

EVALUATION OF SECOND GENERATION
INDIRECT COMPOSITE RESINS

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ABSTRACT

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Evaluation of Second Generation Indirect Composite Resins

Indirect composites were introduced so that the composites can be cured extraorally to improve the degree of conversion and other material properties. These materials are indicated as long term full coverage dental restorative materials. However the mechanical and physical properties of new Second Generation Indirect Composites for this particular application have not been fully evaluated. The purpose of the study was to compare the appropriateness of the four commercially available laboratory composite resins for application as long term full coverage restorative materials. Water solubility and sorption levels, staining resistance, gloss, surface roughness, wear due to tooth brush abrasion, two-body and three-body wear, fracture toughness and radiopacity of four indirect composite restorative materials; Radica (Dentsply), Sculpture Plus (Pentron), Belleglass-NG (Kerr) and Gradia Indirect (GC America) were determined.

The results showed that the four composites differed significantly from each other. Belleglass-NG and Gradia Indirect showed negative water solubility. All the four groups demonstrated less color stability when exposed to coffee slurry for 3 weeks. Significant decrease in gloss and volume occurred when the composites were exposed to simulated tooth-brush abrasion. Sculpture Plus

demonstrated lowest abrasion and attrition wear resistance among the four indirect composites. Radica had the highest fracture toughness and radiopacity of all the composites with values close to or less than dentin.

In conclusion, different indirect composite systems possessed different mechanical and physical advantages when compared to each other. In general, Belleglass-NG demonstrated superior advantages due to its higher abrasion and attrition wear resistance and stain resistance. This was followed by Radica, Gradia Indirect and Sculpture Plus.

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INTRODUCTION

Dental restorative composite materials can be divided into direct composite (directly placed into the oral cavity and cured) and indirect composite (externally fabricated and cured by means of light and / or heat). Indirect composites are also referred as Prosthetic composites or Laboratory composites. In an effort to offset the problems of marginal integrity associated with direct composites, the first generation of these indirect composites were introduced in the 1980's.¹ These materials exhibited low mechanical properties owing to a low percentage of inorganic filler particles and a high percentage of exposed resin. In the 1990's several new indirect composite resins came into the market. These indirect composite resins had higher percentage by volume of inorganic fillers (approximately 66%) and exhibited better mechanical properties. These materials are referred as Second Generation Indirect Composites.

Second generation indirect composites are indicated in several clinical applications such as inlays and onlays, laminated veneers and jacket crowns, implant-supported restorations, for progressive loading of implant-supported prosthesis and for easier repair directly into the mouth.² When compared to the direct composite restorations, the indirect composite technique offers a better potential for generating appropriate anatomic form, as well as proximal contacts and contours³, excellent occlusal morphology and good marginal accuracy.

Comparison with ceramics reveals that mechanical properties and strength of the indirect composites are much inferior. However, indirect composites supplement and compliment rather than replace ceramic restorations as suitable alternatives in some clinical situations. Some of these situations are coronal restoration of dental implants. As ceramics exhibit a high modulus of elasticity and absorb little of the masticatory energy, considerable amount of the masticatory force is transmitted to the implant and the periosseous structure reducing the longevity. Polymers become the materials of choice in this case because they have relatively low modulus of elasticity and absorb the occlusal stress. For patients with poor periodontal structures requiring occlusal coverage, stress absorbing materials like indirect composites are indicated.³

A review of the literature suggests contradictory opinions exist about indirect restorations. Some authors have suggested that indirect composites offer no distinct advantages over the direct composites.^{4, 5} Others have observed improved wear resistance in indirect composites.⁶ In order to overcome some of the drawbacks, these composite systems are featured with newer formulations of resins and fillers and different curing mechanisms. Curing with light and heat is conducted in a vacuum and nitrogen atmosphere to prevent oxygen incorporation into the composite. Few independent and standardized studies on physical and mechanical properties and clinical performance of these second generation indirect composites are available. Also the manufacturer's information cannot be directly compared because of the different methodologies used.

In this study properties of four new second generation indirect composite restorations were investigated. These properties were water-sorption and solubility, staining resistance, gloss and surface roughness after simulated toothbrushing, volume loss as a result of wear when subjected to simulated toothbrushing, three-body Alabama wear testing machine and two-body pin-on-disc test, plane strain fracture toughness and radiopacity.

LITERATURE REVIEW

Indirect Composite Resins

Restorative dentistry has been revolutionized with the introduction of resin composites. The material was introduced nearly four decades ago⁷ and was widely used for both anterior and posterior teeth as a direct restorative material. The use of this material however has been limited to small and incipient lesions and to less stress bearing areas because of the material's inadequate wear resistance, low strength and lack of marginal integrity. They are also more technique sensitive when compared to other restorative materials like amalgam and require greater attention to detail during the insertion phase. Increasing the filler content improved the strength and reduced the polymerization shrinkage. This increased the materials efficiency to be used in posterior areas but it still encounters difficulty in building the proximal contacts and contours directly in the oral cavity.^{8,9}

The indirect composite inlay technique was introduced by Mormann¹⁰ in Germany and Touati¹ and Pissis¹¹ in France. The technique provided the ease of fabrication, reduction in marginal shrinkage and efficiency in building the proximal contacts and contours. Fruits et al¹² showed that restoration with indirect resin was associated with less microleakage than the direct resin groups. Lutz et al¹³ showed improvements in wear resistance of a heat cured formulation over light cured and chemically cured formulations. Wendt¹⁴ reported increase in diametral tensile strength and hardness without a decrease in compressive strength and

modulus when light cured composites were further subjected to heat treatment of 10 minutes at 100°C to 200°C. Cook and Johansson¹⁵ showed an increase in diametral tensile strength, flexural strength and fracture toughness of composites post cured at 100°C for 24 hours. Ferracane and Condon¹⁶ studied the fracture toughness, elastic modulus and surface hardness of composite after 3 different post-curing light treatments. They reported increase in fracture toughness and modulus of elasticity but changes in surface hardness was inconclusive. They also correlated the increase in degree of conversion with the enhancement in mechanical properties. Relaxation of internal stresses at the filler-matrix interface is another outcome of post-cure heat treatment that may improve adhesion between resin matrix and fillers and improve the mechanical properties.

Several clinical studies were conducted to validate the efficacy and the longevity of the indirect composite restorative materials. Wendt et al⁴ investigated the clinical performance of a heat-treated composite resin inlay, using both the direct and indirect methods of clinical evaluation. No significant differences in wear could be measured between conventional light-cured inlays and those with secondary dry heat treatment. Bartlett and Sunderam⁵ conducted a 3-year randomized clinical study comparing indirect and direct resin composites used to restore worn posterior teeth. Their results indicated no significant difference between the two groups. The study also suggested that the use of direct and indirect resin composites for restoring worn posterior teeth is contraindicated. Pallesen and Qvist¹⁷ conducted a 11-year randomized clinical study evaluating the clinical performance of indirect inlays. The most common imperfections of

inlays were wear of the luting composite, marginal discoloration, and lack of color match. The study concluded that additional oven curing had only minor influence on fracture resistance and did not improve the wear resistance of resin inlays compared to direct composite fillings.

Indirect composite resins were observed to overcome the disadvantages of all-ceramic crowns and inlays related to clinical failure due to fracture and long laboratory procedures. Composite inlays were both cheaper and more user-friendly than ceramic inlays.¹⁸ Bilsen Kaytan et al¹⁹ performed clinical evaluation of indirect resin composite and ceramic onlays over a 24-month period. The study observed better color match for ceramic onlays than for the indirect resin composite onlays. Both ceramic and indirect resin composites displayed marginal deterioration over 24 months. Thordrup et al²⁰ conducted a ten year prospective clinical study of indirect and direct composite and ceramic inlays. After ten years of observation, ceramic inlays showed higher survival rate than indirect composites. Survival rates of all the types of materials used were considered clinically acceptable. It must be noted that survival rates were within the range of survival for direct composite restorations. The main reason for the failure was fracture and secondary caries.

Most of these clinical studies investigated the earlier version of indirect resin composites which were also termed as first generation indirect composites. These first generation composites were microfilled with flexural strength ranging from 60 MPa to 80 MPa and elasticity modulus ranged from 2000 MPa to 5000 MPa. The resin volume was higher than 50% and micro particles were small

(0.04 μm).^{1, 2} In mid-1990's, second generation laboratory composite resin systems were introduced. The ratio of inorganic filler in volume was approximately 60% to 70% and exhibited flexural strengths between 120 and 160 MPa with an elasticity modulus of 8500 to 12,000 MPa.² There were different types of processing methods used in this new version of second generation indirect composites. Belleglass HP (KerrLab Corporation) used heat and nitrogen atmospheric pressure, Cristobal Plus used slow cure light and heat.³ Nitrogen Pressure eliminates internal oxygen before the material begins to cure. Elimination of oxygen prevents inhibition of polymerization, voids and microscopic inclusions of air, and thus influences degree of conversion, esthetics, wear and abrasion.³

Kakaboura et al²¹ compared various characteristics of two second generation laboratory composites. Degree of conversion, microhardness, roughness, biaxial flexural strength and polymerization shrinkage strain were evaluated. The two materials (Belleglass HP and Symphony) differed in composition and process of curing. Significant differences were found between the two materials in all the properties. Belleglass exhibited higher degree of conversion, surface microhardness, biaxial flexural strength and increased roughness. Mandikos et al²² compared the wear resistance and hardness of second generation indirect composite resins to a first generation indirect composite. Their results showed that the first generation indirect composite resin had higher wear resistance and hardness than the second generation resins.

The numbers of the in-vitro studies and prospective clinical studies evaluating these second generation indirect composites are inadequate. Indirect composites with newer formulations and processing techniques have come to the market. There is a need to investigate the physical and mechanical properties of these materials.

Water Sorption and Water Solubility

Water plays a major role in degradation and erosion of methacrylate-based composite resin materials. The process of water sorption is a diffusion controlled process and occurs largely in resin matrix.²³ The oral environment is moist and affects the composite resin materials resulting in deteriorated mechanical properties.^{24, 25}

Sorption of water may cause filler-matrix de-bonding and degradation of fillers.²⁶ Some of the fillers that are known to leach are silicon, boron, barium, sodium, lithium and strontium.²⁷ Other than the fillers, free monomers and ions are also known to leach out.²⁸ The monomer TEGDMA has been shown to be a significant monomer released.²⁹ Other constituent resin monomers like bis-phenol A glycol dimethacrylate (BISGMA), urethane dimethacrylate (UEDDMA), methyl methacrylate (MMA) high density dimetha-acrylate (HDDMA) and products like formaldehyde are also known to be released.^{30, 31}

Several factors are known to effect water sorption and solubility rates of the material. Decreased cross-linking promotes increased water sorption since the regions between the highly cross-linked regions, 'microgel agglomerates', are

increased which may accommodate for an increased diffusion of water molecules.³² Water lowers the glass transition temperature (T_g), which results in a decrease in thermal stability and polymer plasticization.³³

Oysaed and Ruyter²⁷ mentioned that high amounts of barium, strontium and zinc which are incorporated in composites to give radiopacity, result in high solubility. His study also demonstrated that size of the filler particles may also play an important role in determining the level of water sorption and solubility. Microfiller particles have a larger total surface area and thus have higher rates of water sorption and solubility.

Ulf Örtengren et al³⁴ studied the effect of pH and the storage time on the sorption and solubility behavior of the composite resin material. It was observed that long term solubility increased at pH of 8 and decreased at a pH of 4 and 6. The sensitivity of the sorption and solubility versus time and pH was related to the hydrophilicity of the resin matrix and chemical composition of the material used. Also a significant increase in solubility for all materials for 1-7 days period was observed. With prolonged time storage, negative solubility (i.e. mass increase) at 180 days of storage was noted. High water sorption was observed upto a seven day period but then there was negligible water increase in sorption. However, others³⁵ have suggested that seven days are not enough to determine the true hygroscopic behavior of contemporary composite materials as it may indicate only 50% of dimensional changes that eventually occur.

It has been argued that diffusion of water into the composites may have a beneficial effect, as it may compensate for the polymerization shrinkage and thus

reduce shrinkage stress and improve marginal seal. Due to this phenomenon of sorption of water by the dental composites, change in weight may be observed. The outward movement of fillers, ions and leached out material will contribute to loss in weight of the material. Conversely hygroscopic expansion of the material will lead to swelling of the material and increase in weight of the material.³⁵

Mode of curing and the intensity of light curing also affect the rate of water sorption and solubility.³⁶ Curing in light ovens under high intensity lights produces higher degrees of conversion and reduces un-reacted carbon-carbon double bonds thus reducing water sorption.

Staining Resistance

Staining resistance is a vital property for the longevity of a facing on a removable or fixed partial denture, a crown or direct restorations in esthetic areas.³⁷ In-vitro studies^{38, 39} have shown that resin based composites are susceptible to staining. Clinical studies have confirmed the in-vitro findings. Setz et al⁴⁰ conducted a double blind pilot study comparing two composites used in veneering telescopic dentures and observed significant discoloration after 1 year. Rosentritt et al⁴¹ measured the color stability of laboratory-made composite veneers with a reflection spectrophotometer and concluded that the discoloration in the test material was clinically unacceptable.

Causes for discoloration of dental composite restorations can be exogenous or endogenous.⁴² Endogenous reasons involve discoloration of resin matrix and the interface of resin matrix and fillers.³⁷ It occurs when the materials

are aged under various physical and chemical conditions such as thermal changes and humidity.^{39, 43} de Gee et al⁴⁴ identified the relation between staining and the degree of conversion. Inadequate staining will favor the sorption of some colorants. Oxygen related polymerization inhibition at the restoration surface and at the periphery of the porosities may induce composite discolorations.⁴⁵ Visible and ultra violet irradiation may affect the intrinsic color of composite material.⁴⁶

Exogenous reasons are adsorption and absorption of stains.⁴⁷ Several studies have reported the data about the staining of composites by coffee, tea and other beverages. Um et al⁴⁸ exposed the resin based veneering materials to boiled coffee and tea at 50°C and evaluated for color stability. He mentioned that discoloration of the materials occurred due to the sorption of the colorants into the organic phase of the veneering materials. Dietschi et al⁴⁹ compared the color stability of ten new-generation light cured composites. Several coloring solutions like coffee, E110 food Dye, vinegar and erythrosin were used. Specimens were subjected to thermocycling, post curing and polishing prior to staining. Erythrosin caused the greatest color change.

Earlier Asmussen⁵⁰ studied the various factors affecting the color stability of restorative composite resins. The study reported that light activated materials were more color stable than chemically activated materials. Type of amine, high concentration of inhibitors and type of polymers used influence the color stability of resin. Later Janda et al⁵¹ investigated the color stability of resin matrix restorative materials as a function of the light activation method. He reported that

the photo-initiator system was responsible for the yellow color in the resin composites. It is influenced by the intensity and the mode of the curing.

Scotti et al⁵² investigated the color stability of acrylic resins by simulating the oral condition through immersing the specimens in synthetic saliva combined with coffee, tea or chlorhexidine in a dark environment at 37°C. They concluded that synthetic saliva and coffee produced the greatest color change.

Gloss and Surface Roughness

Gloss is an attribute of visual appearance that originates from the geometrical distribution of the light reflected by the surface.⁵³ ASTM standards define gloss as “angular selectivity of reflectance, involving surface-reflected light, responsible for the degree to which reflected highlights or images of an object may be seen as superimposed on the surface”.⁵⁴ The concept of gloss has been taken from the paint industries where it is often applied.⁵⁵ Surface gloss affects the aesthetic appearance of the restorations.⁵⁶ Differences in the gloss between the restoration and the surrounding tooth structure can be detected even if the colors are matched.

Gloss may be influenced by a variety of factors such as filler size distribution, mechanical properties, index of refraction of the fillers present in the plastic and the viscosity of the resin matrix components.⁵⁷ Earlier O'Brien et al⁵⁶ established an inverse correlation between gloss and surface roughness. Da Costa et al⁵⁸ studied the effect of different polishing systems on surface roughness and gloss of various resin composites. The study reported significant

interaction between the composites and the polishing systems evaluated in terms of gloss. The particle size and types of abrasives used in the polishing systems as well as the time used for each polishing procedure influenced the gloss and surface roughness.^{58, 59} Heintze et al⁶⁰ studied the influence of polishing time and press-on force on the surface gloss and surface roughness of dental materials by using a three-component rubber based polishing system and reported that gloss and surface roughness were time dependent.

Lee et al⁶¹ observed significant changes in gloss after simulating generalized wear on resin composites. According to them, the resin matrix influences the gloss, but the phenomenon was more affected by the filler size and shape. Also spherical fillers reflected more light than irregular shaped fillers. Lu et al⁶² studied the effect of surface roughness on the stain resistance of dental resin composite and stated that discoloration increased with an increase in R_a .

Wear

Wear is a consequence of many fundamental processes. It may be defined as a progressive loss of substance from the surface of the body as a result of mechanical action.⁶³ Wear resistance is a pre-requisite for a dental material to be accepted by both patients and dentists. High wear resistance contributes to the longevity of the dental restorative materials and thus establishes durable function and esthetics of the restored teeth. Contrary, high wear rate may be related to elongation of antagonist, tilting and movement of teeth and other dysfunction.⁶⁴

Wear of composite resin materials has been evaluated in terms of two main clinical components: occlusal contact wear and contact free wear. Occlusal contact wear is a localized process while contact free wear is more generalized. Mechanical wear of composites occurs mainly by abrasive, adhesive and fatigue wear processes. Abrasive wear (2-body and 3-body) occurs when surfaces pass over one another and the harder material cuts the softer material, resulting in loss of structure. When the friction generated by two moving surfaces causes a local cold welding between the particles on the surfaces, and the small pieces are fractured off, the process is termed adhesive wear. When flaws in the composite become microcracks that propagate through the material, leading to the separation of surface particles, the resulting wear is called fatigue wear.^{22, 65} Wear resistance of a given material is determined by its formulation, the quantity and the size of the reinforcing fillers and the degree of the cure of the polymer matrix.^{66, 67}

Toothbrush and dentifrice abrasion can occur on any exposed tooth surface. However it is most commonly observed on the labial surfaces of the anterior teeth and buccal surfaces of the posterior teeth.⁶⁸ Teixeira et al⁶⁹ evaluated the in-vitro wear and surface roughness of two direct filling restorative composites at different cycles of tooth brushing. Abrasive wear and surface roughness increased with each cycle interval for both materials. Kon et al⁷⁰ studied the effects of occlusal and brushing forces on the wear of composite resins using three different wear tests: a simulated occlusal wear test, toothbrush wear test and a combined wear test which carried out a toothbrush wear test and

occlusal wear test alternatively. In all three wear tests, higher occlusal and brushing forces resulted in significantly greater volume loss and higher maximum worn depth. The authors also suggested that the different wear behaviors of the four composites most probably stemmed from the differences in their filler systems.

Gohring et al⁷¹ conducted a laboratory study to test the attritional wear and abrasive wear behavior of composite materials compared to wear behavior of human enamel. Both indirect and direct composite resins were used and natural human enamel was used as a control. Wear resistance of indirect composite resins were comparable to human enamel when loaded with enamel cusps. Surface disintegration caused by fatigue was observed under scanning electron microscopy. The study also reported that higher filler content of the composite was not synonymous with higher wear rate. Suzuki et al⁷² evaluated the wear rates and abrasiveness of indirect composite restorative materials compared with a type III gold alloy. The study reported that some indirect composite restorative materials (Sculpture, Belleglass and Cristobal Plus) had similar wear resistance as type III gold alloy. It was also reported that some of the antagonistic enamel was abraded by the composite material with high filler content.

Abrasion of tooth occurs in a three-body wear mode, and is generated by the sliding action of one tooth over another with force being transmitted through a layer of food that serves as a third-body medium.⁷³ Numerous studies have been conducted to assess the three-body wear of the dental composites. It is assumed that mechanical properties of polymeric composite materials crucially depend

upon the condition of the interface between the surfaces of the inorganic filler particles and the polymerized organic resin in which the filler particles are embedded. Nihei et al⁷⁴ evaluated the three-body wear resistance of resin composite materials with fillers which were modified with a hydrophobic silane coupling agent and showed that the composites developed higher wear resistance. Decreasing the size of the filler did not influence the wear resistance of the composite. Turssi et al⁷⁵ evaluated the wear resistance of the nano-structured composite and compared it with a microfill composite used as control. The results indicated no significant difference between the two composites. Fatigue has been shown as one of the major factors affecting three-body wear of resin composites.

It has been assumed that the matrix is influenced first in the composite system when subjected to wear.⁷⁶ Reich et al⁷⁷ studied the three-body wear resistance of the veneering composites. Furthermore, they studied the three-body wear of the pure matrices of the materials. The pure matrices of all composite materials showed similar wear results that did not differ significantly from each other. Three-body wear results of complex resin composites are highly influenced by their filler content, filler particle size distribution, kind of filler particles, shape, and their silanization to the matrix.

Attrition occurs in two-body wear mode, and results from the direct contact of opposing teeth where the load level increases higher than that causing abrasion.⁷³ Hu et al⁷⁸ explored the fundamental wear behavior of a dental composite with different filler loadings under two-body conditions. The results

showed that there was a little increase in the rate of wear with filler loadings below 60 wt%, but a sharp increase in the 80-87.5 wt% in filler loadings. Wide striations and bulk loss of material were apparent in the wear surfaces with higher filler loadings. Suese and Kawazoe⁷⁹ suggested that heat cure and mechanical polishing of the surface of the dental composite restoration improved its wear resistance. Wassell et al⁸⁰ evaluated the enamel and steatite abrasers used in the two-body wear test and also compared the wear rates in hybrid and microfilled composites. The study reported steatite as a suitable substitute for enamel and also showed higher wear rates for hybrid composite compared to microfill composites. Marquis et al⁸¹ evaluated two-body wear under different loads and concluded that wear increased with increasing loads.

The design of clinical studies to evaluate the wear of posterior composite restorations is complicated by the influence of numerous clinical variables. Factors such as the tooth restored, the size and the complexity of the restoration, and the presence and nature of occlusal contacts have been identified as affecting the observed wear, in addition to such obvious variables as the materials used and the age of the restoration.⁸² Because of these reasons, numerous in-vitro studies have been developed to predict the materials clinical performance.

Fracture Toughness

In posterior restorative materials both wear and incisal edge chipping can be viewed as fracture processes. Considerable attention has been directed

towards the application of fracture mechanics to these products. This approach enables a description of how an increasing stress applied to the specimen is intensified, around a pre-existing crack. Eventually, a critical value of the stress intensity factor (K_{IC}), also known as the fracture toughness, is attained whereupon the crack propagates catastrophically.¹⁵ The theory of plane strain fracture toughness testing is to determine critical values of strain intensity. Pre-cracked specimens of standard geometry are loaded until they break and then if the fractures are macroscopically brittle, the fracture strength can be used to calculate the toughness directly.⁸³

Uctasli et al⁸⁴ determined the effect of different curing systems used in the inlay/onlay techniques on the fracture toughness of indirect composites. He reported that the fracture toughness of the composites remained irrespective of mode of curing. Pilliar et al⁸⁵ examined the effects of aging composite specimens in air, water and ethanol. They used the mini short rod fracture toughness specimen design and suggested that while water aging does not cause significant changes in K_{IC} , aging in ethanol does produce significant decreases. Scherrer et al⁸⁶ studied the aged indirect composite materials and suggested that due to the low K_{IC} value of these materials, their use must be limited to low stress masticatory areas. Toparli et al⁸⁷ evaluated the fracture toughness of the resin composite and the adhesive interface and concluded that bonded interfaces tend to produce microscopic flaws which could act as critical stress risers promoting interfacial failures.

Radiopacity

As research focuses on increasing the mechanical properties of tooth-colored restorative materials to improve the clinical performance, radiopacity is a valuable property that allows the clinician to assess the adequacy of the restorations, distinguish secondary caries and evaluate marginal adaptation, voids and interfacial gaps. In addition, adequate radiopacity permits detection of inter-proximal contours, contacts and overhangs and serve an important role in detecting aspirated or dislocated restorations.⁸⁸ It thus serves as an important diagnostic tool when evaluating the long term success of the restorations.

Turgut et al⁸⁹ studied the radiopacity of direct esthetic restorative materials like composites, glass ionomers and compomers and compared them to enamel and dentin. The optical densities of each restorative material, along with one tooth section and an aluminum step wedge were measured from radiographic images using a transmission photodensitometer. All materials except for the microfilled resin composite investigated in this study had radiopacity values greater than dentin and possessed sufficient radiopacity to meet ISO 4049 standard. Tviet et al⁹⁰ conducted a radiographic diagnosis of caries and marginal defects in radiopaque composite fillings. They reported that radiopacity increases the detection rate of caries and defects adjacent to the restorations. It was also suggested that moderate radiopacity is preferable to high radiopacity material due to the masking effect of the latter. Bouschlicher et al⁹¹ suggested that composites must be more radiopaque than enamel to enable clinicians to distinguish the restorative material from tooth structure. Several studies quoted

the radiopacity values of enamel and dentin. Turgut et al⁸⁹ reported values of 2.02 mm Al/1 mm enamel and 1.13 mm Al/1 mm dentin while el-Mowafy et al⁹² reported 1.84 mm Al/1 mm enamel and 1.16 mm Al/1 mm dentin. It has also been reported that radiopacity values show variability among studies because of factors like speed of the x-ray film, exposure time, voltage used and the age of the developing and the fixing solutions.⁹²

MATERIALS AND METHODS

Four commercial composites were used in the study. Enamel shades and shade A₂ of dentin were used. The materials were cured in their respective curing units (Figure 1) according to the manufacturer's instructions. Radica was cured in the Enterra curing unit for an initial cure of 5 minutes and final cure of 3 minutes. Sculpture Plus were cured in Sculpture Plus curing unit for initial build up curing time of 5 minutes under vacuum in Nitrogen pressure and 3 minutes of light cure. This was followed by final build up curing time which was same as described for initial build up curing time. Belleglass-NG specimens were initially light cured in LED visible curing light for 60 seconds on each surface. This was followed by long cycle cure of 20 minutes in Belleglass-HP curing unit under a nitrogen atmosphere and temperature upto 140°C. Gradia Indirect specimens were initially cured in a Gradia Step Light for 10 seconds on each surface and finally cured in Gradia Labolite for 5 minutes. The composition and curing process is described in detail in Table I.

Laboratory tests were conducted to determine the following properties:

1. Water sorption and solubility.
2. Staining resistance.
3. Gloss and Surface Roughness.
4. Volume loss due to toothbrush abrasion.
5. Three-body Alabama wear resistance.
6. Sliding wear resistance determined by a pin-on-disc test.

7. Fracture Toughness.
8. Radiopacity.

Water Sorption and Water Solubility

Enamel and dentin shade A₂ were studied in this test. A Mylar strip was placed on a glass slide and a polytetrafluoroethylene mould (15±0.1 mm diameter and 1.0±0.1 mm thick) was placed on it. The mould was slightly overfilled with the material. A second piece of Mylar strip was then placed on the material in the mould and covered with a second glass slide thus displacing the excess material. Having displaced the excess material, the glass slides were removed and cured according to manufacturer's instructions in their respective curing units.

The specimens were then transferred to a desiccator, maintained at (37±1) °C. After 22 hours, the specimens were removed and stored in a desiccator maintained at (23±1) °C for two hours and then weighed to an accuracy of ±0.1mg. This cycle was repeated until a constant mass M₁ was obtained.

The area and volume (V) was calculated after measuring the mean diameter and thickness. The specimens were immersed in water at (37±1) °C for 7 days each in a separate bottle and suspended with an orthodontic wire such that they did not touch the walls of the bottles. After 7 days, the specimens were removed and weighed. The mass was recorded as M₂. They were then

reconditioned to a constant mass as described above. This mass was recorded as M_3 .

The water sorption was calculated using the following formula

$$W_{sp} = \frac{M_2 - M_3}{V}$$

Water solubility was calculated using the formula

$$W_s = \frac{M_1 - M_3}{V}$$

Staining Resistance

Enamel and dentin shade A₂ were used in this test. Six Specimens for each group with dimensions of 12.5 mm diameter and 2 mm thickness were cured as previously described. After dark storage in distilled water for 24 hours at room temperature, baseline color of all specimens was measured. CIE- $L^*a^*b^*$ system (Commission Internationale de l'Eclairage) measurements were obtained with a Minolta Spectrometer CM 2500d (Minolta, Japan) and D65 light against a white background. All measurements were repeated thrice and the medians for the $L^*a^*b^*$ values were calculated.

After the baseline measurements, five specimens from each group were stored in a staining solution and one specimen was stored in distilled water. The staining solution was an instant coffee solution prepared by using 5 ounces of coffee in 750 ml of water which had been brought to boil.

After 72 hours in the solution, color measurements were made using the reflectance spectrometer as described earlier. Prior to the color measurement,

the specimens stored in the staining solutions were rinsed thoroughly under tap water and subjected to 10 strokes of brushing with a soft grade tooth brush. They were gently dried using Kimwipes (delicate task wipers). After measuring, specimens were once again immersed into the staining solution. The process for measuring was repeated every 72 hours for a total of three weeks.

Color change after storing in the solutions was calculated for each specimen at every 72 hours using the color difference formula:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Where, ΔE^* represents the color difference and ΔL^* , Δa^* , Δb^* represents the changes in lightness, red-green coordinate, and yellow-blue coordinate, respectively, after immersion in the solutions. These values were noted in both specular component included (SCI) and specular component excluded (SCE).

Gloss and Surface Roughness

'Enamel' shade composites were chosen for this study. A custom-made, stainless steel mould (Figure 9) was used to reproducibly fabricate 10 specimens of each composite material (n=10). The dimensions of each specimen used in the study were 2 X 5 X 20 mm.

Composite specimens were used according to manufacturer's instructions. They were placed in the mould in 1 mm increments. Each increment was then cured in their respective curing units as previously described. A Mylar strip was

placed over the final increment and pressed with a cover slide to ensure that the material was flush with the surface of the mould. After polymerization, the specimens were carefully removed from the mould and stored in distilled water for 24 hours. They were then finished with SiC discs on a polishing wheel in the order of 400 grit, 600 grit, 800 grit, 1200 grit and further polished with diamond polishing pastes of particle size 1 micron and 0.25 microns. Care was taken to observe the specimen surface under light microscope at 5X magnification during every stage of finishing and polishing to make sure that the scratch lines were decreasing in size.

After the specimens were polished, gloss and surface roughness measurements at baseline were noted. To measure the gloss value a special Jig was created (Figure 10) made with a black cardboard. The jig was formed so that the specimen could be placed in the same position repeatedly. The glossmeter (HORRIBA JAPAN) was then placed and the gloss value is measured at a 60° angle from normal to the surface. Surface roughness was measured along the length of the specimen using a Taylor Hobson Surftronic Profilometer (Figure 11). The length of the stylus movement was 4 mm. Three readings were taken in the different areas but in central region of the specimen.

The specimens were placed in a Pepsodent Toothbrush Abrasion Machine. They were fixed in position with a double sided adhesive tape. The excess of the tape around the specimen was cut with a scalpel. Toothbrush heads with medium grade bristles were used for the test. Slurry made with Colgate Total Tooth Paste and deionized water in a ratio of 1:1 was prepared

and poured equally in each of the six chambers. The paste had the abrasivity of 70 RDA (Relative Dentin Abrasivity). The toothbrush head brushed the specimens at a 170 rpm. After 5000 cycles of toothbrushing the specimens were removed, cleaned with distilled water in an ultrasonic bath and dried with canned air and Kimwipes (delicate task wipers). Gloss readings and surface roughness were measured and then each specimen was placed again in the abrasion machine as described above. The procedure was repeated again after 10,000 and 20,000 cycles.

Toothbrush Abrasion Wear

'Enamel' shade composites were chosen for this study. Six specimens were fabricated in the same manner as described in the gloss test. However for this test the Mylar covered surfaces were not touched and the side surfaces were finished up to 1200 grit with SiC discs. After finishing, the specimens were weighed and measured for length, width and thickness to calculate the volume.

To measure the weight of the specimens they were kept in desiccators, maintained at $(23 \pm 1) ^\circ\text{C}$ and then weighed to an accuracy of ± 0.1 mg. The measurements were repeated until a constant mass M_1 was obtained i.e. each specimen did not change more than 0.1 mg in any 24 hour period. Volume (V_1) was calculated and used to determine the density (ρ) of each specimen, which was expressed in the units of mg/mm^3 .

$$\rho = \frac{M_1}{V_1}$$

The specimens were then brushed in a mechanical toothbrushing machine (Pepsodent Co., Chicago IL) for two hours (20,760 strokes) in a direction perpendicular to the length of the specimen. Aqueous slurry of 1:1 proportion by weight of Colgate Total Tooth Paste and deionized water was used during the brushing. After toothbrushing the specimens were removed, cleaned with distilled water in an ultrasonic bath and dried with canned air and Kimwipes (delicate task wipers).

Specimens were kept in a desiccator and weighed every 24 hours to obtain a constant weight M_2 . The volume of the material lost to toothbrushing was calculated in the units of mm^3 . Materials demonstrating the lowest volume loss were considered as the most resistant against three-body toothbrush abrasion wear.

$$V_2 = M_2 \times \rho$$

$$\text{Volume loss } (\Delta V) = V_1 - V_2$$

Three-body Alabama Wear Resistance

The three-body wear test was done using the Alabama wear test method. The machine's purpose was to simulate in-vitro the wear encountered clinically of various dental materials, but at accelerated rates. The machine was powered by a variable-speed motor providing speed up to 120 contacts per minute.

The sliders were made of Poly-acetyl resin and finished using 240 and 600 grit papers. A Nikon Digimicro dial gauge was used to measure the slider height. The sliders were then screwed into the slider holder and the position

zeroed. The material was placed in increments and cured according to manufacturer's instruction. The last layer was slightly overfilled. The surface was made flat by a piece of matrix strip that was placed on top and covered with a microscope slide. The glass slide was then pressed down to make a flat surface prior to curing. The microscope slide and the matrix strip were then removed. Specimens were then stored in distilled water at 23°C for 24 hours. Four specimens (9.5 mm diameter, 2 mm thickness).each group were prepared at a time. Specimens were then finished with 240 and 600 grit paper using a custom jig.

After the specimens and sliders were finished and measured, the pistons were calibrated in the Universal testing machine (Model MTS Sintech ReNew 1123, Eden Prairie, MN) before running the test. The sliders were then screwed into the end of the piston. The specimen holders were placed into an acrylic specimen holder chamber. A brass collar was put around each specimen. Slurry was prepared by weighing 15.0 grams of HG-5 polymer in a 5 oz plastic cup and adding 9 ml of distilled water and stirring well. It was carefully poured into the collars almost to the top but not overfilled. The chamber was filled close to the top of the collar with distilled water. The load on the piston was adjusted to about 17 lbs of pressure and the speed was set to 75 rpms.

The specimens were measured before testing with a contact profilometer to make sure the surface was flat and level. If after using the profilometer, the results indicated the surface was rounded, then the specimens were refinished with the 600 grit paper and the profile run again. The specimen holders were

numbered and placed in the holder block with the number facing the screw. On the specimen a dot was put close to the top edge at the number and again at 45°, 90°, and 135° positions. After testing, a small clear ruler was taken and a straight pencil line was drawn across the surface of the specimen and holder starting at the number. The intersection of the lines was considered the center of the worn area. The stylus was laid down gently and drawn across the wear track to obtain measurements. The data was tabulated using Surftronic software and necessary graphs taken to calculate wear depth and volume loss.

Sliding Wear Resistance Determined by a Pin-on-Disc Test

Enamel shade composites were used in this test. Six discs specimens approximately 12 mm in diameter and 3 mm thick were fabricated from each material in Teflon molds. They were removed from the mold and stored at 37°C in distilled water for 24 hours before testing. Prior to the wear test, the specimen were mounted in a brass cup using a filled auto polymerizing acrylic resin. They were then rinsed with distilled water in an ultrasonic cleaning machine for 10 minutes.

Sintered calcium hydroxyapatite (5 mm diameter and 10 mm long) cylinders were mounted in small brass slider cups that fit into the wear testing machine. A putty jig system was used to allow the hydroxyapatite slider to be mounted approximately in the center of the cup. They were fixed in the cup using a filled autopolymerizing resin. They were then cut in a lathe to form cylindrical sliders of 2 mm diameter and 1.5 mm height and finished using a 600 grit silicon carbide

paper. They were rinsed in distilled water in an ultrasonic device. The length of each slider was measured and recorded prior to each wear run.

A two-body rotating pin-on-disk wear testing machine was used. It contains four wear stations, each of which consists of a shaft within a set of linear bearings to minimize lateral movements. The specimens were attached to the end of the shafts by a screw on the base of the brass mounting screws. The brass cups containing the specimens were screwed to the upper member of the stations. The sliders were screwed to the lower platforms. The sliders mounted in the smaller brass cups were screwed to platforms rotating at constant speed with a radius of movement of approximately 3 mm.

The wear test was run for 25,000 cycles at 120 revolutions per minute. The wear field was washed continuously with distilled water for the entire period of the test. After the cycles were complete, the sliders were removed and measured under a digital micrometer. The specimens were removed and cleaned with distilled water in an ultrasonic bath. They were then scanned in the Contact Profilometer and area was recorded at six different positions of the wear tract using Taylorlite software and measured as described in three-body wear. Integration was applied to calculate the volume wear loss using the average radius and area from the software.⁹³

Plane Strain Fracture Toughness

Enamel and dentin shade A₂ were used in this test. Six specimens for each group were fabricated according to ASTM standard E39983 for a single-edge notch bar-shaped specimen.

A single-edge notched beam test was used to determine the fracture toughness (K_{IC}). The American Society of Testing Materials guidelines for the single-edge notched specimens (Standard E-39983) was used for the test specimen configuration.⁹⁴ A custom-made, stainless steel mould with a sharp blade in the center was used to reproducibly fabricate 6 specimens of enamel shade and 6 specimens of dentin shade (A₂) for each composite material (n=6). The dimensions of each specimen were 2 X 5 X 25 mm, with a 2 mm long notch on one edge.

Composites were used according to manufacturer's instructions. They were then placed in the mold in 1 mm increments. Each increment was cured in their respective curing unit. A Mylar strip was placed over the final increment and pressed with a cover slide to ensure that the material was flush with the surface of the mould. After polymerization, the specimens were carefully removed from the mold. Any flash present on the border was removed by finishing the specimen with 400 grit and 600 grit sandpaper. Specimens with noticeable notched defects around the notched area were discarded. They were then stored in a 37°C humidior for 24 hours before testing.

A universal testing machine (Model MTS Sintech ReNew 1123, Eden Prairie, MN) was used and a central load was applied to each beam specimen in

a 3-point bending mode at a crosshead speed of 0.1 mm/min until the specimen fractured and the load recorded. The crack length was measured after the fracture with a measuring microscope (Nikon Measurescope UM2)

The fracture toughness was calculated using the equation

$$K_{IC} = 0.0316(PqS/BW^{1.5}) f(a/w)$$

Where

$$F(a/w) = 3(a/w)^{1.5} [1.99 - (a/w)(1-a/w) \times (2.95 - 3.93(a/w) + 2.7a^2/w^2)] / 2(1+2a/w)(1-a/w)^{1.5}$$

Pq = maximum fracture load (N)

B = specimen thickness (mm)

S = supporting span (mm)

W = specimen width (mm)

a = crack length (mm)

Radiopacity

Enamel and dentin shade A₂ were used in the study. Six disk specimens of each group were prepared as described in the water sorption test with a thickness of (1 ± 0.1) mm and diameter of 16 ± 0.1 mm, in accordance with ISO 4049 and Aoyagi et al.⁹⁵ The specimens were finished through 600 grit sandpaper to create a flat surface. Specimens were measured after finishing verifying the critical tolerance of 1.0 ± 0.01 mm. One specimen of each material, dentin disc of 1 mm and a standard propriety aluminum step wedge were

positioned side by side on an occlusal radiographic D speed film. The wedges maximum thickness was 13.5 mm and step size was 1 mm. The films were exposed for 0.37 seconds with a dental radiography unit at 70 KV and 10 mA; the object to film distance was 40 cm. The films were processed in a standard automatic processor. The optical densities of these images were measured with a transmission densitometer.

Hypothesis

The null hypothesis of the study was that, for each of the investigated properties, the four composite materials are not significantly different from each other.

Statistical analysis

For each set of experiments, means and standard deviations were calculated for the enamel and dentin shades of all the four composite groups. For Gloss and surface roughness and wear properties like toothbrush abrasion, three-body and two-body wear only enamel shades were used. All statistical testing was performed with a significance level of 0.05. Water Sorption and Water Solubility, Fracture toughness and Radiopacity test results were analyzed separately for enamel and Dentin shades by one-way analysis of variance (ANOVA) and pair-wise comparison procedures were performed using Tukeys test. The results of two and three body wear tests were compared between materials by using a similar ANOVA model. Gloss, surface roughness and

staining were analyzed using a repeated measures analysis of variance model.

Staining was analyzed separately for enamel and dentin shades.

RESULTS

Water Sorption and Water Solubility

Results from the water sorption and water solubility tests are summarized in the Tables II, III and Figures 2, 3. Water sorption and water solubility values differed significantly among all the test groups. When enamel groups were compared Radica showed the lowest water sorption and solubility. Belleglass-NG demonstrated the highest water sorption followed by Gradia Indirect. No significant statistical differences were found between Sculpture plus and Radica. However, sculpture showed highest water solubility among the four tested groups. Negative water solubility was observed for Belleglass-NG and Gradia Indirect. When dentin groups were compared, Belleglass- NG and Gradia Indirect showed negative solubility. Radica and Sculpture did not differ statistically in water sorption and water solubility values.

Staining Resistance

Results of this test are presented in Tables IV to VII and Figures 4 to 8. Statistically significant changes in ΔE values over a 21 day period were observed in all the laboratory composites for both enamel and dentin shades. The dentin shade of all composites showed more vulnerability to staining than the enamel shade. Belleglass-NG demonstrated the least change in ΔE while Sculpture demonstrated large changes in ΔE . b^* co-ordinate showed maximum changes in its value after each interval for the composites.

Gloss and Surface Roughness

The gloss and the R_a values are presented in Tables VIII and IX. Figure 13 illustrates the decrease in gloss and Figure 14 illustrates the R_a values with increasing number of cycles. Figures 15 to 18 shows surface topography of the four composites observed under a light microscope at 0, 5000, 10,000 and 20,000 cycles. The tracks produced by the toothbrush bristles and the exposure of some fillers were observed by 10,000 cycles for all materials. The results showed that initially all the materials demonstrated high gloss. However, all the test materials showed decrease in gloss and increase in roughness with increasing number of toothbrushing cycles. Compared to the other materials Belleglass-NG demonstrated some ability to retain gloss through the, first 5000 cycles. By the end of 10,000 cycles all the groups showed a decrease of 75% of the initial gloss. At the end of 20,000 cycles Belleglass-NG retained gloss higher than other materials. Surface Roughness was highest observed in Radica and lowest in Belleglass at the end of 20,000 cycles.

Volume Loss Due to Toothbrush Abrasion

Results from this test are summarized in Table X and Figure 20. Statistically, Radica and Belleglass-NG showed the least volume loss while Sculpture Plus showed the highest wear. No significant differences were found in the volume loss of Radica and Belleglass-NG. Another observation made while evaluating the weight loss was the density of the material.

Three-body Alabama Wear Resistance

Results from this test are summarized in Table XI and Figure 21. The results are similar to the volume loss due to the toothbrush abrasion. Radica and Belleglass-NG showed lower volume loss than Gradia Indirect and Sculpture. Statistically no significant difference was observed between Radica and Belleglass-NG.

Sliding Wear Resistance Determined by a Pin-On-Disc Test

Results from this test are summarized in Table XII and Figure 22. The results correlate with the three-body wear results. Sculpture Plus showed the highest volume loss compared to other test groups. Statistically no significant difference was observed between Radica, Belleglass-NG and Gradia Indirect.

Plane strain Fracture Toughness

Results from this test are summarized in Table XIII and Figure 24. Fracture toughness values (K_{IC}) values differ significantly among all the test groups. When enamel groups were compared, the K_{IC} value of Radica was higher than all other test groups. Gradia Indirect was higher than Belleglass-NG and Sculpture Plus. Statistically no significant difference was observed between Belleglass-NG and Sculpture Plus. When Dentin groups were compared, Radica dentin showed highest K_{IC} value compared to the other groups while Sculpture Plus showed the lowest K_{IC} values. Statistically no significant differences were observed between Belleglass-NG and Gradia Indirect.

Radiopacity

Lesser optical density indicated higher radiopacity. Results from this test are summarized in Table XIV and Figure 25. When enamel groups were compared Sculpture Plus showed the lowest optical density i.e. it was most radiopaque among the test groups. Belleglass-NG was least radiopaque and statistically not different from Gradia Indirect. When dentin groups were compared, Sculpture Plus was still the most radiopaque among all test groups while Gradia Indirect was least radiopaque.

TABLES

Table I

Commercial Composites

Material (Shade: enamel)	Matrix	Filler	Curing method
Radica (Dentsply)	Urethane dimethacrylate (UDMA)	Barium fluoroaluminoborosilicate glass (silanated), Amorphous silica	Enterra Curing Light:(heat (80°C approx.+ Halogen light)5 minutes of initial cure +2 minute of Pontic cure)
Belleglass-NG (Kerr corp)	Urethane dimethacrylate (UDMA)	Prepolymerized filler Amorphous Silica	Belleglass Curing Unit (heat and pressure) Initial Light cure with LED visible light cure for 20 seconds followed by 20 minute cure cycle under a nitrogen pressure (60psi).
**Gradia Indirect (GC, Tokyo, Japan)	Urethane dimethacrylate (UDMA)	Silica Powder, Silica Glass Powder and Prepolymerized filler	Gradia Curing unit and step curing light (halogen light). 20 seconds of step curing followed by 5 minute of curing in curing unit
*Sculpture Plus (Pentron Lab)	polycarbonate dimethacrylate, Ethyoxylated Bis- GMA (PCDMA)	Microfiller Barium borosilicate Amorphous silica	Sculpture curing unit: (heat, pressure light) Build up cycle and final cycle of 8 min(5 minute of Nitrogen pressure(80psi and 3minutes of halogen light)

*Sculpture = Micheal Mandikos, etal A comparison of wear resistance and hardness of indirect composite resins J Prosthet Dent 2001;85:386-95

**Gradia Indirect = Masaomi Ikeda et al. Shear Bond strength of Indirect Resin Composites to Hybrid ceramics; Dent Mater J, 2005 Jun;24(2): 238-43

Table II

Water Sorption and Solubility of Enamel Groups

Material Group (ENAMEL)	Water Sorption (Wsp) Mean (std dev) UNIT: $\mu\text{g}/\text{mm}^3$	Water Solubility (Ws) Mean (std dev) UNIT: $\mu\text{g}/\text{mm}^3$
RADICA	13.29 (0.84) ^a	0.5 (0.3) ^a
SCULPTURE	16.6 (0.60) ^c	2.47 (0.58) ^b
BELLEGLASS-NG	25.9 (1.31) ^b	-2.23 (0.16) ^c
GRADIA INDIRECT	23.17 (1.06) ^b	-1.8 (0.3) ^c

Mean values (s.d) in columns are not statistically different $p \leq .05$

Table III

Water Sorption and Solubility of Dentin Groups

Material Group (DENTIN)	Water Sorption (Wsp) Mean (std dev) UNIT: $\mu\text{g}/\text{mm}^3$	Water Solubility (Ws) Mean (std dev) UNIT: $\mu\text{g}/\text{mm}^3$
RADICA	13.83 (1.01) ^a	1.09 (0.57) ^a
SCULPTURE	16.53 (0.63) ^b	0.83 (0.61) ^a
BELLEGLASS-NG	13.13 (0.45) ^a	-0.68 (0.23) ^b
GRADIA INDIRECT	24.06 (0.58) ^c	-0.79 (0.69) ^b

Mean values (s.d) in columns are not statistically different $p \leq .05$

Table IV

Staining Resistance of Enamel Groups (SCE component)

Measurement Interval SCE (ΔE)	Radica Mean ΔE (std dev)	Sculpture plus Mean ΔE (std dev)	Belleglass-NG Mean ΔE (std dev)	Gradia Indirect Mean ΔE (std dev)
baseline	0	0	0	0
Day 3	5.26 (1.17) ^b	17.62 (3.52) ^a	2.59 (1.56) ^c	2.49 (0.94) ^c
Day 6	8.12 (2.23) ^b	23.06 (4.21) ^a	3.49 (2.56) ^c	4.45 (1.23) ^c
Day 9	7.61 (1.58) ^b	26.49 (4.85) ^a	2.97 (1.80) ^c	5.98 (1.48) ^d
Day 12	11.15 (1.77) ^b	28.74 (4.92) ^a	2.15 (1.06) ^c	7.37 (1.62) ^d
Day 15	10.78 (1.96) ^b	30.19 (4.82) ^a	3.28 (1.30) ^c	8.27 (0.28) ^d
Day 18	11.08 (1.75) ^b	31.9 (5.37) ^a	4.26 (1.82) ^c	8.03 (1.12) ^d
Day 21	11.45 (2.01) ^b	32.18 (5.06) ^a	4.69 (2.09) ^c	8.42 (1.02) ^d

Mean Values (s.d.) in rows with same letters are not statistically different $p \leq 0.05$.

Table V

Staining Resistance of Enamel Groups (SCI component)

Measurement Interval SCI (ΔE)	Radica Mean ΔE (std dev)	Sculpture plus Mean ΔE (std dev)	Belleglass-NG Mean ΔE (std dev)	Gradia Indirect Mean ΔE (std dev)
Baseline	0	0	0	0
Day 3	4.48 (0.98) ^b	15.83 (2.56) ^a	2.26 (1.29) ^c	2.24(0.63) ^c
Day 6	7.17 (2.18) ^b	19.3 (3.24) ^a	3.09 (1.27) ^c	3.61 (1.15) ^c
Day 9	7.24 (1.49) ^b	22.7 (3.46) ^a	2.74 (1.59) ^c	5.35 (1.36) ^b
Day 12	10.26 (1.99) ^b	23.7 (3.21) ^a	1.86 (0.79) ^c	6.59 (1.17) ^d
Day 15	9.87 (1.83) ^b	24.64 (3.3) ^a	2.93 (1.07) ^c	7.27 (0.43) ^d
Day 18	10.27 (1.67) ^b	25.13 (3.01) ^a	3.79 (1.14) ^c	7.25 (1.13) ^d
Day 21	10.55 (1.80) ^b	25.6 (3.14) ^a	4.52 (1.79) ^c	7.72 (1.35) ^d

Mean Values (s.d.) in rows with same letters are not statistically different $p \leq 0.05$.

Table VI

Staining Resistance of Dentin Groups (SCE component)

Measurement Interval SCE (ΔE)	Radica Mean ΔE (std dev)	Sculpture plus Mean ΔE (std dev)	Belleglass-NG Mean ΔE (std dev)	Gradia Indirect Mean ΔE (std dev)
Baseline	0	0	0	0
Day 3	3.93 (1.9) ^b	14.52 (10.3) ^a	2.77 (0.98) ^b	3.14 (0.57) ^b
Day 6	6.69 (2.32) ^b	16.69 (10.51) ^a	3.13 (1.13) ^c	4.4 (0.80) ^c
Day 9	6.00(2.61) ^b	16.92 (9.86) ^a	3.35 (1.59) ^c	5.45 (0.71) ^b
Day 12	6.91 (2.22) ^b	21.12 (11.34) ^a	3.84 (1.28) ^c	6.02 (0.84) ^b
Day 15	7.63 (2.4) ^b	22.4 (10.75) ^a	4.28 (1.19) ^c	6.61 (0.84) ^b
Day 18	8.03 (2.58) ^b	22.83 (11.39) ^a	4.60 (1.33) ^c	7.23 (0.77) ^b
Day 21	8.35 (2.67) ^b	22.9 (10.95) ^a	4.78 (1.16) ^c	7.13 (0.79) ^b

Mean values (s.d) in rows are not statistically different $p \leq .05$

Table VII

Staining Resistance of Dentin Groups (SCI component)

Measurement Interval SCI (ΔE)	Radica Mean ΔE (std dev)	Sculpture plus Mean ΔE (std dev)	Belleglass-NG Mean ΔE (std dev)	Gradia Indirect Mean ΔE (std dev)
Baseline	0	0	0	0
Day 3	3.25 (1.67) ^b	12.13 (9.13) ^a	2.07 (0.58) ^b	2.67 (0.52) ^b
Day 6	5.19 (1.9) ^c	13.92 (9.22) ^a	2.66 (0.97) ^b	3.81 (0.69) ^{b, c}
Day 9	5.01 (1.81) ^c	14.4 (8.05) ^a	2.93 (0.96) ^b	4.91 (0.64) ^{b, c}
Day 12	5.81 (1.99) ^c	17.15 (9.53) ^a	3.32 (1.22) ^b	6.16 (1.78) ^c
Day 15	6.53 (2.02) ^c	18.06 (8.71) ^a	3.89 (0.95) ^b	6.18 (0.74) ^c
Day 18	7.04 (2.23) ^c	18.61 (9.19) ^a	3.81 (0.85) ^b	6.69 (0.83) ^c
Day 21	7.21 (2.24) ^c	18.4 (8.75) ^a	4.1 (0.91) ^b	6.80 (0.59) ^c

Mean Values (s.d.) in rows with same letters are not statistically different $p \leq 0.05$.

Table VIII

Gloss and Surface Roughness

CYCLES	GLOSS				R _a			
	Radica	Sculpture	Belleglass	Gradia	Radica	Sculpture	Belleglass	Gradia
0	87.3 ^a (1.5)	89.5 ^a (1.5)	81.5 ^b (1.9)	78.3 ^b (2.21)	.023 ^b (.008)	.012 ^a (.003)	.011 ^a (.007)	.025 ^b (.003)
5000	35.8 ^b (9.9)	12.7 ^a (3.7)	45.3 ^c (9.11)	18.4 ^a (4.4)	.226 ^a (.06)	.223 ^a (0.05)	.134 ^b (.03)	.283 ^c (.05)
10,000	15.5 ^a (6.0)	12.6 ^a (2.9)	26.2 ^b (10.2)	13.7 ^a (2.3)	.3 ^a (.06)	.252 ^b (0.07)	.204 ^b (.04)	.306 ^a (0.06)
20,000	9.5 ^a (2.4)	9 ^a (1.9)	19.8 ^b (7.9)	9.8 ^a (2.9)	.447 ^a (.07)	.355 ^{b,c} (0.09)	.3 ^c (.07)	.374 ^b (.05)

Mean Values (s.d.) in rows with same letters are not statistically different $p \leq .05$.

Table IX

Gloss and Surface Roughness

CYCLE	RADICA		SCULPTURE		BELLEGLASS		GRADIA	
	Gloss	Ra	Gloss	Ra	Gloss	Ra	Gloss	Ra
0	87.3 ^a (1.5)	.023 ^a (.008)	89.5 ^a (1.5)	.012 ^a (.003)	81.5 ^a (1.9)	.011 ^a (.007)	78.3 ^a (2.21)	.025 ^a (.003)
5000	35.8 ^b (9.9)	.226 ^b (.06)	12.7 ^b (3.7)	.223 ^b (0.05)	45.3 ^b (9.11)	.134 ^b (.03)	18.4 ^b (4.4)	.283 ^b (.05)
10000	15.5 ^c (6.0)	.3 ^c (.06)	12.6 ^b (2.9)	.252 ^b (0.07)	26.2 ^{b,c} (10.2)	.204 ^c (.04)	13.7 ^b (2.3)	.306 ^b (0.06)
20000	9.5 ^c (2.4)	.447 ^d (.07)	9 ^b (1.9)	.355 ^c (0.09)	19.8 ^c (7.9)	.3 ^d (.07)	9.8 ^b (2.9)	.374 ^c (.05)

Mean Values (s.d.) in columns with same letters are not statistically different $p \leq .05$

Table X

Volume Loss Due to Toothbrush Abrasion

Material (enamel)	Mean Volume loss and SD (mm ³)
Radica	1.75 (0.59) ^{a, b}
Sculpture Plus	4.58 (1.18) ^c
Belleglass-NG	1.18 (0.21) ^a
Gradia Indirect	2.42 (0.82) ^b

Mean Values (s.d.) in columns with same letters are not statistically different $p \leq .05$

Table XI

Three-body Alabama Wear

Material (enamel)	3-body Alabama wear Volume Loss and SD (mm ³)
Radica	0.311 (0.13) ^b
Sculpture Plus	0.562 (0.11) ^a
Belleglass-NG	0.12 (0.04) ^c
Gradia Indirect	0.658 (0.23) ^a

Mean Values (s.d.) in columns with same letters are not statistically different $p \leq .05$

Table XII

Two-body Wear

Material (enamel)	Volume Loss and SD (mm ³)
Radica	0.164 (0.08) ^b
Sculpture Plus	0.718 (0.34) ^a
Belleglass-NG	0.124 (0.03) ^b
Gradia Indirect	0.190 (0.07) ^b

Mean Values (s.d.) in columns with same letters are not statistically different $p \leq .05$

Table XIII

Fracture Toughness

SPECIMEN	RADICA		SCULPTURE		BELLEGLASS		GRADIA	
	ENAMEL	DENTIN	ENAMEL	DENTIN	ENAMEL	DENTIN	ENAMEL	DENTIN
1	2.229	2.07	0.85	0.906	0.795	1.349	1.48	1.317
2	2.417	1.685	0.95	0.776	0.923	1.502	1.222	1.229
3	2.017	1.657	0.914	0.728	0.878	1.422	1.247	1.288
4	2.465	2.17	0.773	0.636	0.891	1.378	1.143	1.304
5	2.445	1.76	0.996	0.741	0.951	1.295	1.189	1.106
6	2.308	1.953	0.845	0.697	0.937	1.34	1.136	1.204
MEAN	2.313	1.883	0.888	0.747	0.896	1.381	1.236	1.241
STDEV	0.171	0.213	0.081	0.091	0.056	0.073	0.127	0.08
MIN	2.017	1.657	0.773	0.636	0.795	1.295	1.136	1.106
MAX	2.465	2.17	0.996	0.906	0.951	1.502	1.48	1.317

Table XIV
Optical Density

SPECIMEN	RADICA		GRADIA		BELLEGLASS-NG		SCULPTURE PLUS	
	ENAMEL	DENTIN	ENAMEL	DENTIN	ENAMEL	DENTIN	ENAMEL	DENTIN
1	1.15	1.11	1.43	1.47	1.43	1.22	1.12	1.09
2	1.11	1.18	1.39	1.49	1.44	1.2	1.14	1.05
3	1.21	1.14	1.45	1.45	1.47	1.18	1.12	1.13
4	1.26	1.08	1.39	1.5	1.45	1.19	1.11	1.12
5	1.16	1.12	1.41	1.43	1.44	1.24	1.1	1.09
6	1.19	1.09	1.3	1.44	1.42	1.23	1.03	1.05
MEAN	1.18	1.12	1.395	1.46333	1.44166	1.21	1.10333	1.08833
STD DEV	0.052154	0.036332	0.052058	0.028048	0.017224	0.02366	0.03829	0.03371

FIGURES

Figure 1

Curing Units



Gradia Indirect



Belleglass-NG



Sculpture Plus



Radica

Figure 2

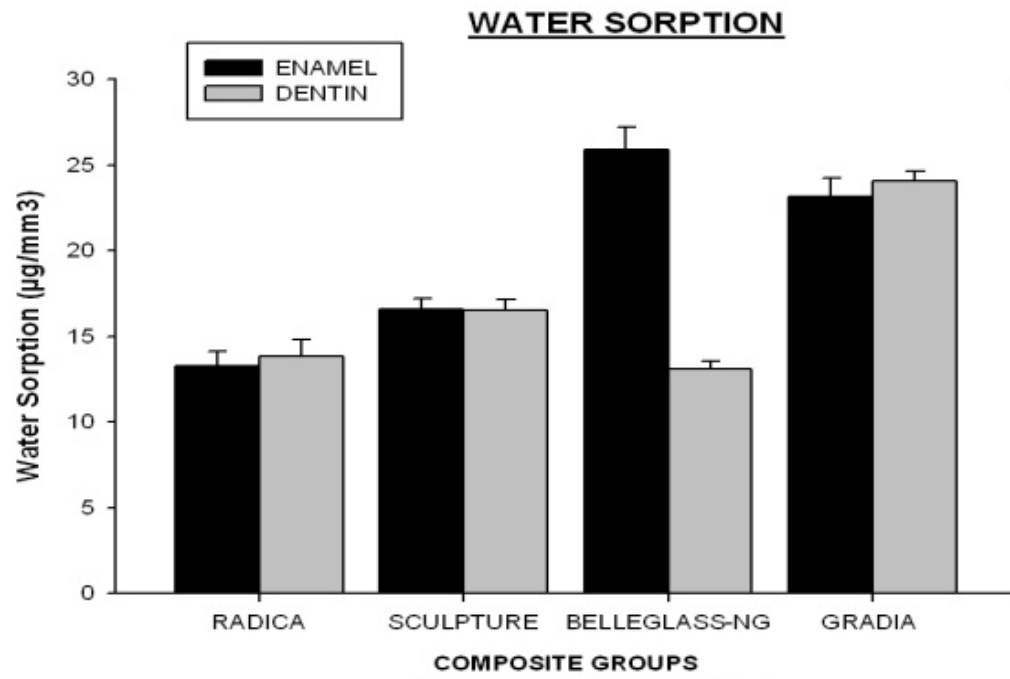
Water Sorption Results

Figure 3

Water Solubility Results

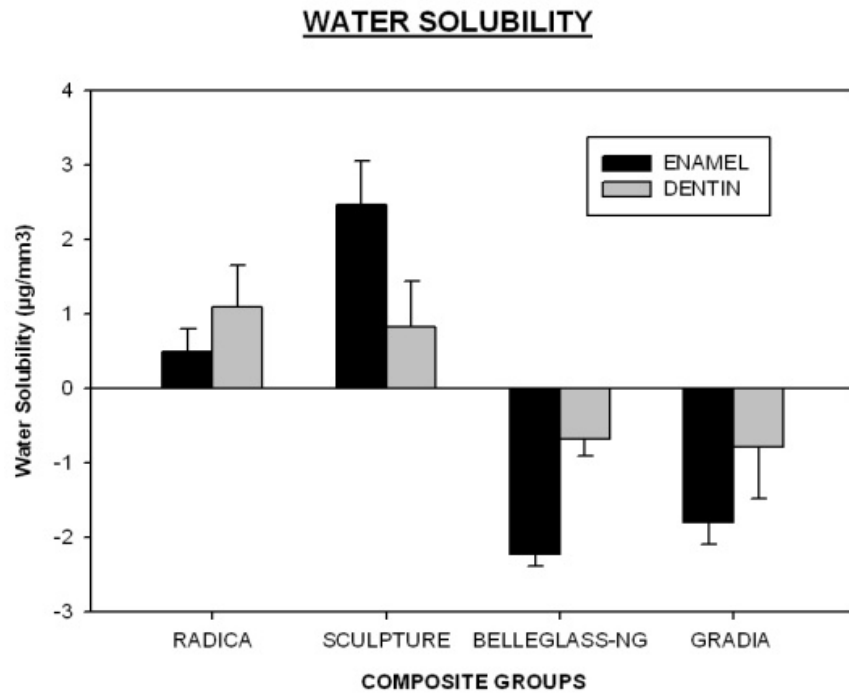


Figure 4

Apparatus for Staining Test

Left to Right: a) Spectrophotometer b) calibration plate c) Jig with a white background

Figure 5

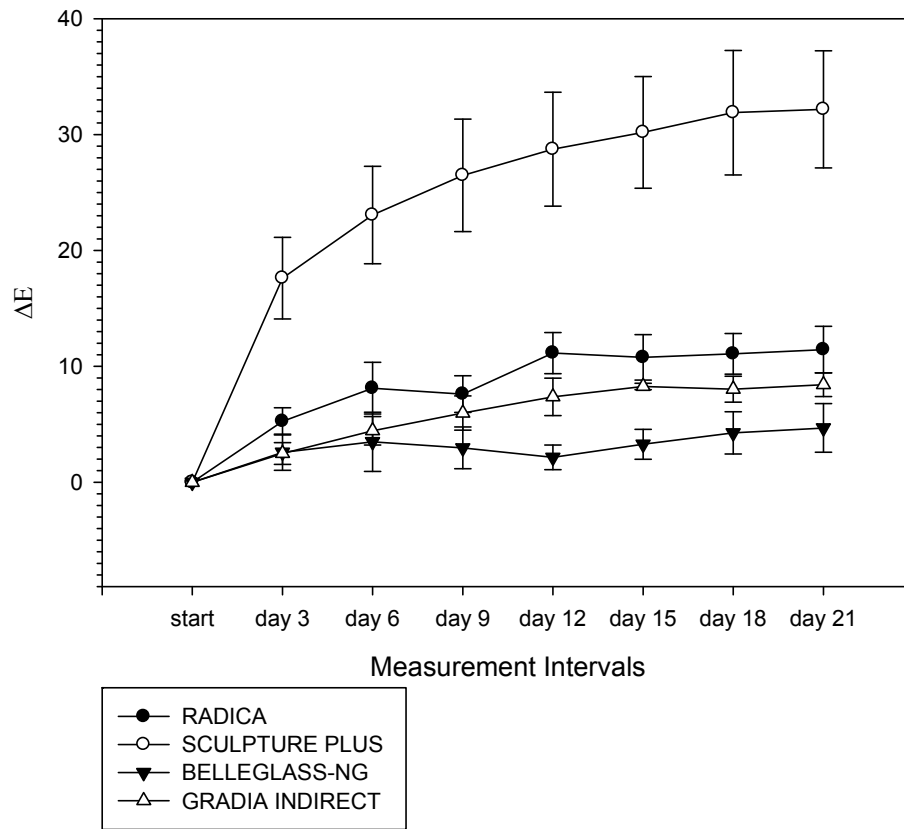
Staining Resistance (Enamel) – SCE component

Figure 6

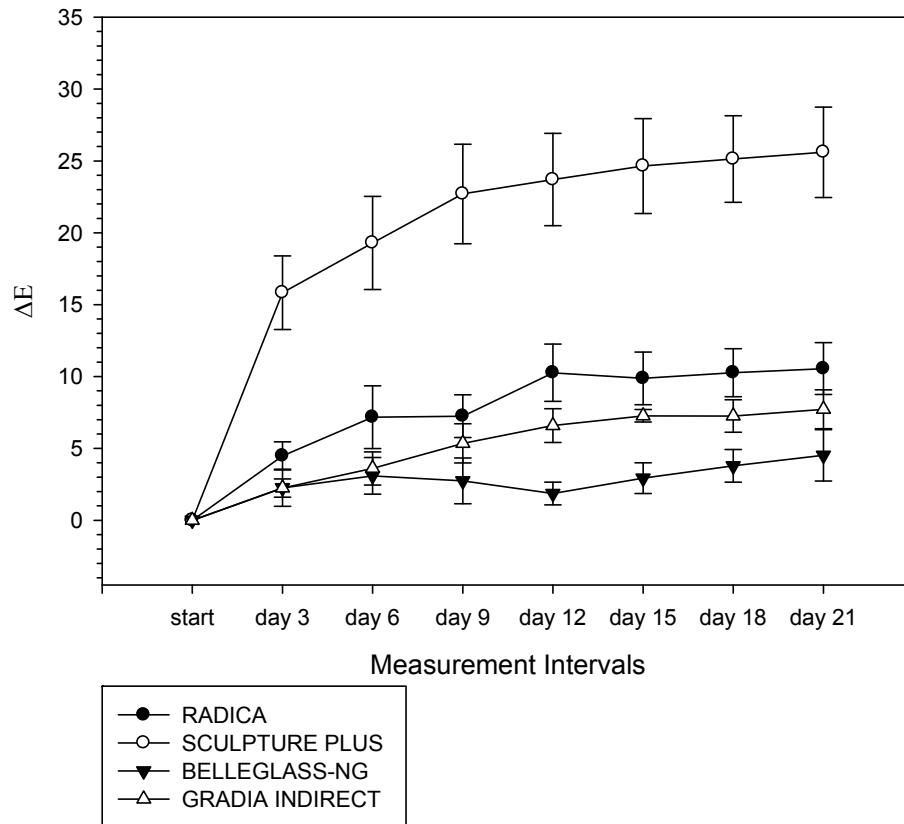
Staining Resistance (Enamel) – SCI component

Figure 7

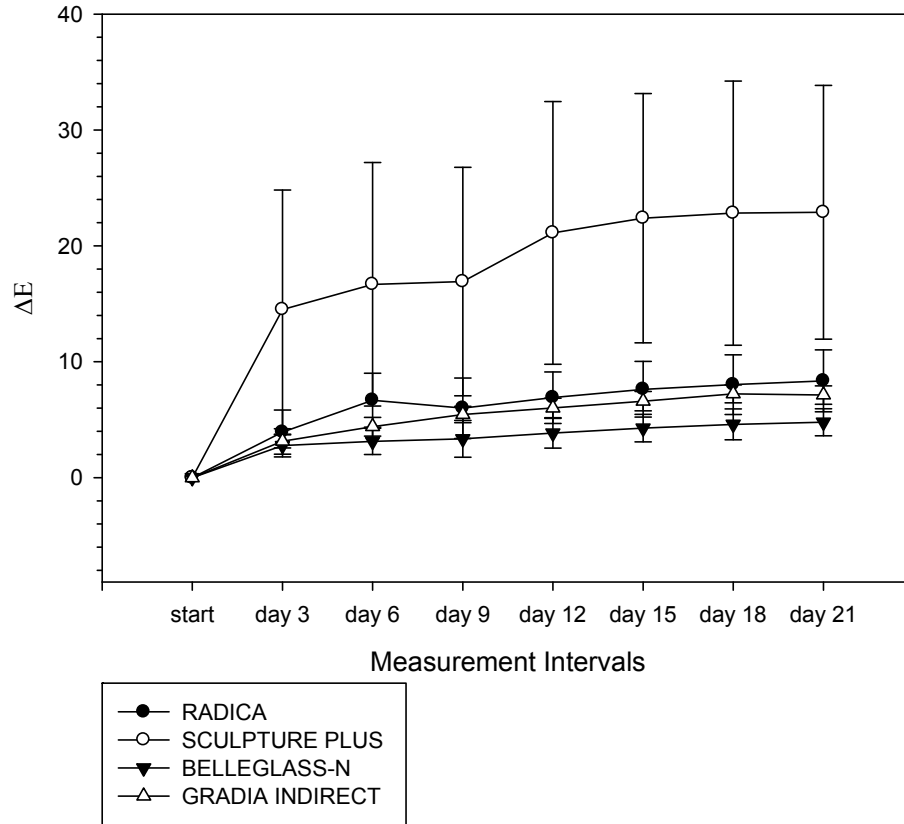
Staining Resistance (Dentin) – SCE component

Figure 8

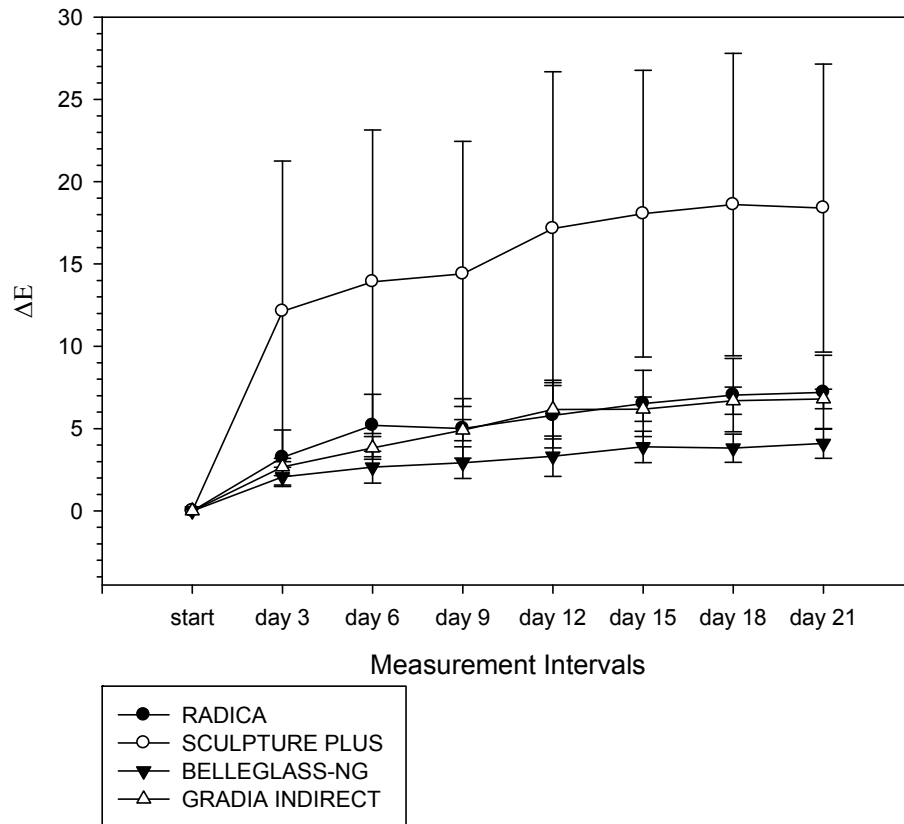
Staining Resistance (Enamel) – SCI component

Figure 9

Stainless Steel Mold for Gloss and Surface Roughness Test

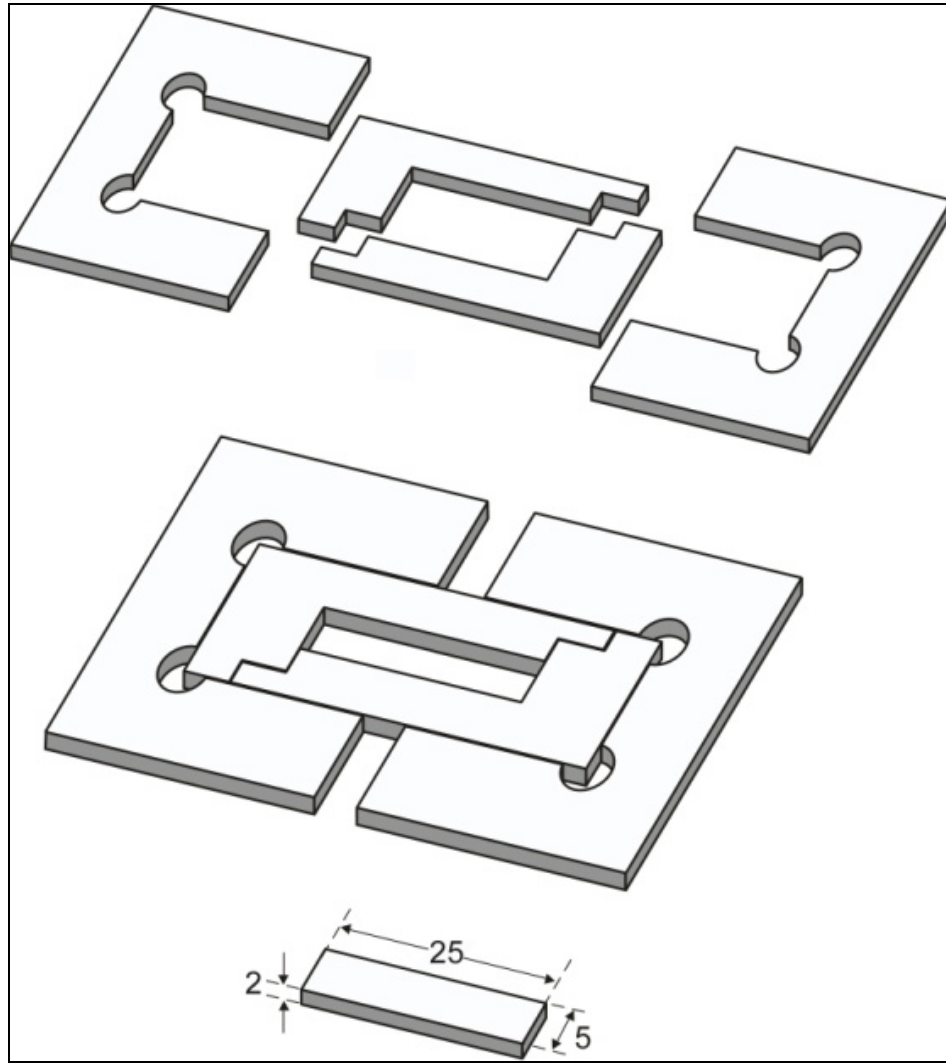


Figure 10

Glossmeter and Jig

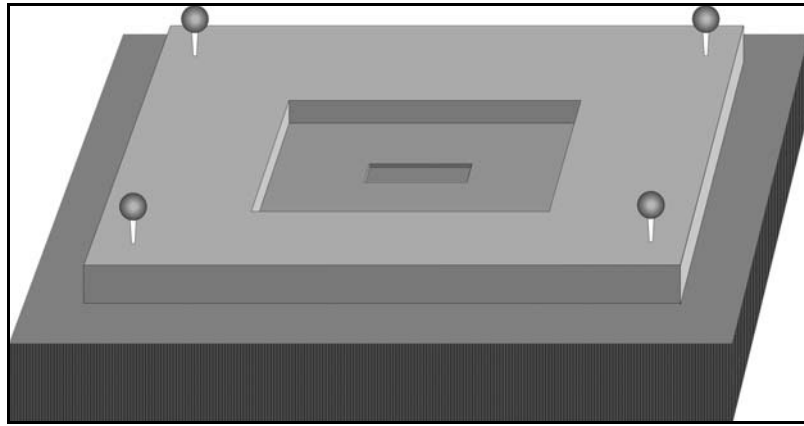


Figure 11

Profilometer for Measuring Surface Roughness



Figure 12

Tooth brush Direction for Gloss and Surface Roughness Test

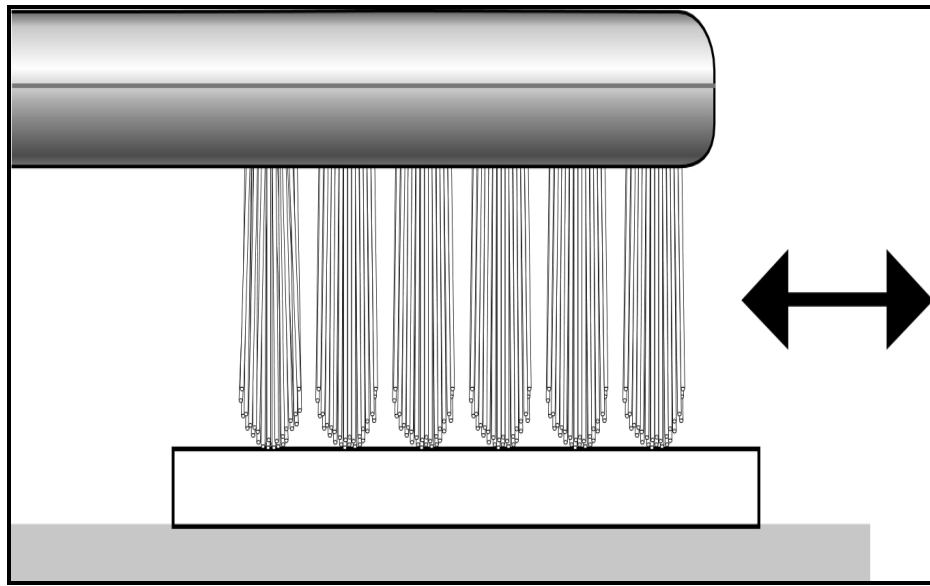


Figure 13

Gloss Versus Toothbrushing Cycles

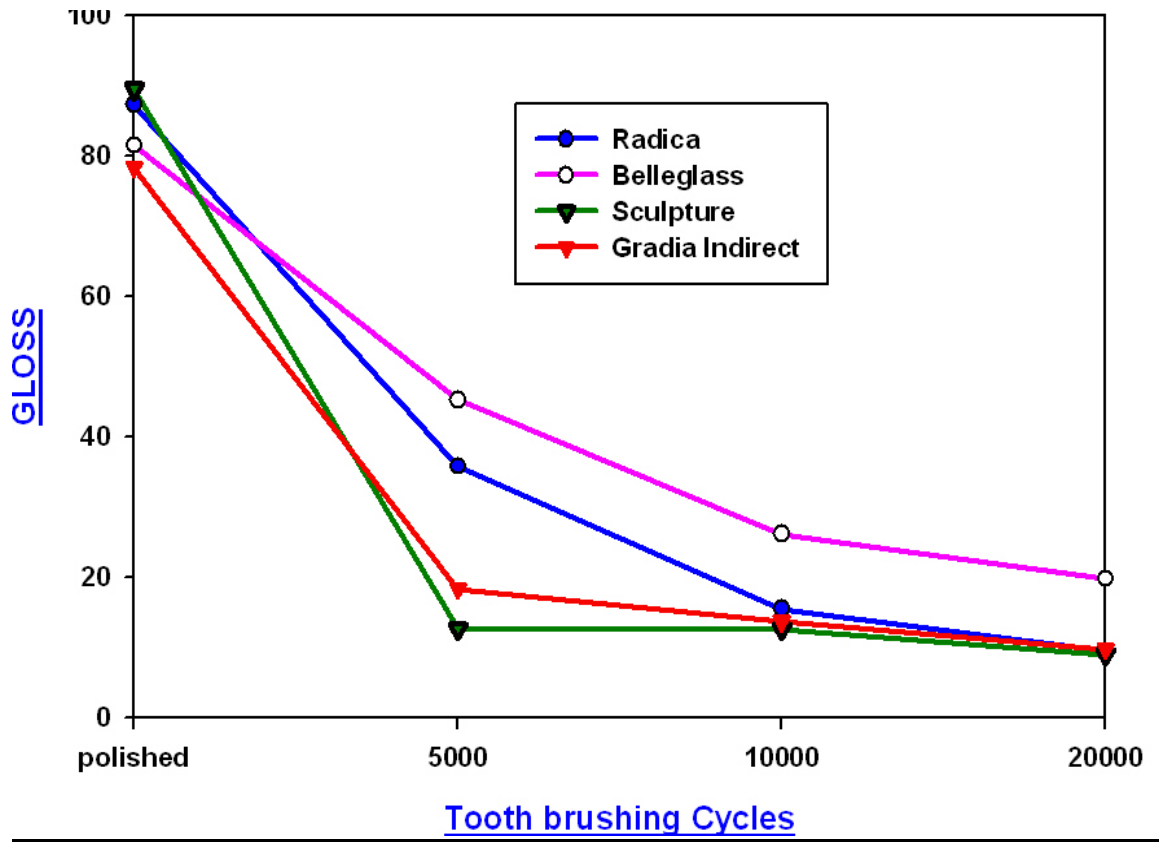


Figure 14

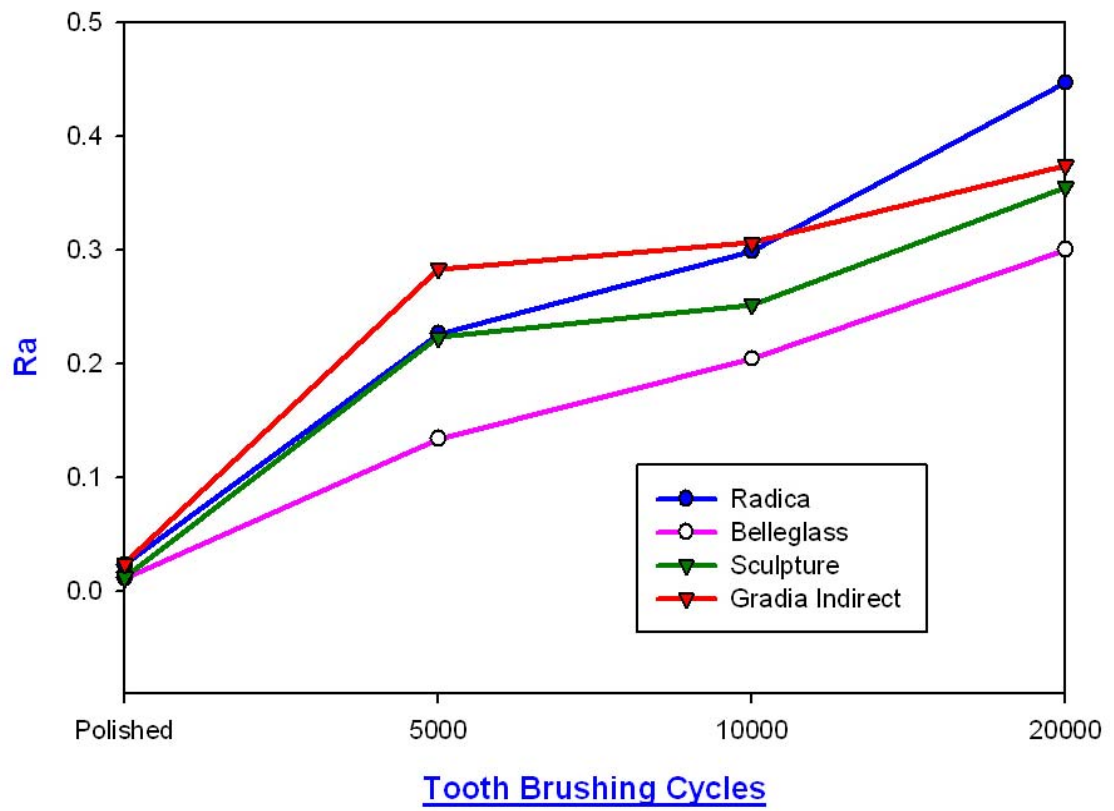
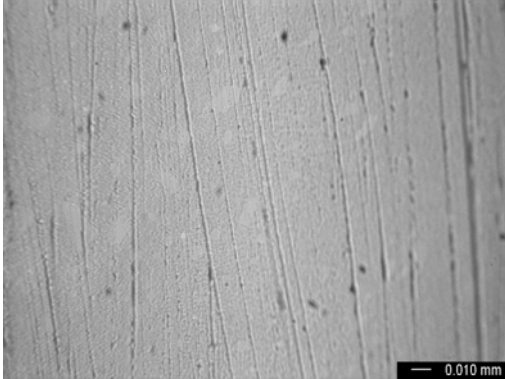
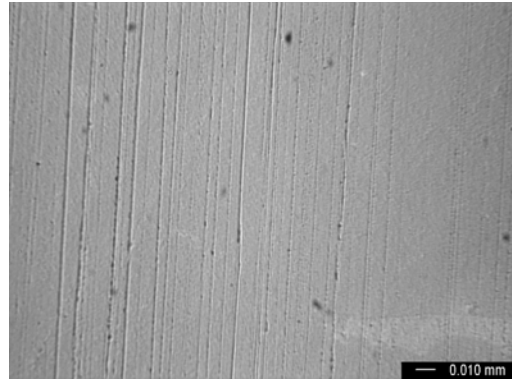
Surface Roughness (R_a) Versus Toothbrushing Cycles

Figure 15

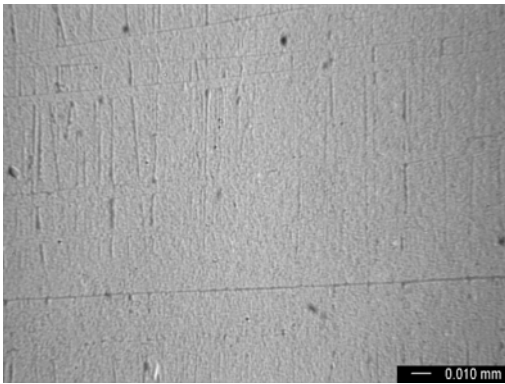
Surface Topography at 0 Cycles



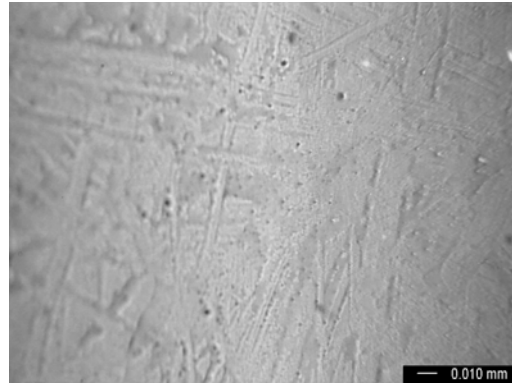
Radica



Belleglass-NG



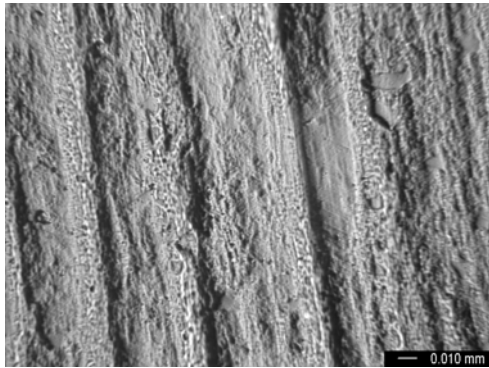
Sculpture Plus



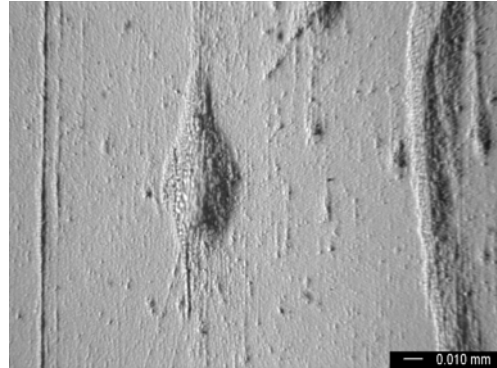
Gradia Indirect

Figure 16

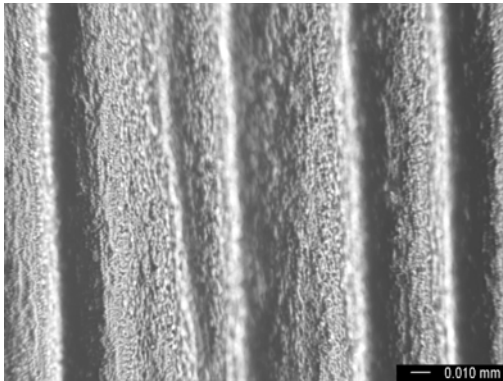
Surface Topography at 5000 Cycles



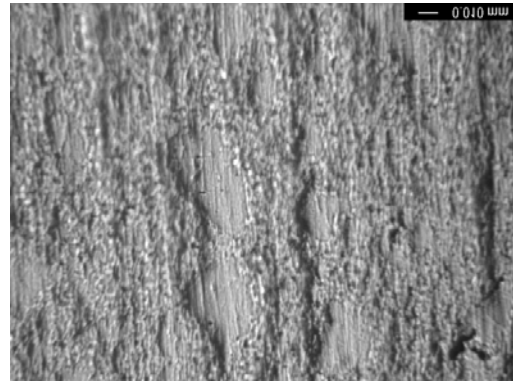
Radica



Belleglass-NG



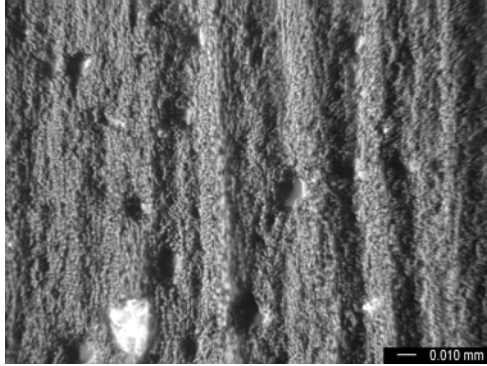
Sculpture Plus



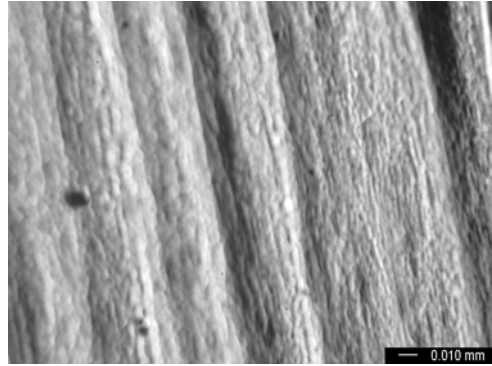
Gradia Indirect

Figure 17

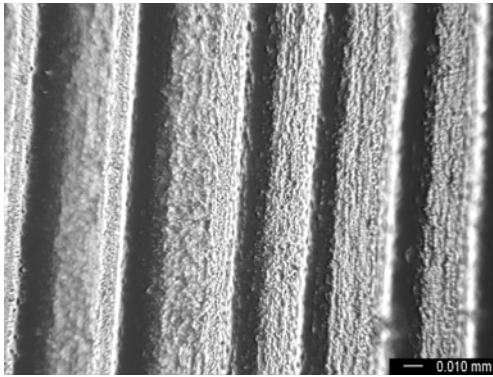
Surface Topography at 10,000 Cycles



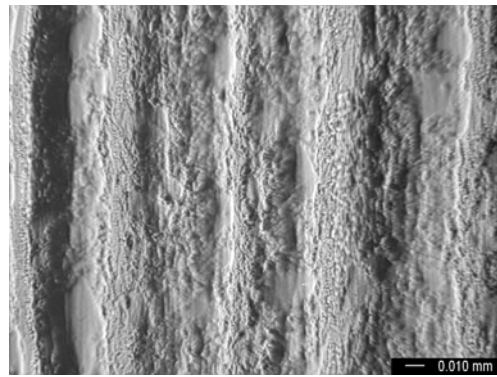
Radica



Belleglass-NG



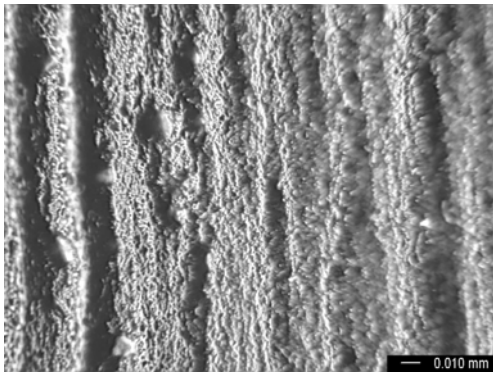
Sculpture Plus



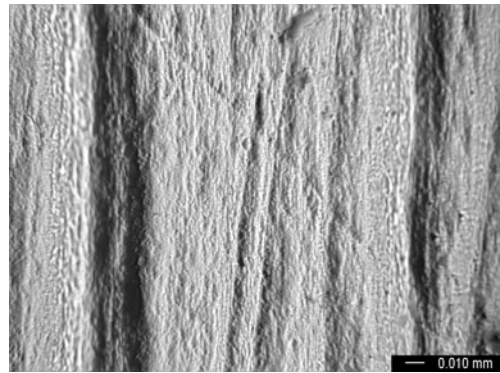
Gradia Indirect

Figure 18

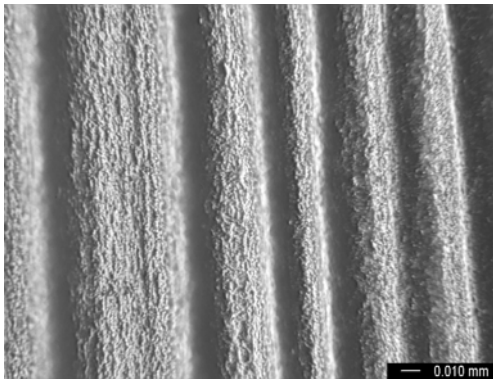
Surface Topography at 20,000 Cycles



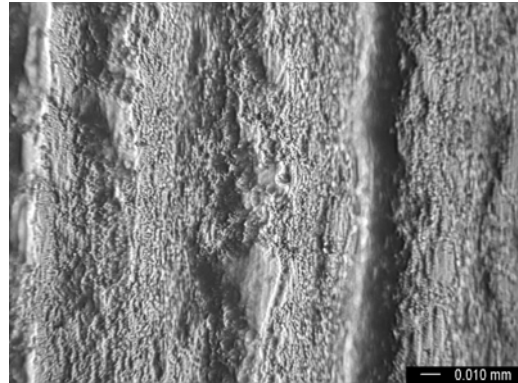
Radica



Belleglass-NG



Sculpture Plus



Gradia Indirect

Figure 19

Concept of Gloss

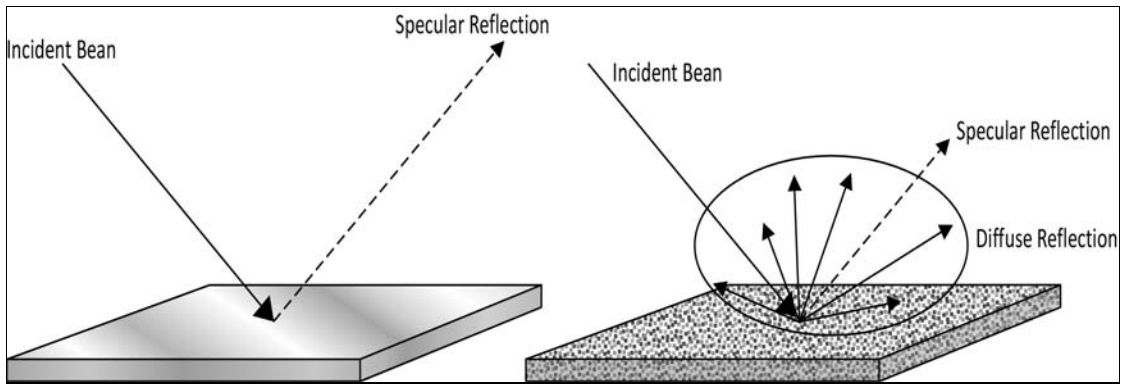


Figure 20

Volume Loss Due to Toothbrush Abrasion

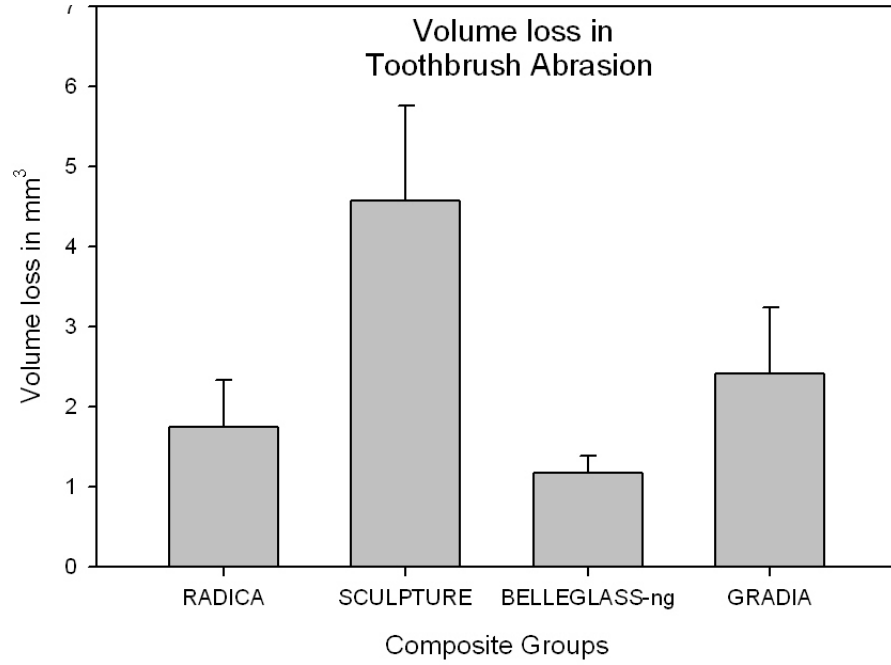


Figure 21

Three-body Alabama Wear Loss Results

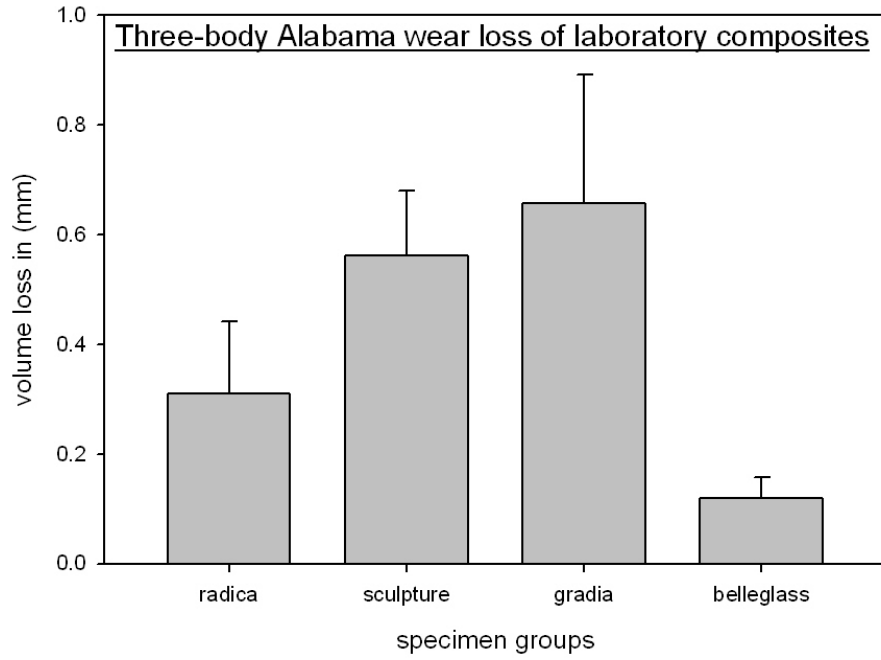


Figure 22

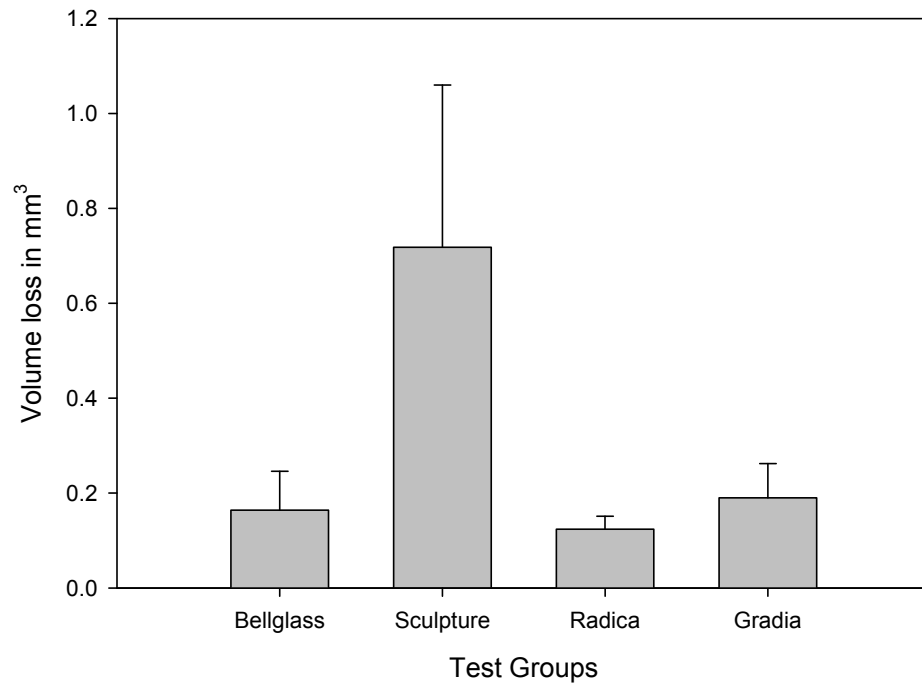
Two-body Wear Results

Figure 23

Fractured Surface Under the Digital Microscope

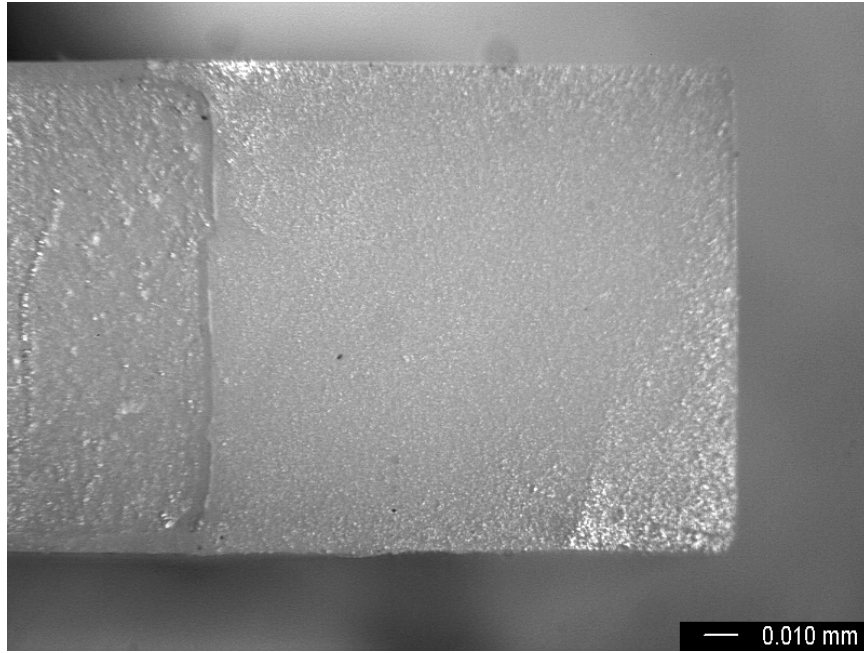


Figure 24

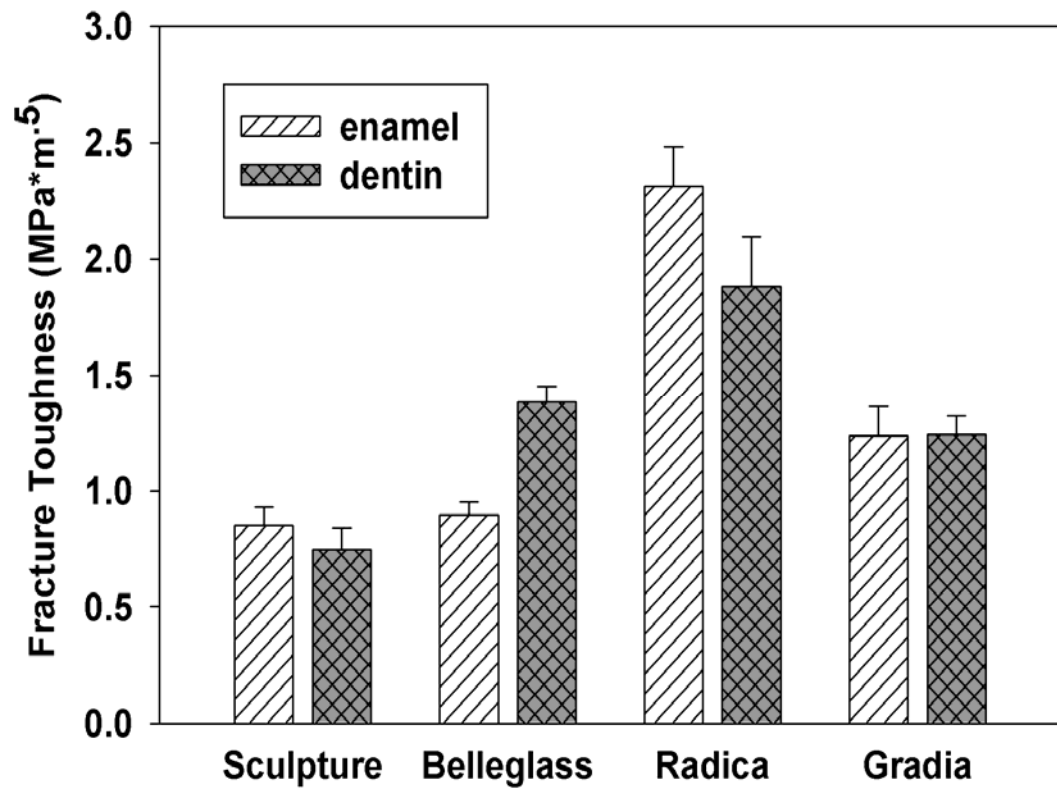
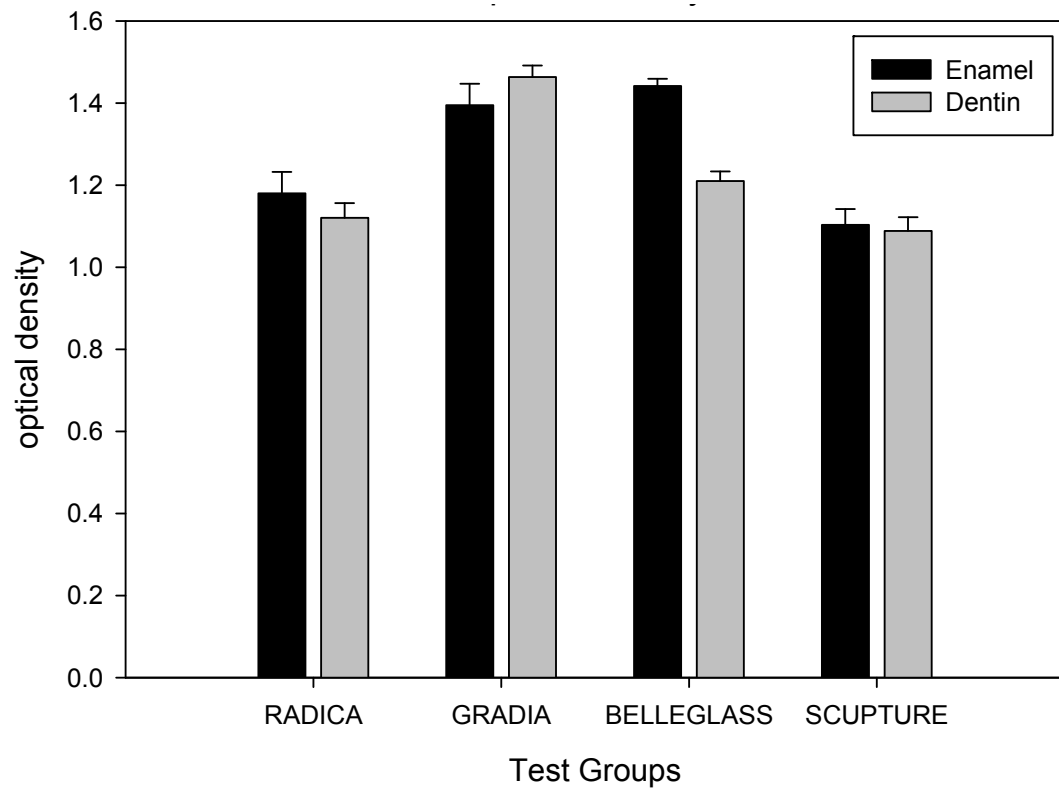
Fracture Toughness Results

Figure 25

Optical Density Results



DISCUSSION

Results from the evaluation suggest that there are indeed differences in the properties of the four commercial second generation indirect composites. The research hypothesis of the present study was rejected because there were significant differences in the changes of the properties in the four composites investigated.

Water Sorption and Solubility

Water plays an important role in the chemical degradation of the composite materials of the composite materials, resulting in both a hydrolysis reaction and swelling of the materials.^{34, 96} In that respect, the water sorption and solubility behavior of composite materials is of great interest.

In the present study, the null hypothesis that 'Water sorption and solubility values of the investigated composites are same' was rejected because significant differences were observed. Both enamel and dentin shades were compared. Indirect composites systems vary in composition according to shade. This is referenced by some manufacturers as well as in literature.⁹⁷ In the present study water sorption values were not influenced by the shade for most of the materials except for Belleglass-NG. However the solubility values were different for enamel and dentin shades for all the groups. Enamel shade showed higher solubility than the dentin shade in all groups but Radica.

When enamel shades were compared, water sorption values for Radica was lowest (mean = $13.29 \mu\text{g}/\text{mm}^3$) while Belleglass-NG had the highest water sorption (mean = $25.9 \mu\text{g}/\text{mm}^3$). When dentin shades were compared Gradia Indirect showed the highest water sorption (mean = $24.06 \mu\text{g}/\text{mm}^3$) while Radica and Belleglass-NG showed the lowest mean values of $13.83 \mu\text{g}/\text{mm}^3$ and $13.13 \mu\text{g}/\text{mm}^3$ respectively. This data is in agreement with other studies conducted on indirect composites for the same storage time.^{34, 36} Comparing with the data of direct composites from the literature^{98, 99}, it is observed that these indirect composites may show higher water sorption than the current commercial direct composites in the market. Sideridou et al¹⁰⁰ reported higher water sorption in composites with hydroxyl and urethane groups. Since Urethane Dimethacrylate (UDMA) is one of the primary constituents of the resin matrix in these composites, it may be one of the possible explanations for high water sorption.

Negative water solubility was observed in both enamel and dentin shades of Belleglass-NG and Gradia Indirect. This occurs because of the increase in the mass of the specimens which can be explained by two reasons. One of the explanations would be if the specimens are not completely dried and desiccated, then the presence of water will cause an increase in mass giving negative solubility values. However, adequate care was taken to prevent this and specimens were measured for a long period of time to get constant mass M_3 . Ortengren et al¹⁰¹ showed that increase in mass may occur due to a chemical reaction with water within composite. The glass filler and the metal oxides in the

composite material may hydrolyse with the metal hydroxides in reaction with water. This phenomenon was earlier described by Soderholm et al.²⁶

The sorption and solubility of the composites is known to be affected by elution of filler content, the unbound organic content and due to hydrophilic components in the matrix.^{34, 102} Storage time influences the water-sorption and solubility.³² The present study used the specifications proposed by ISO 4049. Ortengren et al³⁴ reported that pH influences the sorption and solubility behavior of the composite resins. In the present study de-ionized water was used for all the groups. The pH of the de-ionized water measured at 23°C was 7.4. Also the effect of water vapor absorption is substantial¹⁰³ and hence care was taken to measure the specimen in a constant temperature room (room temperature 23°C). The mode of curing may also influence these properties as it will improve the cross-linking density resulting in less elution of the substances.³⁶ The current study did not investigate the eluting substances and the effect of storage time and pH on the indirect composites. Further studies investigating these factors are recommended.

Staining Resistance

In this proportion of the study, the null hypothesis was rejected as significant differences were found among the four indirect composite groups in both enamel and dentin shades at each interval. All four groups showed increase in the mean ΔE over a 21 day period. The perceptible color changes (ΔE) ranged between 4.69 to 32.18 for enamel in SCE geometry and 4.52 to 25.6 in SCI

geometry. ΔE for dentin ranged between 4.79 to 22.9 in SCE geometry and 4.1 to 18.4 in SCI geometry. The mean ΔE values of all indirect composites were higher than that thought to be clinically acceptable ($\Delta E = 3.3$).^{48, 104} Data in the present study showed higher ΔE values than that reported by Stober et al.³⁸ Also dentin shade which is more opaque than enamel/incisal shade had lesser ΔE values. This observation was also reported by Lee et al.¹⁰⁵

Among the 4 groups, Sculpture Plus showed the lowest stain resistance compared to other indirect composites. Several factors may be responsible for this color instability. Differences in photoactivator formulation may be a causative factor.¹⁰⁶ Camphor-quinone is often added to the composite material as a photo-initiator. Residual molecules of this photo-initiator after polymerization are often responsible for yellowness taking place over time.⁴² The physico-chemical properties of the monomers in the resin matrices influence the adsorption of stains. The unconsumed initiators or monomers may have hydrophilized groups to uptake hydrophilic color molecules.⁴⁷ Dietschi et al.⁴⁹ suggested that composite with high filler content increases the staining resistance. As the exact composition is unknown, the actual causative factor has not been determined.

Restorations with high surface roughness are more susceptible to staining.⁴⁹ In order to decrease the influence of roughness in the current study, specimen surfaces were cured under a poly-ester film (Mylar strip). However, according to Shintani et al.¹⁰⁷ and Patel et al.¹⁰⁸, a poly-ester film finished surface caused a large color change. This may happen because the surface beneath the

polyester film strip does not appear to have the same degree of polymerization as the bulk of the resin material. It was expected that secondary curing in the form of heat and light, increased the degree of conversion¹⁰⁹, which may improve the staining resistance. However, the results obtained from the current study suggest that post-curing with increased temperature did not significantly improve the staining resistance of the composite to the clinically acceptable criterion.

The b^* co-ordinate indicates the yellowness in color. Among L^* , a^* and b^* values, the contribution of the b^* co-ordinate to the color change was highest. This may be as a result of the staining solution used in the study. Coffee contains yellow colorants, which have different polarities. The sorption of colorants into the organic phase of the material was probably due to compatibility of the polymer phase with the yellow colorants of the coffee.⁴⁸

The spectrophotometer with an integrating sphere can operate two different measuring geometries, Specular Component Included (SCI) and Specular Component Excluded (SCE). The specular component is the reflected light from the surface such that the angle of reflection is equal to the angle of incidence. The surface of the dental materials is not totally reflecting or matte. Thus both SCI and SCE may be important for color measurement in dental materials. Lee et al¹⁰⁵ showed that color changes measured with the SCE geometry were higher than those measured with SCE geometry. This observation correlates with the data in the present study.

This test was conducted in-vitro under stringent conditions. Specimens were tested under high concentration of coffee slurry for three consecutive days

between each interval. Clinical investigations on these materials may provide more conclusive data.

Gloss and Surface Roughness

In this proportion of the study, the null hypothesis was rejected.

Statistically significant changes were observed in gloss and surface roughness after toothbrushing. The images in Figures 17 to 20 correlate with the results presented in the table. Initially all the composite materials exhibited high gloss. By end of the first 10,000 cycles, all materials showed more than 75% reduction in the gloss. Decrease in gloss may be observed due to disruption of surface and exposure of filler caused by toothbrushing. Toothbrushing produces microscopic and macroscopic roughness that causes the incident light to be reflected in a diffuse manner, thus reducing the gloss.

Surface roughness was measured in terms of R_a which is an average of peaks and valleys of a surface finish. Several parameters are used to measure roughness such as R_z , R_{max} , R_t . However, in the dental literature, surface roughness is often reported in R_a . Therefore to correlate the data to other studies, the R_a parameter was used to measure surface roughness. Belleglass-NG showed the lowest R_a values at all intervals. This could be explained by the curing mechanism of Belleglass-NG. These composites were cured under nitrogen pressure at 140°C for 20 minutes which may have resulted in improvement of surface properties and degree of conversion.²¹ High roughness obtained for the Radica may be because of an increased number of microscopic

voids that were exposed when subjected to toothbrush wear. These microscopic voids could not be avoided during the fabrication of specimens as it was beyond the control of operator. These may remain a concern for the manufacturers as it adversely affects the properties of the materials.

Measurement of surface roughness was conducted using a contact profilometer with a stylus that moved along the length of specimen and parallel to the direction of toothbrushing. Three measurements were taken and their average was reported as mean R_a . Some authors have reported these measurements perpendicular to the direction of toothbrushing.⁶¹ These may result in different values than reported in the present study. R_a values measured parallel to the direction of toothbrushing may incorporate roughness caused due to loss of fillers. Sophisticated techniques such as Non Contact Optical Interferometers and Atomic Force Microscope (AFM) are available that measures roughness at a much higher resolution and include large area. Future investigations may avail these techniques for further in-depth analysis.

Increased resistance of the surface of the composite to toothbrush wear may improve the gloss property. This may explain why one composite, Belleglass showed higher gloss and lower R_a values than other comparative materials. However it cannot be inferred, whether the filler-matrix ratio, filler size and shape, mode of curing or any other factor or combination of factors played the most significant role in this process and thus further investigations are required.

Volume Loss due to Toothbrush Abrasion

In this proportion of the present study, the null hypothesis was rejected. All materials showed a volume loss after toothbrush abrasion. Statistically significant changes were observed in volume loss when compared among the four groups. Similar to the gloss and roughness test, the results of this test showed that Belleglass-NG demonstrated the least volume loss and thus exhibited the highest abrasion wear resistance when subjected to simulated toothbrushing. Mandikos et al²² reported Sculpture with lower wear and volume loss than Belleglass. The materials involved in the current study involve different versions, i.e. Belleglass-NG and Sculpture Plus which may have different composition and curing units. Sculpture Plus demonstrated high susceptibility to toothbrush wear. As explained in the gloss study, post-curing with high temperature and under nitrogen pressure may result in improvement of surface properties and degree of conversion. Although Sculpture Plus is cured in nitrogen pressure, the increased temperature, composition of resin and filler and their interaction may be possible factors in the low wear resistance. As the exact composition is unknown, the causative factors cannot be inferred.

To get an initial constant weight M_1 , the composites were kept in a dry and a desiccated environment. This does not simulate the oral environment. It also affects mechanical properties as observed in the water solubility test. Some studies²² have described obtaining M_1 weight in a 100% humid environment. However, as pursued obtained a constant weight to an accuracy of ± 0.1 mg.

Three-body Wear

Abrasion of tooth occurs in a three-body wear mode, and is generated by the sliding action of one tooth past another with force being transmitted through a layer of food that serves as a third-body medium. To simulate this phenomenon in the laboratory, the current study used, a three-body Alabama wear testing machine. This mechanism is different from toothbrush abrasion which is a much more complex wear phenomenon. The results obtained, however, were similar to the results in the toothbrush abrasion wear. The null hypothesis was rejected and statistically significant differences were observed among the four indirect composites. Belleglass-NG had the least volume loss while Sculpture plus and Gradia Indirect had the highest volume loss.

Condon and Ferracane⁶⁶ observed a linear relation between wear and volume of filler. Decrease in volume of filler was associated with increased wear. This may be a possible factor for high abrasion wear resistance in Belleglass-NG. Post curing with high temperature and nitrogen pressure may improve the surface conditions and abrasion wear resistance. According to de Gee et al¹¹⁰ heat treatment will accelerate the relaxation of the local stress conditions around the filler particles into a more homogenized distribution which will be maintained after cooling. In addition, the resin matrix and filler-matrix coupling may also influence the wear resistance.¹¹¹ As the exact compositions of the commercial composites are unknown, no correlations were made between the above factors.

Two-body Wear (Pin-on-disc test)

Attrition occurs in a two-body wear mode, and results from the direct contact of opposing teeth where the load level increases higher than that which produces abrasion.⁷³ In the current study, the null hypothesis was rejected. Significant differences were observed in the two-body wear among the four indirect composites. However, statistically no significant differences were observed among Belleglass-NG, Radica and Gradia Indirect. This may suggest that the material responded differently in the attrition and abrasion wear tests. Hence no correlation can be established between the Alabama wear test and Pin-on-disc test. This is consistent with a study conducted by Cha et al.¹¹² Sculpture Plus showed the lowest attrition and abrasion wear resistance.

The diameter of the antagonist in the current study was around 2 mm \pm 0.1 mm. The size of the antagonist is consistent with a previous study by Mark Beatty.⁹³ Other studies have reported an antagonist diameter of 5mm and 10mm.¹¹² Jaarda et al¹¹³ reported that a reduced diameter resulted in higher attrition and had no significant influence on other forms of wear. Thus a diameter of 2 mm would represent higher attrition and also be more clinically relevant. Marquis et al⁸¹ reported that wear increased steadily under increasing loads. In the current study, a constant load of 2.8 kg-force was applied that produced contact stresses of 0.81-1.00 kg/mm² (1 MPa = 1 N/mm² = 0.102 Kg/mm²) which is clinically relevant.¹¹⁴

Fracture Toughness

In this proportion of the present study, the null hypothesis was rejected as significant differences were found in the K_{IC} values of the four indirect composite groups for both enamel and dentin shades. The highest K_{IC} values were observed for both enamel and dentin shades of Radica. When enamel shades were compared among other groups, Sculpture Plus and Belleglass-NG showed the lower K_{IC} values compared to Gradia Indirect. When dentin shades were compared, Sculpture Plus showed lower K_{IC} values than Belleglass-NG and Gradia Indirect.

All indirect composites in this study may have nearly similar filler content but the viscosity of the resin may play an important role in increasing the fracture toughness. Musanje and Ferracane¹¹⁵ reported that medium-viscosity composites containing an equal mixture of BIS-GMA/TEGDMA/UDMA provided optimum mechanical properties. Earlier Uctasli et al⁸⁴ reported that increasing temperature and pressure during processing may improve the fracture properties. However in the current study, the effect of temperature was not observed. Belleglass-NG has highest curing temperature, but showed very low K_{IC} values.

Filler addition may result in branching of cracks increasing the crack surface area and resultant fracture energy.¹¹⁶ Kim et al¹¹⁷ reported an increase in fracture toughness with an increase in filler level up to a threshold level of 55% volume. Pre-polymerized fillers decreased the net filler volume. However in the

current study, the net filler volumes of the composites are not reported and therefore its influence on the K_{IC} cannot be determined.

Although several methods are available for measuring fracture toughness, such as the 3-point bend test, indentation hardness and the single-edge notch beam, it is essential that the fracture toughness is measured in plane strain condition to obtain a true measure of fracture toughness.¹¹⁸ The current study used the ASTM standard.⁵⁴ Theoretically to satisfy the plane strain conditions, the specimens have to be infinitely thick. A thicker specimen would allow minimal plastic flow adjacent to the crack tip, to test the material in true plane strain conditions.¹¹⁹ Kowarik et al¹²⁰ showed that plane strain conditions were satisfied if the specimen thickness was greater than 1.6 mm. In the current study the thickness of the specimen was 2 mm. This is clinically relevant as many of the restorations have similar thickness.

Radiopacity

The radiopacity test performed according to the ISO 4049 test method provided the comparative information for the test materials. Radiopacity is an essential property of all the restorative materials and the ISO specification 4049 requires that minimum radiopacity must be equal to or greater than a 1 mm of aluminum thickness. The current study was carried out to compare the radiopacity of four indirect composites. Comparison was made on the basis of the optical density because the composites showed radiopacity greater than a minimum aluminum thickness of 1 mm and less than 2 mm thickness. The optical

density measured by the transmission densitometer used in the study gives the indication of radiopacity. The higher the optical density, the lower is the measure of radiopacity.

In the current study optical density of the dentin disc of 1mm thickness was 1.25 units. Using the conventional X-ray films all the composites except Gradia Indirect and the enamel shade of Belleglass-NG showed lesser optical density than a dentin disc. This means they were less radiopaque than dentin. The null hypothesis was rejected as significant differences were observed in the radiopacity of different materials. The highest radiopacity was observed in Sculpture Plus and the lowest was observed in Gradia Indirect. Enamel and dentin shades did not have a significant difference in radiopacity except in Belleglass-NG. This may suggest that fillers in the enamel and dentin shade responsible for bringing translucency and shade difference may not significantly influence the radiopacity.

Langland and Langlias¹²¹ classified factors influencing radiopacity as primary and secondary factors. Primary factors include milliampere, exposure time, kilovoltage and source-film distance. Secondary factors include development conditions, type of film, intensifying screens and grids. In the current study these factors were kept constant as specified in ISO 4049. Sabbagh et al¹²² reported filler percentage and types as the most important factors that influence a material's radiopacity. Toyooka et al¹²³ reported that radiopacity is linearly proportional to the amount of radiopaque oxide in the filler.

Radiopacity of human enamel and dentin vary considerably depending on the individual, age, site and storage conditions. Only one dentin disc was used in this investigation. This is a limitation in this study. Future investigations must be carried out, considering these factors. Within the limitations of this study, the indirect composites investigated with the ISO specification showed radiopacity close to or less than the dentin disc used in this investigation. Use of materials with radiopacity close to or less than dentin may result in future diagnostic challenges. The clinical application of these materials requires further study.

SUMMARY AND CONCLUSION

The purpose of this study was to evaluate and compare the water sorption and water solubility, staining resistance, gloss and surface roughness after simulated tooth-brushing, volume loss after simulated toothbrush wear, three-body wear, two-body wear, plane strain fracture toughness and radiopacity of the four commercial second generation indirect composite systems. The null hypothesis was 'there is no significant difference between the four commercial indirect systems for each of the investigated properties'.

Low water sorption and solubility was observed for both enamel and dentin shades of Radica compared to other comparative materials. Negative water solubility was observed in Belleglass-NG and Gradia Indirect. Negative solubility values may be due to chemical reaction of water within the composite. All composites showed color change (ΔE) in the stain resistance test when exposed to coffee slurry. The mean ΔE was higher than the clinically acceptable value of 3.3. Belleglass-NG demonstrated the least color change while Sculpture Plus demonstrated the highest color change. Among L^* , a^* and b^* values, the contribution of the b^* co-ordinate to the color change was highest. Dentin shade showed less color stability than enamel shade.

All the indirect composites investigated in this study showed a significant decrease in gloss and increased surface roughness. Belleglass-NG showed some resistance to retain gloss during the first 5000 cycles. However, by the end of 10,000 cycles, all the materials had lost the initial high gloss, by more than

75%, achieved after polishing. Surface roughness was highest in Radica. Micro-porosities were observed under the microscope and this could be a possible factor in the increased roughness observed for this material.

Belleglass-NG demonstrated the least volume loss and thus exhibited the highest abrasion wear resistance when subjected to simulated toothbrushing and three-body Alabama wear testing. The lowest wear resistance (two-body and three-body) was observed in Sculpture Plus. Radica exhibited the highest fracture toughness. All the indirect composites investigated in this study had radiopacity equal to or less than dentin.

In conclusion, significant differences were determined in the second generation composite systems evaluated in terms of water sorption and water solubility, staining resistance, gloss, surface roughness, three-body wear, two-body wear, plane strain fracture toughness and radiopacity. Differences in the formulations and/or the curing mechanisms of these indirect composites offer different advantages. Staining and wear resistance is a concern for long term use of indirect composites in clinical application. Nevertheless, controlled long term clinical studies are required to confirm the clinical significance of these differences in the investigated properties.

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CURRICULUM VITAE

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Education

Master of Science in Dental Materials, Indiana University, Indianapolis, Indiana

Bachelor of Dental Surgery (BDS), Government Dental College and Hospital, Mumbai, India

Research and Training Experience

- Oral Health Research Institute (OHRI, Indianapolis, Indiana):
Development of the model comparing potential of dentifrice to polish the tooth surface.
- Dentsply International, York Town, PA: Wear analysis and investigation of nightguard materials.
- Materials Science Engineering Department, Indian Institute of technology, IIT- Mumbai, India: Synthesis and Development of Glass Ionomer Cements for dental Applications.

Conferences Attended

- 'Gloss and Surface Roughness in Second Generation Indirect Composite Resins' poster presentation at the annual conference of Academy of Dental Materials, (ADM) Fort Lauderdale, Florida (October 2007).
- 'Wear in Second Generation Indirect Composite Resins.' poster presentation at the annual conference of American Association of Dental Research (AADR), Dallas, Texas (April 2008)
- 'Color Stability in Second Generation Indirect Composite Resins' poster presentation at the annual IUSD Research day, an event organized by the Indiana section of AADR (April 2008).