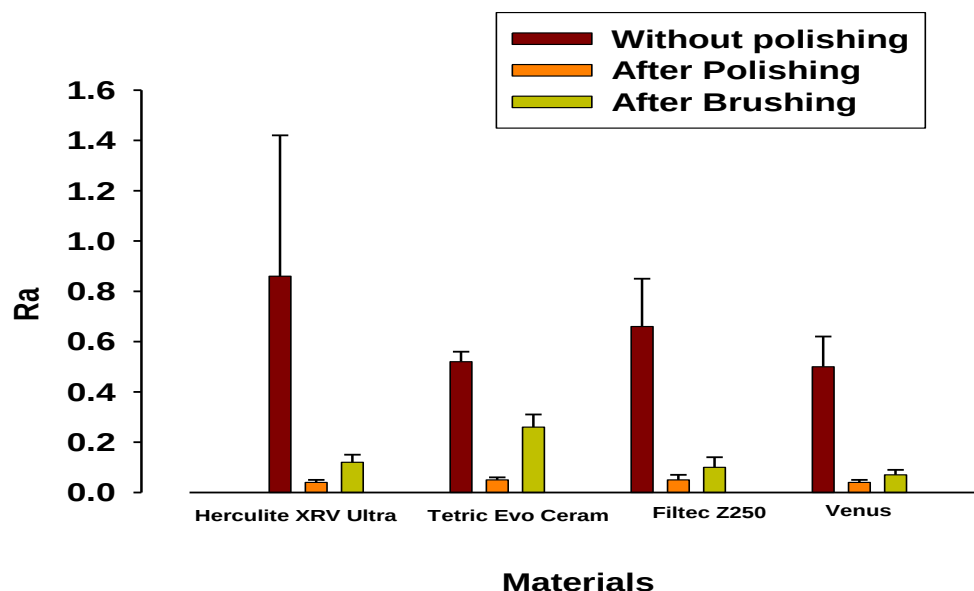


Fig 3.4 Error bars showing surface roughness mean and standard deviation for materials tested

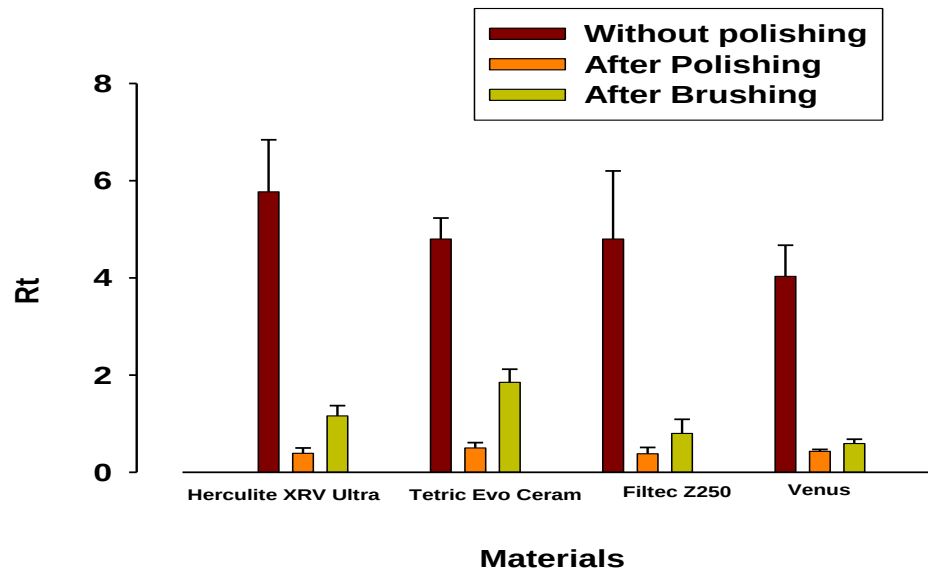


Fig 3.5 Error bars showing Rt mean and standard deviation for materials tested

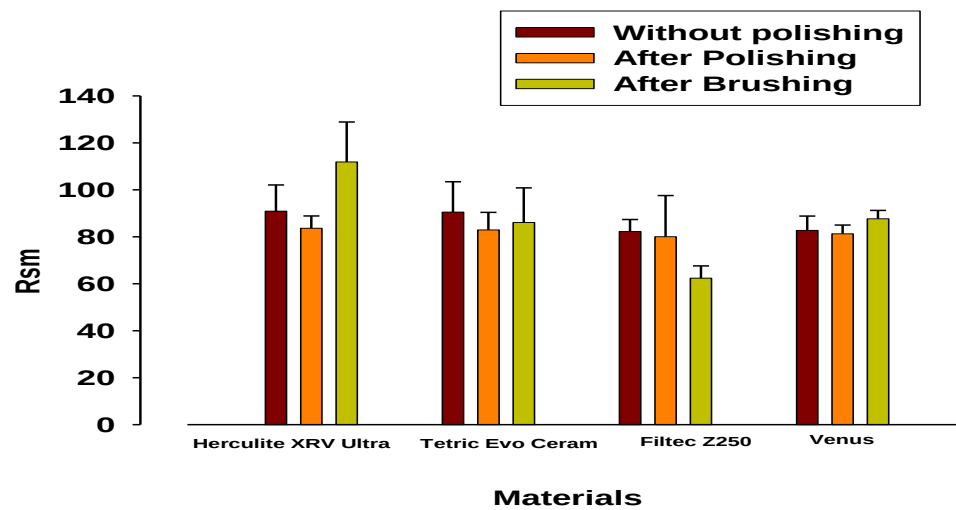


Fig 3.6 Error bars Rsm mean and standard deviation for materials tested

3.5 Gloss measurements results:

Gloss mean values and standard deviations for all tested materials are presented in **Table 3.6**

Materials	gloss before polishing		gloss after polishing		gloss after brushing	
	Mean	Sd	Mean	Sd	Mean	Sd
Herculite	8.53	1.501	74.850	4.530	44.852	0.934
Tetric	4.28	0.421	68.296	0.715	22.82	0.778
Feltik	4.48	0.773	60.100	0.678	9.392	0.320
Venus	6.832	0.199	52.284	0.363	42.576	2.025

Table 3.6 Gloss mean values and standard deviation of the materials tested before polishing, after polishing, and after brushing

3.5.1 Effect of polishing and toothbrushing abrasive on gloss:

T-test for paired data exhibited that the polishing and brushing procedures had a significant effect on the gloss of all tested materials. For each material there were significant differences between the following

- Gloss before polishing and Gloss after polishing.
- Gloss after polishing and Gloss after brushing.

For all materials tested the gloss results exhibited that the polishing increases the gloss and the brushing decrease the gloss.

3.5.2 The differences in surface gloss between the materials:

One way (ANOVA) with Tukey's post-hoc test ($\alpha=0.05$) illustrated that there were significant differences in the surface gloss mean values before polishing between the materials tested except between Tetric Evo Ceram and Filtek Z250.

After polishing and brushing there were significant differences in mean values of surface gloss between all materials.

The surface gloss mean value of Herculite XRV Ultra was the highest (74.85) after polishing, and the material was the best one in retaining gloss after brushing, while the

lowest surface gloss mean value was recorded by Venus after polishing (52.28), but Filtek Z250 was the wares material in retaining gloss after brushing.

3.6 3D surface roughness results:

Sa, St, Sds mean values and standard deviations for all tested materials are presented in **Table 3.7**

Materials		Sa		St		Sds	
		Mean	Sd	Mean	Sd	Mean	Sd
Herculite	A	6.73	1.92	74	14.5	944	14.07
	b	10.46	1.04	136.50	51.62	862	18.38
Tetric	A	7.925	0.15	82.60	4.81	898.5	51.62
	b	11.2	.707	107.1	11.17	802.5	30.41
Filtek	A	9.195	0.98	92.35	8.98	1034	4.24
	b	10.65	.778	140.5	31.81	815	16.97
Venus	A	16.9	1.27	125	35.36	937	5.6
	b	19.4	.141	148.5	45.69	786	46.67

Table 3.7 Sa, St, and Sds mean, and standard deviation values for all materials tested
A: the values after polishing, b: the values after brushing.

One way (ANOVA) with Tukey's post-hoc test ($\alpha=0.05$) showed that after polishing and after brushing there was significant differences between venus Sa mean value and all other material testes $P \leq 0.05$. Venus Sa mean value was the roughest material (16.9) after polishing and (19.4) after brushing, the other materials exhibit no significant differences in the mean values $P \geq 0.05$.

After polishing and after brushing St Mean values did not exhibit significant differences between all materials $P \geq 0.05$.

Also there was no significant differences in Sds mean values after polishing between the materials except for Tetric Evo Ceram VS Filtek Z250 $P=0.044$, but after brushing there was no significant differences between all materials $P \geq 0.05$.

3.7 3D surface v roughness (Sa) and 2D surface (Ra) Correlation:

The correlation between the 3D surface roughness (Sa) and the 2D surface roughness (Ra) for all tested materials after polishing and after brushing are plotted in **Figure 3.7**

After polishing There were positive correlation between Sa and Ra $r^2=0.9$, and after brushing also there was positive correlation $r^2=1$.

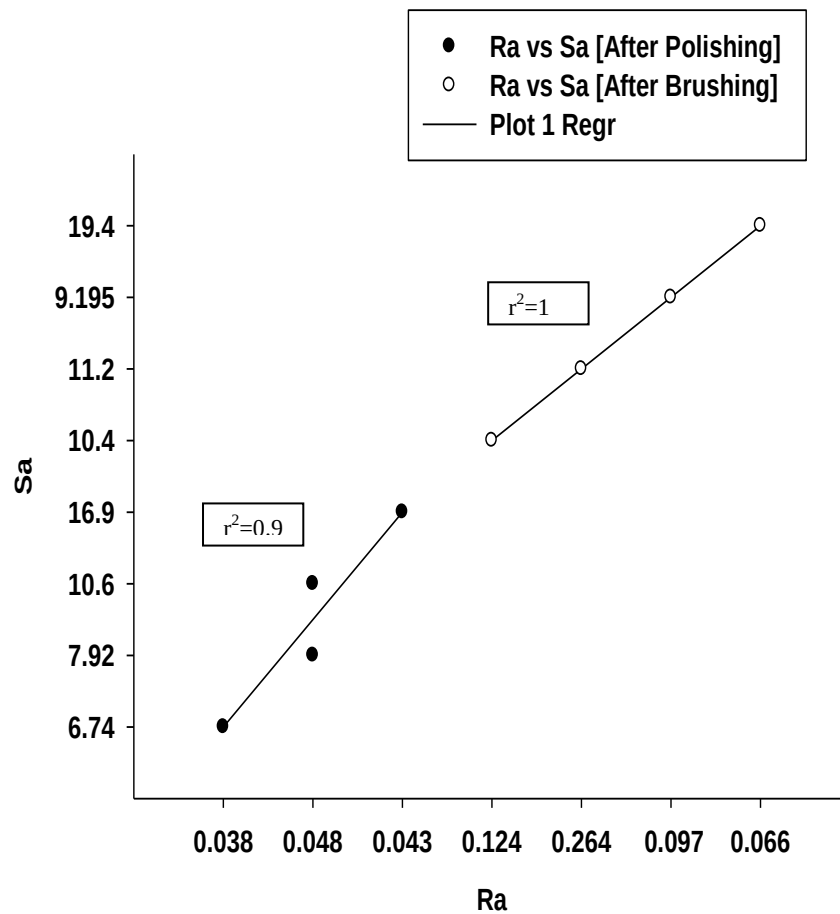


Figure 3.7 Correlation between 3D surface v roughness (Sa) and 2D surface (Ra) After polishing and after brushing.

Chapter 4

Discussion

4. Discussion

4.1 Mechanical properties:

Compressive strength and flexural strength (FS) are measures of the material strength under different force condition. The stronger material has the higher value of compressive strength and flexural strength.

International Organization for Standardization (ISO) specification 4049 for polymer-based restoratives classifies dental polymer based restorative materials into two different types. The first type is type I which is the material claimed by the manufactures to be appropriate for restoration involving occlusal surfaces. The second type is type II which includes all other polymer-based filling materials. The lowest value of flexural strength required for type I is 80 MPa and 50 MPa for type II {ISO, 2000}. In this study all materials investigated showed higher mean flexural strength values than ones recommended by the ISO suggesting that these materials can be used as direct restorative materials.

Weinmann *et al.* {Weinmann, *et al*, 2005} observed a higher mean FS value for composite Filtek Z250 (166 MPa) than that observed in the present study (136.7 MPa), which in turn were higher than the mean FS value reported by Palin *et al.* (92 MPa) {Palin, 2003}. However, Studies conducted by {Borba, 2009} {Rocha, *et al*, 2006} {Lien, *et al*, 2010} reported mean values 135.4 MPa, 134.5 MPa, and 153 MPa respectively, which was in agreement with the result of this study.

Herculite XRV Ultra exhibit FS mean value (83.37 MPa) less than the mean values reported by Tran, 2010 (137 MPa) {Tran, *et al* 2010}. Also Tetric Evo Cerm exhibite mean value (70.21 MPa) which was less than literature means (98.61) {Fischer, *et al*, 2010; Azzam, 2010; Ilie, and Hickel, 2009; Ilie, and Hickel 2009a; Lendenmann, 2006}. The differences between mean values might be attributed to the test condition such as crosshead speed, temperature, storage time and storage condition.

In the present study materials evaluation, ranking of flexural strengths from lowest to highest determined by three point bending test was as the follows: Tetric Evo Ceram< Venus<Herculite XRV Ultra<Filtek Z250. The results can be explained by the differences in filler content of the composites. Many Studies have reported the relation between mechanical properties and volume fraction of fillers {Braem, *et al*, 1989;

Chung, and Greener1990}. Composites with higher filler content such as Z250 and Herculite XRV Ultra are expected to be stronger than those with lower filler content.

The low FS of Herculite XRV Ultra could be attributed to the fact that PPF are not well bonded to the polymer matrix, so they become debonded and dislodged under high stress.

The significantly lower FS observed with Tetric Evo Ceram (83.75) in spite of its high filler content may be attributed to composition of the filler. PPF and Ytterbium trifluoride which incorporated into Tetric Evo Ceram for fluoride release might be related to low FS.

Compressive strength testing is important in vitro analyses that have typically been considered good indicators for simulating the forces that the restorative materials are subjected under mastication {Anusavice, 2003; Powers, and Sakaguchi 2006}. High compressive strength materials translate to persistent resistance against a heavy load, especially when used as a posterior restoration. As shown from this study, the microhybrid, Filtek Z250, demonstrated the highest compressive strength CS, while the other materials tested performed modestly.

For a Filtek Z250 study by Mitra *et al.* (2003) they observed higher values for compressive strength (454 MPa) than that in the present study (405 MPa). Other studies by Lien, et al, Manolea, et al and Dentsply, reported mean values of (390 MPa, 369.7 MPa, and 380 MPa) respectively which were less than the mean values of the present study. Overall the mean value of the present study was in agreement with the literature mean value {Mitra, *et al* 2003; Lien, *et al* 2010; Densply, 2003; Manolea, *et al* 2009}.

Herculite XRV Ultra exhibited compressive strength values significantly less than the results obtained by (3M ESPE, 2010) (440 MPa) and approximated to the result reported by (Tran, 2010) (349 MPa). {3M ESPE, 2010} {Tran, et al, 2010} Over all microhybrid composite material had higher compressive strength than nanohybrid composites.

Flexural modulus describes stiffness, a measure of the resistance to deformation under load of the material, with a high number indicating greater stiffness. There are debates on what magnitude of modulus resin-composites should possess. {Choi, *et al*, 2000} {Leinfelder, *et al* 1998} The ideal value should be similar to that of tooth structure, so that the restoration could have similar deformation with the surrounding tooth structure under load. When compared to the moduli of human enamel and dentin, which are about 84 GPa and 14 GPa, respectively, resin composites had much lower values. In contrast,

dental amalgam and gold, whose moduli are about 50 GPa²³ and 90 GPa, respectively have successfully served as posterior restorative materials for quite long time {Lu, *et al* 2006}. Filtek Z-250 yielded higher flexural modulus values (7.9 GPa) which means they are less flexible materials, other materials tested exhibited less FM mean values (5.63 GPa, 5.45 GPa, and 4.96 GPa).

The results showed statically significant differences between Filtek Z250 and Venus. Even though these two materials are microhybrid composite, they have different chemical compositions. Venus contains BisGMA and TEGDM, and Z250 contain Bis GMA, UDMA and BisEMA.

The composition of monomer has an effect on the mechanical properties of resin composites. Studies reported that flexural strength increases when BisGMA or TEGDMA are substituted by UDMA. Furthermore, a reduction in flexural strength was observed when BisGMA was substituted by TEGDMA {Asmussen and Peutzfeldt 1998}. This is an isolated factor that could explain the results of present study.

The presence of TEGDMA in the matrix composition of composite resin has been linked to a significant decrease in the flexural strength of the material, whereas, it has also been associated to an increase in the modulus of elasticity {Asmussen, and Peutzfeldt 1998}. The characteristic flexibility of TEGDMA allows the creation of a dense and flexible polymer network {Sideridou, *et al*, 2003} that increases the composite elastic deformation. Although the result of present study showed no differences in the mean values of flexural strength and flexural modulus for composites presenting TEGDMA in their composition indicating that, the mechanical properties result from a complex combination of microstructural and compositional factors that vary in the manufacturing process and that cannot be considered in an isolated way {Adabo *et al*, 2003; Asmussen, and Peutzfeldt 1998}.

Overall, Z250 microhybrid composites had higher CS, FS, and FM than other materials. These higher properties can be explained by their higher filler loadings.

The filler content could be an important factor affecting physical and mechanical properties of different composite materials. Li, *et al*. 1985 found that increasing the filler content lead to increase hardness, compressive strength, and stiffness, while water sorption decreased {Li, *et al*, 1985}.

Other studies also investigated the relationship between the mechanical properties of composites and the difference in filler volume. The authors reported that the materials with higher filler volumes exhibit better mechanical properties {Carreiro *et al*, 2004; Manhart *et al*, 2000}. These observations seem to be not confirmed by the results of this study, especially when Tetric Evo Ceram which have filler content 68% by volume did not exhibit better mechanical properties compared with Filtek Z250 60% by volume. The composite resins with higher filler contents, Filtek Z250 (84.5wt %), showed significantly higher mean FM, FS, and CS, but Herculite XRV Ultra (97wt %) did not show high mean values. Therefore, we can conclude that the filler content with other factors such as filler size, composition, morphology, and amount of initiators and the quality of silanization can also contribute to the development of physical and mechanical properties.

The filler size of commercial composites has continuously decreased over the years from the traditional to the nano-hybrid materials in order to achieve aesthetics properties. But the larger surface area to volume ratio of the fillers present in the nanofilled materials also tend to increase the water uptake and that lead to degradation of the filler/matrix interface affecting the mechanical properties when compared to a microhybrid composite (Filtek Z250) {Curtis, *et al* 2008}.

In addition The morphology of the fillers has a great effect on the properties of composite resin, as they have been shown to be determining factors in both the filler loading {Adabo, 2003; Kim, 2002; Sabbagh, 2004}, and the material strength {Adabo, *et al* 2003; Kim, *et al* 2002} filler with smooth spherical shaped tend to increase volume fraction of the filler due to the improved packing of the particles and also to higher fracture strength. This could explain the high flexural strength and modulus and compressive strength obtained with Filtek Z250, which contain small round shaped particles. Furthermore, the mechanical stress tend to distribute more uniformly with rounded particles rather than with irregular shaped particles that present sharp angles, already known as stress concentration areas from where the cracks may start {Sabbagh, *et al* 2004}.

4.2 2D surface roughness measurements:

Now a day many method are available to evaluate the surface texture of any materials including contact stylus tracing scanning , electron microscopy , laser reflectivity, non contact laser metrology and compressed air measuring. The most common method is the contact stylus tracing {Wennerberg, et al, 1996}. This method was used in the present study as it was fast, simple and reliable for comparative assessment of surface roughness properties.

It has been established previously that the resin composite surface quality is material and polishing procedure related. In the current study, the same polishing system was applied for all the materials, to avoid any differences that might be caused by different polishing systems. Optic discs polishing system was the system of choice. Regarding the experimental procedure, every effort was applied to standardize the polishing, in terms of the number, direction and duration of the strokes; also one operator prepared all samples.

The surface roughness against the polyester matrix film has been reported by many studies to be the smoothest surface for most tooth coloured restorations. Although, this surface is polymer rich making it quite unstable. The polymer rich layer is commonly clinically removed by finishing and polishing which produce surface irregularities that increase the roughness to the polished surface of varying degrees depending on the polishing systems and materials used {Jung, *et al*, 2007 }. In the present study the resin rich layer that form a smooth surface was removed by grinding with grit paper.

In this study vertical roughness parameter such as Ra, Rt are used to describe the surface irregularities by their amplitudes only. The Ra parameter is only used by many researchers to estimate the surface quality of composite resin. Also other parameter was used which is spacing parameter Rsm that measure the horizontal feature of the surface.

Finishing and polishing of composite resin play a critical role to enhancing the aesthetic and longevity of restored teeth {Jefferies, 1998}. Finishing refer to contouring of the composite whereas polishing refer to reducing the roughness produced by finishing

instrument {Yap, *et al*, 1997}. Poorly polished composite are susceptible to pigmentation, plaque adhesion, gingival irritation and recurrent caries {Weitman, and Eames, 1975; Shintani, *et al*, 1985; Strassler, and Bauman, 1993}.

According to Chung {Chung, 1994}, the Ra value determined by 2D profilometry was less than 1 μm when the composite surfaces were visibly smooth. On the other hand, if 2D surface roughness (Ra) were above 0.2 μm , it exceeded the clinically acceptable threshold for composite resin restorations {Bollen *et al*, 1997}. According to Bollen *et al.*, higher Ra values were accompanied by increased plaque accumulation and higher risk for dental caries and periodontal diseases.

Carbide paper creates peaks and valleys on the surface of the specimen. Consequently polishing, the peaks are abraded off rather than the valleys on the surface. The least square line is again fitted to the data to obtain the mean line when calculating the new (Ra), and therefore the new mean line is different from the previous one {Watanabe, *et al* 2005}.

In the present study at the base line the surface roughness of the materials ranges from 0.49 μm to 0.86 μm which was greater than the clinical threshold surface roughness for plaque accumulation, whereas Ra values after treatment with finishing and polishing system were generally less than 0.2 μm which range from 0.043 μm to 0.05 μm , which is clearly illustrate the effect of finishing and polishing on the materials tested. Also, it was observed that all materials exhibited close results for Ra, Rt, Rsm valuses, before and after finishing and polishing procedures. The differences in the results were after brushing.

When using the same finishing and polishing system for different resin composites, differences between materials compositions should be responsible for different (Ra) values {Watanabe, *et al* 2005}. Materials with larger filler size are generally showing higher surface roughness than those materials with smaller filler size {Tjan, and Chan 1989; Senawongse, and Pongprueksa 2007}.

Although Herrgott *et al* {Herrgott, *et al*, 1989} reported that surface roughness of finished resin-composite materials was not dependant to the size of filler particle. More recent study confirmed that surface roughness of finished resin-composite materials was dependant to the size, shape and type of filler particles {Marghalani, and Pongprueksa

2010}. Marghalani noted that the surface roughness value increased with increase the filler particle and with irregular shape filler.

In oral cavity, resin composites have to be able to endure toothbrushing that causes wear to the materials {Yap, *et al*, 2004 a}. Materials that can resist the wearing process and maintain a comparatively smooth surface when compared with the surface against matrix are preferred {Weitman, and Eames 1975; Shintani, *et al*, 1985}. In this study, toothbrush abrasion increased all roughness values, which is in accordance with some other studies {Takeuchi, *et al*, 2003; Cho, *et al*, 2002}.

The profilometer results after toothbrushing abrasion indicate that, of the materials tested; Herculite XRV Ultra, Filtek Z250, and Venus could be expected to withstand the wear caused by brushing. All these material exhibited Ra values less than 0.2 μ m. Roughness values greater than 0.2 μ m might result in a simultaneous increase in plaque accumulation, increase the risks of secondary caries and periodontal inflammation {Bollen, *et al*, 1997}. Tetric Evo Ceram Exhibit highest Ra values more the 0.2 μ m, this result might attribute to the present of prepolymerized filler. The loss of PPF from the nanohybrid resin composites after brushing has been reported previously {Endo, *et al*, 2010; Senawongse, and Pongprueksa, 2007}. The disruption of the filler matrix interface from the loss of PPF may explain the significantly greater Ra values observed. The second possible explanation for Tetric Evo Ceram high surface roughness that irregular particles tend to have higher surface roughness as that the irregular particle tend to protrude from the surface and from the microstructure perspective, the stress concentration around the irregular filler may lead to their pull out from the surface, thus increasing the roughness of the materials.

After brushing the highest roughness value presented by the horizontal parameter (Rsm) was recorded by Herculite XRV Ultra which expresses low vertical roughness (Ra) compared with Tetric Evo Ceram. The small size filler provided less vertical dimension, however they can result in filler agglomeration which may be responsible for increasing the horizontal dimension of the roughness profile.

Nanocomposites were introduced with the so-called advantage of increased polish and gloss retention, as only small particles would be dislodged during wear, leaving the

surfaces with defects smaller than the wavelength of light {Mitra, *et al*, 2003}. nanohybrids take the approach of combining nanomeric and conventional fillers, thus, nanohybrids may still suffer from the loss of large particles. Therefore, it becomes questionable whether the similar material can be defined as a “nanofiller loaded resin composite”.

Both materials wear and surface profile after abrasion have been previously reported {Garcia, *et al*, 2004; Kanter, *et al*, 1982; Wang, *et al*, 2004}. No correlation was found between the filler size and the surface roughness after toothbrushing {Heintze, *et al*, 2005}. study by Moraes *et al* {Moraes, *et al*, 2008} reported that nanohybrid composites shows more wear resistance and less roughness than microhybrid. In this present study nanohybrid composite recorded the highest surface roughness than microhybrid composites. The materials assessed in this study present different outcome for surface change, and it is clear that the mechanism according for these phenomena is more complex than can be explained by filler components alone.

4.3 3D surface roughness measurements:

At the present time, 3D surface roughness measurement not yet covered by international standards. Although the literature for composite resins to compare 2D surface roughness values obtained from the present study with other investigation are available, such study for 3D Sa parameter is not yet available.

The results of the current study reported that the polishing and brushing have significant effect on Sa values for all the material tested. The values of St did not show any significant difference between the materials after polishing and after brushing, on the other hand there was a significant differences between the materials tested in Sds values after polishing. Also the study revealed that after polishing there were positive correlation between Sa and Ra $r^2=0.9$, and after brushing also there was positive correlation $r^2=1$.

In general, the surface roughness measurements by contact stylus profilometers facilitate a quantitative measure of the surface irregularities. In the current study, the surface roughness was assigned by Ra parameter. Although Ra is the most commonly

recorded value to verify surface roughness in dental materials, it is considered as a poor indicator of surface texture. The Ra parameter does not fully describe the surface roughness and is considered as an inadequate for describing the specific character of material roughness. The 3D surface measurements can represent the natural characteristics of a surface, while 2D surface measurements do not achieve this {Abu-Bakr, *et al*, 2001; Marigo, *et al*, 2001}.

3D surface parameters are more realistic than those obtained from 2D profiles. The information that can be obtained by 3D measurement gives a complete description of surface topography and is more comprehensive than the 2D measurement. Stylus type profilometers used in the current study give definitions of surface features for a scale size related to the probe dimensions.

Due to the radius of the tip (5 μm), the sensitivity of profilometer is low, as the tip could not penetrate deep valley because of its size {Wassell, *et al*, 1994} and could not represent surface features, which were narrower than the stylus tip size. This problem results in underestimation of the surface roughness.

Comparing the results of 2D profilometer Ra with 3D Sa, 2D profilometer Ra values were lower than 3D Sa which might be partly attributed to the smaller size of the area examined. Moreover, the 2D surface profilometer determines roughness in either horizontal or vertical directions, while 3D surface roughness parameter identifies area roughness monitored on a whole surface. It is therefore unwarranted for Ra values from a 2D profilometer to be compared with 3D Sa parameter.

4.4 Surface gloss:

Gloss also is an essential property which plays an important role in the aesthetic appearance of composite restorations and their blending to surrounding teeth {O'Brien, *et al*, 1984}. High gloss minimizes the effect of a colour difference between a composite resin and adjacent enamel. The colour of reflected light is predominant rather than the colour of the underlying composite {O'Brien, *et al*, 1984}. Many studies have revealed that there was a strong correlation between surface gloss and surface roughness {Lu, *et al*, 2005; Heintze, *et al*, 2010}. It has been reported that when the surface roughness is increased, the degree of random reflection of light will increase, therefore

the gloss decreased {Watanabe, *et al*, 2006}. However this was not observed in this study. Even though no differences were noted between surface roughnesses of the materials after finishing and polishing the gloss of the materials differed significantly. In this study, toothbrush abrasion decreased all gloss values, which is in accordance with other study {Lee, *et al*, 2005}. The highest gloss was achieved by Herculite XRV Ultra and Tetric Evo Ceram and the lowest gloss was with Venus.

Diffuse reflection decreases with small filler particles and the surface looks glossy, {Takanashi, *et al*, 2008}. According to this statement it might be expected that Herculite XRV Ultra with a smaller filler size would have showed glossier surfaces than other materials. The results of this study were in accordance with this statement and Herculite XRV Ultra illustrates the highest values for gloss.

It might be expected that smoother surfaces would demonstrate higher gloss values. Lu et al {Lu, *et al* 2005; Lee, *et al* 2005} stated that the gloss was directly influenced by the surface roughness. On the other hand, Lee, et al. 2005 found that the gloss was not only influenced by the surface roughness but also by other factors such as the difference in refractive indices of the resin matrix and the fillers. In the present study, Tetric Evo Ceram exhibited the highest surface roughness but it did not exhibit the lowest gloss after toothbrush abrasion, therefore, it might be concluded that the composition of the material rather than the roughness might have an effect on the gloss. Heintze et al {Heintze, *et al*, 2006} also stated that the gloss was material dependent.

4.5 Conclusion:

While nanohybrid composites may have the advantage of higher surface gloss over microhybrid resin composites, their mechanical properties were not superior to those of the microhybrid composites examined. Although, the surface gloss of Herculite XRV Ultra and Tetric Evo Ceram were the highest, Filtek Z250 exhibited the best mechanical properties (Flexural strength, flexural modulus, and compressive strength).

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