AN *IN VITRO* STUDY OF MICROLEAKAGE AND SURFACE MICROHARDNESS OF NANOCOMPOSITE RESTORATIVE MATERIALS

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Keywords

Microleakage

Sealing ability

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Surface microhardness

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Ormocers

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SUMMARY

The demand for posterior aesthetic restorations has dramatically increased in recent years. Several new materials have been developed with improved aesthetic, adhesive and mechanical properties. However microleakage elimination, especially if the margins are on dentine or cementum, and attainment of optimum mechanical properties for posterior use, are still questionable. **Aim:** The aim of this study was to investigate microleakage and surface microhardness of a nanofilled, a nanohybrid and an ormocer based nano-ceramic composite restorative material. **Materials & Methods:** Four light-cured dental restorative materials in combination with their respective bonding agents were investigated: Z100/Adper Scotchbond (3M ESPE), Ceram X/Prime and Bond NT (Dentsply), Grandio/Admira Bond (VOCO), Premise/Optibond Solo Plus (Kerr). For the determination of *Microleakage,* one hundred and sixty Class II slot cavities (3 x 5 x 2 mm) were prepared in extracted human molars and filled with the four restorative materials. Half the samples had their gingival margin on enamel and the other half on dentine or cementum. After thermocycling and immersion in 0.5% methylene blue dye solution, the teeth were sectioned and dye penetration was scored under a stereomicroscope at 10X magnification. For the measurement of *surface microhardness* sixty-four disc shaped specimens 5mm in diameter and 2 mm in height were used. Microhardness values were measured on the top and the bottom surfaces using a Vickers hardness tester. **Results:** The microleakage tests revealed that the differences between Ceram X, Premise and Grandio were not statistically different ($p>0.05$) in their sealing ability at an enamel margin. However the differences were statistically significant when compared to Z100 ($p\leq0.05$). The differences in the sealing ability of Ceram X, Premise, Grandio and Z100 in their sealing ability at dentin or cementum margin were statistically significant ($p\leq0.05$). The differences between Ceram X and Premise were also statistically significant ($p\leq0.05$) from Grandio and Z100. The differences

for Grandio were also statistically significant ($p \leq 0.05$) from Ceram X, Premise and Z100. For surface microhardness the test revealed that Z100 and Grandio were significantly different ($p<0.05$) from Ceram X and Premise. There was no significant difference between Z100 and Grandio and between Ceram X and Premise. Ceram X and Premise also had differences that were statistically significant ($p<0.05$) for the top and bottom surface hardness values. **Conclusion:** Within the limitations of this study no material was able to eliminate microleakage at dentine or cementum margin. Grandio + Admira Bond showed the best marginal sealing ability as compared to the other tested materials. Z100 and Grandio also showed higher surface microhardness values both at the top and bottom surfaces of tested specimens.

DECLARATION

I hereby declare that *An In vitro Study of Microleakage and Surface Microhardness of Nanocomposite Restorative Materials* is my own work, that it has not been submitted before for any degree or examination in any university, and that all the sources I have used or quoted have been indicated and acknowledged by complete references.

Abdul Majeed

September 2005

Signed:……

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DEDICATION

To my mother and my father for their constant support and sacrifice.

***To my supervisor whose guidance, encouragement, help and support made this project possible*.**

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CHAPTER 1

INTRODUCTION

Dental composite restorative materials have been available since the early 1960s (Loguercio *et al*, 2004). Their use in posterior teeth has been recommended for more than 20 years (Türkün, Aktener and Ateş, 2003). In recent years, the demand for posterior resin composite restorations has dramatically increased because of their ability to match tooth color, absence of mercury, biocompatibility and bond to tooth structure (Herrero, Yaman and Dennison, 2005, Hilton, Schwartz and Ferracane, 1997).

Modern posterior resin composites undergo 2.6 to 7.1 % volumetric contraction during polymerization (Hilton, Schwartz and Ferracane, 1997). This shrinkage can result in a gap formation between the composite material and tooth structure, particularly if the restoration margin is placed in dentine or cementum (Yazici, Celik and Ozgunaltay, 2004). Bacteria, fluids, molecules, or ions can pass through this gap between the resin composite and the cavity wall, a process called *microleakage* (Hilton, Schwartz and Ferracane, 1997). Microleakage is thought to be responsible for hypersensitivity, secondary caries, pulpal pathoses and failure of restorations (Franco *et al*, 2003).

The success and longevity of a dental composite restorative material is also dependent upon the attainment of optimum mechanical properties (Coffey *et al*, 2004). Mechanical properties such as microhardness and wear resistance amongst others change with the degree of monomer conversion (Tantbirojn *et al*, 2003). Restoration softness, excessive wear, loss of biocompatibility, color shift, breakage and loss of retention

have all been associated with inadequate polymerization (Coffey *et al*, 2004).

Over time several changes have been made in formulation to produce materials for adequate clinical success. The latest innovations are the development of dental composites based on nanotechnology. The newly available nanomaterials, such as nanofillers and nanohybrids enable the dental composites to be improved (Mitra, Dong and Holmes, 2003). These materials have a very low degree of polymerization shrinkage and excellent esthetic properties. Their physical properties are comparable to other composite materials (hybrids and packables). These materials can be used for anterior and posterior restorations (Ho, 2004).

However, problems, such as wear, technique sensitivity and microleakage arise when resin-based composite restorations are placed in posterior teeth. Direct Class II restorations can be placed to an acceptable standard if the gingival margin is on sound enamel; however, the quality of the marginal sealing of adhesive restorations located below the cementoenamel junction is still questionable (Loguercio *et al*, 2004).

Therefore the aim of this *in vitro* study was to investigate the microleakage in Class II restorations using nanocomposite dental restorative materials. The surface microhardness of these composite restorative materials would also be determined. The materials that would be tested in this study have only recently been launched commercially; as such independent data regarding *in vitro* or *in vivo* studies about these materials is limited.

CHAPTER 2

LITERATURE REVIEW

2.1- INTRODUCTION

The demand for posterior aesthetic restorations has led to the development of several new restorative materials. Recently, nanotechnology or molecular engineering is being used extensively to produce restorative materials with improved aesthetics, adhesive and mechanical properties. Materials based on nanotechnology are also known as nanocomposites.

Ceram X by Dentsply (Germany), Premise by Kerr (USA), and Grandio by Voco (Germany) are some of the newly marketed nanocomposite restorative materials.

Microleakage at the tooth-restorative interface results in postoperative sensitivity, marginal deterioration, recurrent caries and pulpal injury. It is also a major concern influencing the clinical longevity of composite resin restorations (Chimello *et al*, 2002). Among other factors, the attainment of optimum mechanical properties upon curing is another requirement for the longevity of resin composite restorative materials. Such properties are dependent on adequate polymerization, which ensures that the residual monomer content in the restoration is minimized (Coffey *et al*, 2004).

The literature review will focus on microleakage and factors contributing to microleakage, different methods of evaluating microleakage, surface microhardness, and developments in resin-based composite restorative materials. In the methods used to evaluate microleakage the main

emphasis is on dye penetration and the sectioning of the teeth. In the latter part of the review, the tested materials will be discussed briefly.

2.2- MICROLEAKAGE

2.2.1- Introduction and Definition

The integrity and durability of the marginal seal has always been of prime concern in the investigation of the performance of a dental restorative material (Gwinnett *et al*, 1995).

"Microleakage is defined as the clinically undetectable passage of bacteria, fluids, molecules or ions between a cavity wall and the restorative material applied to it" (Kidd, 1976a).

Bergenholtz *et al*, (1982) as well as Brannstrom (1987) clearly documented the effects of bacterial leakage upon the dental pulp. Clinically microleakage can be identified as a dynamic phenomenon that results in two consequential manifestations known as the sensory component and the pathologic component. Hypersensitivity to thermal and osmotic stimuli as a result of a compromised marginal seal that causes hydrodynamic fluid movement through a degrading smear layer into the patent dentinal tubules underneath is referred to as the *sensory* component of microleakage. Bacteria and their products that pass through such potential gaps along the axiopulpal floor and result in recurrent caries and subsequent pulpal pathoses accounts for the *pathologic* component of microleakage (Gwinnett *et al,* 1995).

Besides pulpal irritation and secondary caries, microleakage also results in marginal discoloration (Going, 1972, Fleming *et al*, 2005). It has also been reported as one of the major causes of composite resin restoration failure (Rosin *et al*, 2002).

2.2.2 – Factors Contributing to Microleakage

Several factors affect the integrity of the tooth-restoration interface and can contribute to microleakage. These factors include:

- Physical properties of materials
    - Polymerization shrinkage
    - Coefficient of thermal expansion
    - Modulus of elasticity
- Hydroscopic expansion
- Adhesive bond strength
- Dry versus wet or moist bonding
- Light polymerization concepts and units
- Cavity configuration factor
- Thermal cycling and occlusal stresses

Polymerization Shrinkage

Polymerization shrinkage is one of the most critical properties of resin based composite restorative materials (Chen *et al*, 2001). It is considered as one of the major problems that still imposes limitations in the application of direct aesthetic restorative techniques (Loguercio *et al*, 2004, Yazici, Celik and Ozgunaltay, 2004, Yap *et al*, 2000).

Polymerization shrinkage of dental composites ranges between 2% and 6% by volume (Braga and Ferracane, 2004). Resins shrink during polymerization because the monomer units of the polymer are located closer to one another than they are in the original monomer (Chen *et al*, 2001). Besides volume reduction, chain growth and cross-linking during polymerization of resin composites also results in an increased elastic modulus (Dauvillier *et al*, 2000a).

During polymerization, *gelation* or *gel point* is a stage in monomer conversion at which the elastic modulus of the composite increases substantially to a level that does not allow plastic deformation or flow to compensate for the reduction in volume (Braga and Ferracane, 2004).

Total polymerization shrinkage can be divided into two components: the pre-gel and post-gel phases. During the pre-gel polymerization, the cross linking density is low and polymeric chains are able to assume new positions (flow), causing stress relieve within the structure (Calheiros *et al*, 2004, Yap *et al*, 2000). During post-gel polymerization additional contraction produces clinically significant stresses in the composite-tooth bond and surrounding tooth structure (Braga and Ferracane, 2004).

Post-gel polymerization stresses are not uniformly distributed along the cavity walls (Shono *et al*, 1999) and the bond strength between tooth and composite also varies along the bonded interface (Kinomoto and Torii, 1998). Therefore, in areas where shrinkage forces are higher than the composite-tooth substrate bond, a gap may develop leading to bond failure and microleakage with associated postoperative sensitivity and secondary caries (Franco *et al*, 2003, Yap *et al*, 2000). Polymerization contraction stresses transferred to the tooth can cause tooth deformation that results in post-operative sensitivity and may open pre-existing enamel microcracks (Versluis *et al*, 1996). Another consequence of polymerization shrinkage in composite restorations is cuspal movement. Movements up to 46 μm have been reported when restorations were placed in a single step (Ensaff, O'Doherty, and Jacobsen, 2001).

Manhart, Garcia-Godoy and Hickel, (2002) stated that the extent of the shrinkage depends on the molecular weight and the functionality of the monomer, the filler load and the technology of the filler particles.

Based on the above, bonding agents with bond strengths greater than contraction stresses, composites with low polymerization shrinkage and optimal cavity designs should be developed to obtain a stable marginal seal of composites in dentinal cavities (Davidson, de Gee and Feilzer, 1984).

Polymerization contraction stress is mainly influenced by the composite's volumetric shrinkage and its visco-elastic behavior that is usually described in terms of elastic modulus development and flow capacity (Calheiros *et al*, 2004).

Modulus of Elasticity

Young's modulus of elasticity describes the rigidity or stiffness of a material (Phillips, 1991). The modulus of elasticity can influence the sealing ability of a resin composite material. During the pre-gel phase of polymerization, cross linking density is low and the resin composite is able to flow, that is, the resin composite has a low modulus of elasticity that helps to relieve the polymerization contraction stresses (Calheiros *et al*, 2004, Chen *et al*, 2001). Following gel formation, there is a rapid increase in the elastic modulus of the resin composite. This results in further contraction stress development but the material is too rigid to allow sufficient plastic flow to compensate for the original volume with possible gap formation (Chen *et al*, 2001).

Volumetric shrinkage and elastic modulus are highly dependent upon the filler content of materials, though in opposite ways (Labella *et al*, 1999). Composites with relatively higher filler content have a low resin matrix fraction that actually determines the volume reduction observed during the formation of a dense cross-linked polymeric network. Conversely materials heavily filled with inorganic particles present high stiffness that is also associated with high stress levels by reducing the

materiel's flow capacity (Dauvillier, Aarnts, and Feilzer, 2000b). This may cause destruction of the tooth-restoration bonded interface, thereby increasing the chance of microleakage. In general terms, the higher the volumetric contraction or the faster the material acquires elastic properties after the beginning of polymerization, the higher the stresses will be (Calheiros *et al*, 2004).

The resin-dentine interdiffusion zone or hybrid layer has a relatively low Young's modulus and may therefore also relieve some polymerization contraction stresses (Van Meerbeek *et al*, 2003).

Another potentially serious effect of shrinkage is thought to be the stress placed on the tooth substance, particularly on the residual cusps of posterior teeth when composite restorative materials are placed in relatively large Class II cavities (McCabe and Walls, 1998). The modulus of elasticity of enamel (33.6 GPa) and dentine (11.7 GPa) is greater than that of composites at 10.5 GPa for condensable composites (Agosta and Estafan, 2003). Micromovement of resin may occur under stress because composite resin is a flexible material with elastic properties due to the internal weak bonds, while enamel does not deform under compressive strength before fracturing. This may cause bond failure at the tooth restoration interface resulting in microleakage and percolation of fluids or a fracture of the tooth surface (Agosta and Estafan, 2003).

Coefficient of Thermal Expansion

The restorative materials constantly undergo changes of a thermal nature when placed in the oral environment, due to intake of food and fluids at varying temperatures. Such changes, if significant, can have unfavorable effects on the margins of the restorations in terms of

maintaining the seal at the tooth-restoration interface (Sidhu, Carrick, and McCabe, 2004).

Dimensional changes of a substance in response to thermal variations are measured in terms of its Coefficient of Thermal Expansion (CTE). The restorative materials have a different coefficient of thermal expansion from that of enamel and dentine (Hilton, Schwartz and Ferracane, 1997).

The coefficient of thermal expansion of a tooth has been reported within a range of 11-14 x 10^{-6} $^{o}C^{-1}$ (Sidhu, Carrick, and McCabe, 2004). While for some commercial pit and fissure sealants this coefficient has been found to be in the range of 70.9 to 93.7 x 10^{-6} $^{o}C^{-1}$. For the various commercial resin composites this range has varied from 20 to 80 x 10^{-6} $^{o}C^{-1}$, in the temperature range of 0–60^{o}C (Sideridou, Achilias, and Kyrikou, 2004).

A great difference in the coefficient of thermal expansion (CTE) between tooth and restorative material results in different dimensional changes, occurring when the restored tooth is subjected to varying oral temperatures (Craig and Powers, 2002). The restorations tend to expand and contract differently as compared to the tooth structure. Such expansion and contraction develops stresses at the tooth-restorative interface that may lead to debonding and gap formation or cusp fracture if the bond persists. These complications result if the tooth is not able to tolerate the changes induced by the temperature variations (Sideridou, Achilias, and Kyrikou, 2004, Sidhu, Carrick, and McCabe, 2004, Agosta and Estafan, 2003).

Failure of the adhesive bond is a result of a number of factors; however the difference in the CTE between tooth structure and restoration is regarded as the major factor (Sideridou, Achilias, and Kyrikou, 2004). A strong correlation has been found between microleakage and the

differences in the coefficient of thermal expansion of the various materials (Bullard, Leinfelder, and Russell, 1988).

Hydroscopic Expansion

Polymerization reaction and subsequent interaction with the aqueous oral environment may result in a series of physical changes in the resin based composite restorative materials (Martin, Jedynakiewicz, and Fischer, 2003). Resin based composite restorative materials may absorb significant amounts of water when exposed to the oral environment (Soderholm *et al*, 1984). Water sorption may produce some undesirable effects such as dissolution, hydrolysis, expansion, plasticization, microcrack formation and fatigue. This degradation of its physical properties decreases the life expectancy of dental restorative materials (Laguvardos *et al*, 2003).

Most polymer-based materials absorb water through a diffusion-controlled process. A number of factors including type of resin, filler fraction, filler size, reactivity of the glass, and the presence of silane and non-silane coupling agents, determine the diffusion coefficient of dental composite restorative materials. Of these, the nature of the resin matrix has the most significant bearing on the amount and rate of hydroscopic expansion for any given resin-based composite restorative material (Martin, Jedynakiewicz, and Fischer, 2003).

Water sorption will cause a change in the dimension and the weight of the set material (Momoi and McCabe, 1994). Hirasawa *et al*, (1983) reported a direct correlation between the mass of absorbed water and the linear expansion of the composite resin. This expansion may relieve some of the internal stresses produced during polymerization shrinkage of the restoration or may close marginal leakage gaps (Martin, Jedynakiewicz, and Fischer, 2003). However, the adhesive bonds that

were broken by the polymerization shrinkage will not be re-established by hydroscopic expansion (Allen *et al*, 1994).

Adhesive Bond Strength

Several factors affect the quality of the bond including the thickness of the smear layer, variations in resin penetration into the demineralized surface and stresses developed at the adhesive–dentin interface during polymerization shrinkage and function (Jacobsen *et al*, 2003).

Kanca (1989) reported that there was no statistically significant correlation between bond strength and microleakage. It may seem logical for bond strength and microleakage to have an inverse relationship, however leakage can take place around foci of adhesion and not necessarily dependent on a total break between tooth and restoration (Borem and Feigal, 1994).

A bond strength of 20 to 24 MPa is necessary to resist polymerization contraction stresses of resin composites and to prevent microleakage at the dentine-resin interface (Retief, Mandras, and Russell, 1994). Current dentine bonding agents are incapable of preventing microleakage because they cannot withstand the shrinkage forces generated during the polymerization reaction (Van Meerbeek *et al*, 2003 and Perdigão *et al*, 1996).

Application of flexible adhesive lining has been shown to release some of the stresses at the tooth-restoration interface while preserving the integrity of the interface (Ausiello, Apicella, and Davidson, 2002).

Dry *versus* Wet Bonding

The basic mechanism of bonding to enamel and dentine is essentially an exchange process. Minerals removed from the hard dental tissue are replaced by resin monomers that upon *in situ* setting provide micro-mechanical interlocking in the created porosities (Van Meerbeek *et al*, 2001).

Enamel and dentine should be properly treated to allow for full penetration of the adhesive monomers. A dry condition is theoretically preferred on the enamel surface whereas on the dentine site; a certain amount of water is desirable to avoid collapse of the exposed dentine collagen scaffold (Perdigão *et al*, 1995).

Dry and wet bonding techniques have been demonstrated to achieve adequate hybridization depending upon the type of adhesive and the kind of solvent of the primer. Dry bonding procedures have been recommended for adhesive systems with water based primers to re-hydrate and re-expand the dried collapsed collagen network, allowing efficient inter-diffusion of the resin monomers. The other way is to keep the acid-etched dentine surface moist and to rely on the hydrophilic properties of the acetone-based primers. This technique is known as wet-bonding (Van Meerbeek *et al*, 2001).

Vargas and Swift (1994) reported no difference in microleakage between the dry and moist bonding techniques to dentine while Tay *et al*, (1995a) reported severe microleakage along the resin-dentine interface when water was not completely removed from the primer solvent.

Light Polymerization Units and Concepts

In recent years, several new polymerization techniques and curing units have been introduced in an attempt to affect polymerization shrinkage. Conventional quartz halogen curing lights with higher intensities, plasma arc curing lights, blue light emitting diode curing lights, and argon lasers are used for polymerization of direct resin-based restorative materials (Manhart, Garcia-Godoy and Hickel, 2002).

There are three main techniques with reference to photocuring:

Soft start polymerization: this utilizes multimode quartz halogen curing lights. It is characterized by an initial low intensity light followed by a high intensity light for the final cure, ideally after passing the gel point. The system increases the gel phase resulting in flow and better adaptation of the composites to the cavity walls. This minimizes the internal stresses and reduces the marginal gap formation (Kubo *et al*, 2004).

The pulse delay cure technique: this utilizes an initial low intensity cure for a short duration to provide a sufficient network formation on the top surface while the gel point in the deeper resin is delayed until the final high intensity is started (Deliperi, Bardwell, and Papathanasiou, 2003). Pulse activation has been advocated to reduce the contraction stresses at the cavosurface margins (Kanca and Suh, 1999).

High intensity and fast polymerization: this concept was introduced to decrease exposure time and to have a greater depth of cure compared to the conventional lights (Deliperi, Bardwell, and Papathanasiou, 2003).

Argon lasers are also used to polymerize composite resins. Lasers offer greater depth and degree of polymerization but reports about their

disadvantages including increased shrinkage, brittleness and marginal leakage caused by increased polymerization, negates their use (Manhart, Garcia-Godoy and Hickel, 2002).

In recent years, blue light emitting diode (LED) technology has been proposed for polymerizing resin-based composite restorative materials (Jandt *et al*, 2000). LED light curing units do not require filters because the spectral output falls conveniently within the absorption spectrum of camphoroquinone photoinitiators *i.e*. between 400 and 500 nm (Manhart, Garcia-Godoy and Hickel, 2002). Oberholzer, Du Preez, and Kidd, (2005) also demonstrated that the use of LED light for polymerization in the soft start mode resulted in a reduced polymerization shrinkage and microleakage and also LEDs have an expected lifetime of several thousand hours without significant degradation of the light flux over time.

Versluis, Tantbirojn, and Douglas (1998) reported that composite resins do not shrink towards the light, but that the direction of the shrinkage is predominantly determined by the shape of the cavity and the quality of the bond.

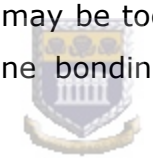
Cavity Configuration Factor

When a resin composite restoration is cured, it bonds to the walls and the floor of the cavity preparation. During polymerization the restorative resin shrinks and pulls the opposing walls and floor of the cavity closer together. The magnitude of this phenomenon depends upon the configuration of the cavity and, hence, is called the cavity configuration factor or C-factor (Choi *et al*, 2004). The configuration factor has been defined as the ratio of the bonded surface area to the free surface area of the cavity (Franco *et al*, 2003).

Higher C-factors have been reported to produce higher contraction stresses by limiting the flow capacity of the resin composites (Franco *et al*, 2003). Class I and deep Class V cavities have high C-factor values with possible high contraction stresses (Nikolaenko *et al*, 2004). Hashimoto *et al*, (2000a) reported relatively rapid *in vivo* degradation of bonds in high C-factor Class I restorations.

Santini *et al*, (2004) demonstrated that cavity configuration did not affect microleakage around a hybrid composite restoration when using a self etching or a total-etch adhesive system. However the configuration factor did affect the mechanical properties of the composite restorative material and the strength of the bond at the interface between the composite and the tooth structure (Choi *et al*, 2004).

However the presence of a high C-factor is a risk for bonding because the polymerization stresses may be too great to be counteracted by the bond strength of the dentine bonding agent (Shirai *et al*, 2005 and Nikolaenko *et al*, 2004).



Thermal Cycling and Occlusal Stresses

In vitro microleakage studies using thermocycling provide results that correlate laboratory findings more accurately with clinical performance (Mathew, Paramaswaran Nair, and Krishnan, 2001).

Thermal stresses can be pathologic in two ways. Firstly, differential thermal changes induce mechanical stresses that can cause crack propagation through the bonded interface. Secondly, gap volume changes associated with changing gap dimensions pump pathogenic oral fluids in and out of the gaps with possible pulpal complications (Gale and Darvell, 1999).

Bedran-de-Castro *et al*, (2004) found no statistically significant differences as regards to the influence of thermocycling, mechanical load cycling or the combination on microleakage and shear bond strength of composite resin restorations. Jang *et al*, (2001) reported that load cycling significantly increased microleakage at the gingival margins of Class V flowable and packable composite resin restorations.

Gale and Darvell, (1999) also reported that thermal stressing of the restoration interface is only of value when the initial bond is already known to be reliable. Most of the restorative materials available today do not fall into this category.

2.2.3- *In Vitro* Microleakage Studies

Microleakage studies have been carried out both *in vivo* and *in vitro*, but *in vitro* studies evaluating the integrity and durability of the marginal seal of different resin composites at cavity margins are more common (Raskin *et al*, 2003, Taylor and Lynch, 1992).

Flowable resin composites (Yazici, Celik and Ozgunaltay, 2004), hybrid composites (Aranha and Pimenta, 2004; Yazici, Celik and Ozgunaltay, 2004), and packable resin composites (Loguercio *et al,* 2004; Aranha and Pimenta, 2004; Yazici, Celik and Ozgunaltay, 2004) were able to prevent microleakage at cavity walls with enamel margins but they showed some degree of microleakage at dentine or cementum margins. However, the ormocers (organically modified-ceramics) have demonstrated a better sealing ability at the dentine margins of the tooth–restorative interface (Yazici, Celik and Ozgunaltay, 2004 and Rosin *et al*, 2002)

The use of flowable composite as a liner in class II restorations appeared to reduce, but did not eliminate, the microleakage at the

gingival margins apical to the cemento-enamel junction (Deliperi, Bardwell, and Papathanasiou, 2003; Ernst *et al*, 2002; Leevailoj *et al*, 2001).

Franco *et al*, (2003) reported that the adhesive system primarily influenced the marginal sealing of resin restorations rather than other factors that could also contribute to the formation of marginal gaps.

Resin-modified glass-ionomer cements used in the open sandwich technique seemed to improve the sealing ability of proximal class II restorations in comparison to modern adhesive systems (Besnault and Attal, 2003).

The use of an incremental placement technique has been reported to reduce microleakage associated with Class II resin-based composite restorations (Poskus, Placido, and Cardoso, 2004a). Visible light-cured (VLC) composite resins and autocure composite resins showed extensive microleakage when the margins were on dentine or cementum irrespective of the polymerization technique used (Hilton, Schwartz and Ferracane, 1997).

2.2.4–Conclusion

From the literature review it appears that despite the use of different restorative materials, polymerization techniques, lining cements, and cavity designs, microleakage is still a problem in resin-based composite restorations especially if the margins are in dentine or cementum.

2.3- SURFACE MICROHARDNESS

Microhardness is defined as the resistance to permanent deformation only caused by indentation after load (Poskus, Placido and Cardoso, 2004b).

Microhardness of a composite resin has often been used for the estimation of depth of cure (Tantbirojn *et al*, 2003). Incremental and bulk placement techniques (Tagtekin *et al*, 2004), effectiveness of different light curing devices; LEDs, Quartz-halogen lamps, Plasma arc, (Tagtekin *et al*, 2004; Coffey *et al*, 2004; Oberholzer, Du Preez, and Kidd 2005) and aging (Yap *et al*, 2004) affect microhardness of resin composite restorative materials.

Oberholzer, Du Preez, and Kidd (2005) demonstrated that polymerization of a resin composite with a halogen light curing unit resulted in lower microhardness values as compared to that obtained when a LED curing unit was used. The top surfaces of resin composite materials show higher microhardness values compared to the bottom surfaces (Coffey *et al*, 2004). When using conventional light curing devices a minimum exposure time is required to achieve optimal microhardness, beyond which prolonged curing does not have a proportionate hardening effect (Tagtekin *et al*, 2004).

Resin composites are subjected to masticatory forces of varying magnitude and rate of change as well as fluctuating temperatures when used in load-bearing areas of the posterior dentition. The polymer matrix is more susceptible to these changes compared to the filler and thus dominates the mechanical behavior of the resin-based composites (Musanje and Darvell, 2004). The restorative materials with higher microhardness values show higher wear resistance (Tagtekin *et al*, 2004). Da Fonte Porto Carreiro, Dos Santos Cruz and Vergani (2004)

reported that aging in water reduced the microhardness of resin composite materials. Say *et al*, (2003) also reported a significant negative correlation between hardness and three body wear of resin composites.

Tantbirojn *et al*, (2003) reported that under similar conditions such as equal storage time periods and within the same composite system microhardness of a composite resin could be used for the determination of the fracture resistance of that material.

2.4 – COMPOSITE RESTORATIVE MATERIALS

(RECENT DEVELOPMENTS)

2.4.1 – Introduction

To obtain a good marginal seal to the tooth structure especially to dentine or cementum still seems to be a major challenge in dentistry. For this reason several new materials have been developed with modifications in filler technology, filler distribution, filler loading and alterations in the matrices (Manhart, Garcia-Godoy and Hickel, 2002). In this section, packable composites, ormocers, nanotechnology and nanocomposites will be reviewed briefly.

2.4.2 – Packable Composites

Packable composite resins have attracted increasing attention in recent years. These are highly filled composite resins with a different consistency compared to hybrid resins. Manufacturers' claim that this class of composite resin is an alternative to amalgam restorations in posterior teeth (Manhart, Garcia-Godoy and Hickel, 2002).

These composites are recommended for use in Class I, II, and VI (MOD) restorations. These are composed of light activated, dimethacrylate resins with a higher percentage of irregular (mixture of irregular particles and glass rods) or porous fillers (Fortin and Vargas, 2000). Filler loading in these composite resins varies from 60% to 80% by volume. Resin modifications and interaction of filler particles make these composites packable (Craig and Powers, 2002).

These materials are stiffer and less sticky and can be better packed against the margins without stick-pull back commonly associated with hybrid composite resins. (Loguercio *et al*, 2004). They hold their shape

and do not slump on placement that allows for a more consistent achievement of proximal contacts in Class II restorations (Meiers, Kazemi, and Meier, 2001).

Important properties of these materials include a high depth of cure up to 5mm, low polymerization shrinkage, radiopacity and a low rate of wear of 3.5 μm per year that is almost similar to that of amalgam (Craig and Powers, 2002).

Despite the increased filler loading, the mechanical properties of packable composites do not appear to be superior to those of the universal composites such as the small particle hybrids (Van Noort, 2002 and Cobb *et al*, 2000).

The high viscosity of the packable composites may make close contact and adaptation to the dentine bonding agent and the walls of the cavity preparation more difficult and less consistent (Meiers, Kazemi, and Meier, 2001) This is an important factor to explain microleakage associated with packable composites especially at the margins of the proximal box in Class II restorations, where it is difficult to confirm that the composite resin has adapted to all the line angles (Loguercio *et al*, 2004; Agosta and Estafan, 2003).

2.4.3 – Ormocers (Organically modified Ceramics)

Different strategies have been introduced to overcome the shortcomings of resin composite materials. New monomers have been developed to reduce the polymerization shrinkage and to overcome the physico-mechanical properties of the resin composites (Peutzfeldt, 1997).

The term "ormocer" or organically modified ceramic describes a new type of resin composite that is comprised of organic-inorganic hybrid co-

polymers. These monomers were first introduced for electronic applications (Kournetas *et al*, 2004). They were introduced as dental filling materials in 1998 with improved mechanical properties and low polymerization shrinkage when compared to other resin composite materials.

Instead of bisphenol glycidyl methacrylate (bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacyrlate (TEGDMA), multifunctional urethane- and thioether(meth)acrylate alkoxysilanes as sol-gel precursors have been developed for the synthesis of the inorganic-organic co-polymer ormocer resin composites as dental restorative materials (Yazici, Celik and Ozgunaltay, 2004).

The alkoxysilyle groups of the silane allow the formation of an inorganic Si-O-Si network by hydrolysis and polycondensation reactions, and the methacrylate groups are available for photochemically induced organic polymerization (Kournetas *et al*, 2004).

The novel inorganic-organic co-polymers in their formulation allow the modification of mechanical properties over a wider range as compared to the conventional composites (Manhart, Garcia-Godoy and Hickel, 2002).

2.4.4 – Nanotechnology and Nanocomposites

Today, nanotechnology has become the most popular discipline in science and technology (Mitra, Dong and Holmes, 2003).

Nanotechnology also known as molecular engineering is the production of functional materials and structures in the range of 0.1 to 100 nanometers by various physical and chemical methods (Mitra, Dong and Holmes, 2003).

This is of great interest in the development of dental materials particularly for purpose-designed nano- and microstructures, used to produce composites with low shrinkage, high wear resistance and biocompatibility (Moszner and Klapdohr, 2004).

Nanofillers are very different from traditional fillers. A bottom-up approach is used for manufacturing nanoparticles as compared to a top-down approach used for the production of traditional fillers (Mitra, Dong and Holmes, 2003). Larger particles of mined quartz, melt glasses, ceramics are crushed down to smaller particles to produce fillers of macrofilled, hybrid, and microhybrid composites. However, these milling procedures can only reduce the filler particle size up to 100 nm (1 nm = 1/1000 μm), while nanofiller production involves different physical and chemical processes to produce building blocks at a molecular level. These materials are then assembled into progressively larger structures and transformed into nanosized fillers suitable for a dental nanocomposite (Mitra, Dong and Holmes, 2003).

The modulus of elasticity can be increased and the optical properties of dental composites can be improved by nanofillers (Moszner and Klapdohr, 2004). There is greater scattering of light with the small sized nanoparticles as compared to a larger-particle composite. More scattering of light produces excellent blending of the restoration (the "chameleon effect") and gives it a life-like appearance (Ho, 2004).

2.4.5 – Conclusion

The manufacturers are developing new dental restorative materials with promising properties but they lack scientific support from independent *in vitro* and *in vivo* research. There is a need for more independent research to provide a better understanding of these materials and the limitations of their use. Therefore the aim of this *in vitro* study is to

investigate microleakage and surface microhardness of nanotechnology based new dental resin composite restorative materials.

2.5- *IN VITRO* MICROLEAKAGE EVALUATION METHODS

2.5.1 – Introduction

Microleakage has been regarded as one of the main reasons for the failure of restorations (Raskin *et al*, 2003). Numerous *in vitro* studies have been performed in the past to test the sealing ability of restorative materials at cavity margins. To date, no direct correlation has been established between the results of *in vitro* tests and clinical findings regarding microleakage (Déjou, Sindres and Camps, 1996; Raskin *et al*, 2003).

In vitro tests should be regarded as setting a theoretical maximum amount of leakage that may or may not occur *in vivo* (Pashley, 1990). *In vitro* studies help in the selection of restorative materials and techniques and are essential for research and developmental purposes (Déjou, Sindres and Camps, 1996, Raskin *et al*, 2003).

A large variety of methods have been used to evaluate the microleakage of restorative materials. These microleakage tests include color producing micro-organisms, radioactive isotopes including ^{45}Ca, ^{131}I, ^{35}S, ^{22}Na, air pressure method, neutron activation analysis, electrochemical studies, scanning electron microscopy, thermal and mechanical cycling, chemical tracers and dye penetration studies (Taylor and Lynch, 1992).

2.5.2 – Dye Penetration Studies

A dye penetration measurement on sections through restored teeth is one of the most common techniques used for microleakage evaluation because it is simple and fast (Déjou, Sindres and Camps, 1996). This method allows the production of sections showing leakage in contrasting colors to both tooth and restoration without the need for further chemical reaction or exposure to potentially hazardous radiation (Taylor and Lynch, 1992).

Different techniques using different dye solutions have been reported on in the literature (Taylor and Lynch, 1992). Tsuchiya, Zidan and Gomez-Martin, (1986) described a technique that involved examination of the restoration margin under magnification following exposure to a dye substance. The proportion of the margin that exhibited leakage was measured. The disadvantage of this technique is that it did not give an idea about the behavior of the material in the section of the interface below the restoration margin, where large unrecordable gaps may have existed (Taylor and Lynch, 1992).

Douglas and Zakariasen (1981) described a dye-recovery technique. Following leakage the dye was collected from the specimen to give a quantitative value for the amount of dye taken into the marginal gap. However it is not clear if complete recovery of the dye particularly that taken up by the dentinal tubules was possible.

Spangberg, Acierno and Cha, (1989), Goldman, Simmonds and Rush, (1989) have shown that if specimens were placed in a vacuum before immersion in the dye solution, it would result in the removal of any entrapped air from within the system. This would significantly increase the dye penetration along the marginal defects.

Dyes that can bind to tooth substance or to the restorative materials are a potential source of error in leakage studies because penetration studies in dentine also exhibit some dentine staining that should be distinguished from the actual gap between the cavity wall and the restorative material (Taylor and Lynch, 1992).

Commonly used dyes such as basic fuchsin solutions particularly those in propyl glycol co-solvent preferentially bind with carious dentine (Kidd, Joyston-Bechal and Smith, 1989). This propensity has been made use of in the manufacturing of the caries-disclosing agents. This makes the effectiveness of such dyes questionable particularly when demonstrating an inert space between tooth substance and restorative material as an area of stained dentine in cross-section that can be mistaken for a larger gap than actually exists (Taylor and Lynch, 1992).

Dyes used in dental research are provided as either solutions or particle suspensions of differing particle size dependent upon manufacturer and individual behavior of the dye. The literature reveals that the choice of dyes used continues to be based on an apparent *ad hoc* basis with little attention given to the different size of dye molecules/particles and their behavior (Taylor and Lynch, 1992).

2.5.3 – Microleakage and Number of Tooth Sections

In vitro microleakage detection around dental restorations has been extensively reviewed in the literature (Alani and Toh, 1997; Taylor and Lynch, 1992; Kidd, 1976b; Going, 1972). The most commonly applied method is the use of dyes and a single midline section through the restoration in the tooth. Microleakage is assessed on an ordinal score and is expressed as linear leakage length, or a percentage of leakage length related to the total length of the measured surface line (Federlin *et al*, 2002).

Most of the studies investigating microleakage are based on only one section of the restored tooth. Raskin *et al*, (2003) in a literature review on microleakage of 144 published articles showed that 47% of the researchers used only one section, 20% used two sections, and 12% used three sections. It is difficult to assess a restoration as a whole or even a single surface on the basis of only one section of the tooth (Federlin *et al*, 2002). The observed section may not be representative of the total leakage distribution (Youngson *et al*, 1998) since dye penetration may vary from one zone to another of the same restoration-tooth interface (Tay *et al*, 1995b; Hilton, Schwartz and Ferracane, 1997). Gale, Darvell and Cheung (1994) reported that microleakage is a three-dimensional phenomenon and different locations and angles of sectioning might result in completely different dye penetration scores for that interface.

Various methods have been described to overcome these shortcomings over the past decade. Multiple-surface scoring methods have been regarded superior to single-surface scoring methods because the results obtained seem to be more representative of the total microleakage (Mixon *et al*, 1991).

Raskin *et al*, (2003) recommended that at least three sections should be used to avoid an underestimation of the microleakage observed. Based on these findings this study will utilize three sections of each restoration to evaluate microleakage so that the results are more representative of the actual leakage.

2.5.4 – Conclusion

Diverse *in vitro* microleakage methods have been used to demonstrate microleakage between tooth and restorative material. Each method has its own advantages and disadvantages (Raskin *et al*, 2003).

Three-dimensional techniques may reveal more clinically-relevant patterns than observed in conventional sectioning techniques that only give a two-dimensional picture. However, these are more time consuming, difficult to quantify and expensive (Hilton, Schwartz and Ferracane, 1997).

Dye penetration measurements on sections of restored teeth remains the most common method to determine microleakage due to its simplicity and cost effectiveness (Yap, Ho and Wong, 1998; Déjou, Sindres and Camps, 1996).

2.6 – REVIEW OF MATERIALS TESTED

The materials used in the study have been discussed briefly. The scientific information of each material given below is taken from product leaflets or the manufacturer's website. The materials are also summarized in Table 2.1.

2.6.1 –: CERAM•X™

Ceram•X is a light cured, radiopaque ormocer-based nano-ceramic material by Dentsply, DeTrey, Germany. It is recommended for anterior and posterior restorations of primary and permanent teeth. According to the manufacturer the use of nano-ceramic technology offers superior aesthetics and handling properties.

Ceram•X is available in two shade systems.

Ceram•X mono: the Single Translucency System comprises seven shades of intermediate translucency comparable to conventional composites.

Ceram•X duo: the Double Translucency System comprises four dentine shades with translucencies similar to natural dentine and three enamel shades.

Composition

Ceram•X was developed from nano-ceramic technology, that is the merger of hybrid filler technology and advanced nano-technology. Ceram•X consists of organically modified ceramic nano-particles of 2.3

nm and nanofillers of 10 nm combined with conventional glass fillers of ~1µm. Filler loading is 76% by weight and 57% by volume (Table 2.1).

Organically modified ceramic nano-particles are achieved via controlled hydrolysis and condensation reactions and are highly dispersed (Fig.2.1) (Dentsply, 2003).

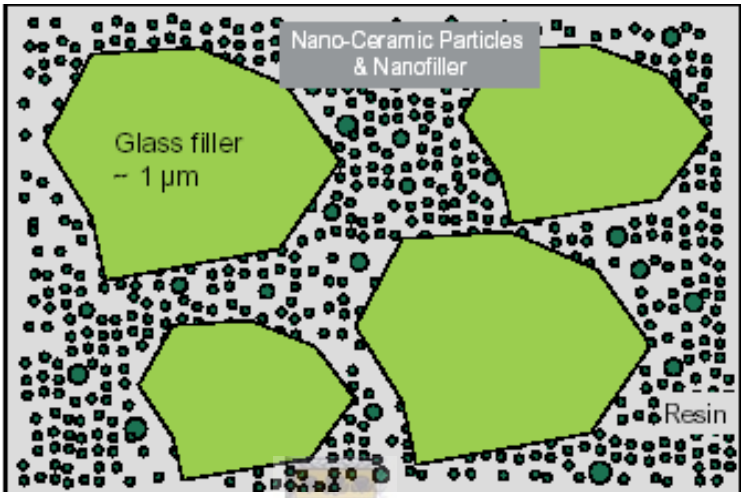


Fig.2.1: Composition of Ceram•X
(Ceram•X Scientific Compendium, Dentsply)

Nano-particles comprise of a polysiloxane backbone. Methacrylic groups are attached to this backbone via silicon-carbon-bonds. Nano-particles may be best described as inorganic-organic hybrid particles. The inorganic siloxane part provides strength while the organic methacrylic part makes the particles compatible and polymerizable with the resin matrix (Fig. 2.2).

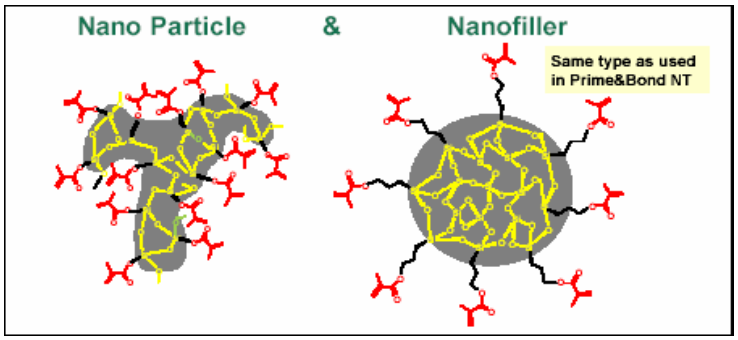


Fig.2.2: Nano-particle and nanofiller
(Ceram•X Scientific Compendium, Dentsply)

The resin matrix used consists of a methacrylate modified polysiloxane and a dimethacrylate resin.

Advantages of Ceram X (according to the manufacturer; Dentsply, Germany)

- High biocompatibility
- High fracture toughness
- Low polymerization shrinkage
- Excellent working time under ambient light
- Superior handling characteristics

Interaction with Other Materials

Eugenol and hydrogen peroxide containing dental materials may prevent the complete setting of Ceram X and cause its softening.

2.6.2 – PREMISE™

Premise is a universal light cured resin-based nanofilled dental composite restorative material developed by Kerr Hawe, USA. It is designed for direct placement and can be used for both anterior and posterior restorations. The trimodal filler system of Premise provides high polishability and low polymerization shrinkage of the set material.

Premise is available in a variety of shades.

Enamel and Dentine Shades: the enamel and dentine shades of Premise are based on the VITA shading system and are developed to match the opacity of enamel and dentine.

Translucent Shades: these are placed very thinly as a final layer to give a lifelike appearance to the restoration. Each translucent shade has a specific contrast ratio and slight color.

Extra Light Shades: these are lighter than the lightest VITA shade. The extra light shades are useful to match bleached and deciduous teeth.

Packable Shades: three packable shades are available for posterior restorations. The overall filler loading and viscosity of the packable shades is higher than that of other Premise shades.

Composition

Premise is a highly filled nanocomposite restorative material. Filler loading is 84% by weight and 69% by volume (Table 2.1). In Premise a *trimodal filler* approach is used that consists of three types of fillers (Fig.2.3):

- Prepolymerized filler (PPF), 30 to 50 µm
- Non-agglomerated "discrete" silica nanoparticles, 0.02 µm
- Barium glass filler 0.4 µm

Prepolymerized filler is a blend of a low shrinkage resin, barium glass and discrete nano-filler particles. It helps to achieve a high filler loading and reduces the stickiness of the material.

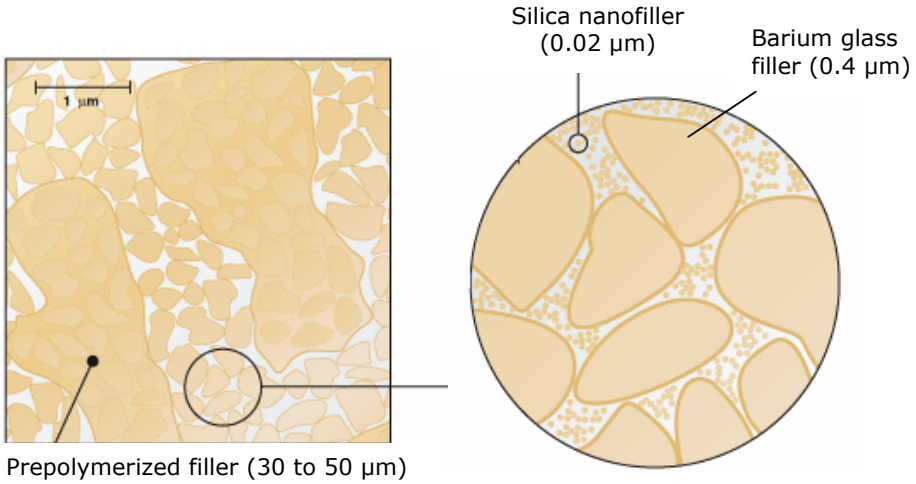


Fig.2.3: Composition of Premise (KerrPremise.com)

The resin consists of an ethoxylated bis-phenol-A-dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), light-cure initiators and stabilizers.

Advantages of Premise (according to the manufacturer; Kerr, USA)

- Ultra low shrinkage 1.6%
- Superior polishability that can be sustained over time
- Optimal handling
- Reliable strength

2.6.3 – GRANDIO™

Grandio is a radiopaque, light-cured nanohybrid restorative material combining the proven composite with innovative nanotechnology developed by Voco, Germany. High filler loading provides excellent surface hardness, wear and fracture resistance. According to the manufacturer Grandio can be used for:

- restoring Class I, II, III, IV and V cavities
- reconstruction of traumatically affected anteriors
- repair of veneers
- filling of deciduous teeth
- core-build-up under crowns
- composite inlays

Grandio is available in a variety of shades with a Grandio™ shade guide.

Composition

Grandio is a highly filled, nanohybrid composite. The inorganic filler loading is 87% by weight and 71.4% by volume (Table 2.1) that is

achieved by combining spherical nanoparticles (silicium dioxide, 20-50 nm) with traditional glass ceramic particles of fine-tuned size (Fig. 2.4). The traditional fillers form a hard network and the nanoparticles fill the spaces between them. This results in maximum packing density and leaves a nominal resin content of 13%, this is 30-50% less resin than that of most microhybrids.

Fig. 2.4: Nanofiller and unfilled resin for Grandio (VOCO, Germany)

The resin matrix consists of BIS-GMA and triethylene glycol dimethacrylate (TEGDMA).

Advantages of Grandio (according to the manufacturer; Voco, Germany)

- Low polymerization shrinkage 1.57%
- High fracture resistance
- Increased surface hardness
- Increased abrasion resistance
- Ease of handling

2.6.4 – Z100™

Z100 is a visible-light activated, radiopaque, microhybrid composite developed by 3M ESPE, USA. It can be used for both anterior and posterior restorations.

Z100 is available in a variety of shades.

Composition

Z100 is a microhybrid composite with an inorganic filler loading of 72% by weight and 66% by volume. The filler used in Z100 is zirconia/silica with a particle size range of 3.5 to 0.01 micron (Table 2.1).

The resin matrix contains BIS-GMA and triethylene glycol dimethacrylate (TEGDMA).

Z100 (3M ESPE, USA) will be used as the control in this study.

Table 2.1: Restorative materials used in the study

Product (manufact--urer)	**Monomer** (matrix)	**Filler (%)**		**Filler**		**Shrinkage (%)**
		Weight	Volume	Composition	Size (μm)	
Ceram X Dentsply Germany	Methacrylate modified polysiloxane, Dimethacrylate resin	76	57	Glass (Si-Ba-Al-Bo) Methacrylate SiO_2 (nanofiller) SiO_2 nano-particle	1.1–1.5 0.1 0.023	2.3
Premise Kerr,USA	Ethoxylated bis-phenol-A-dimethacrylate & TEGDMA	84	69	PPF Barium glass Silica	30-50 0.4 0.02	1.6
Grandio Voco, Germany	Dimethacrylate Triethylene glycol dimethacrylate	87	71.4	Conventional Glass particles Silicium dioxide	Fine-tuned size 0.02-0.05	1.57
Z100 3M ESPE USA	Dimethacrylate Triethylene glycol dimethacrylate	72	66	Zirconia/ silica	3.5-0.01	2.8

BIS-GMA = dimethacrylate, TEGDMA= Triethylene glycol dimethacrylate, Si-Ba-Al-Bo = Barium-aluminium-borosilicate, SiO_2 = silicon dioxide, PPF = prepolymerized filler

2.7 – CONCLUSION

In recent years the demand for aesthetic restorations has dramatically increased (Herrero, Yaman and Dennison, 2005). Tooth colored restorative materials are being used increasingly not only to replace failed or unaesthetic amalgam restorations but as the first choice material to restore new carious lesions in the aesthetic zone (Fleming *et al*, 2005). Despite the remarkable improvements in the resin composite materials and the adhesive systems, clinical failures are still being reported particularly when restorations are placed in stress bearing areas (Kournetas *et al*, 2004).

The most common clinical problems associated with posterior composite restorations are poor marginal adaptation along the cervical margin, secondary caries and material fracture (Hickel and Manhart, 2001). The open margins make the teeth more susceptible to hypersensitivity, discoloration, secondary caries, pulpal pathoses and overall failure of the restoration (Cenci, Demarco, and Carvalho, 2005, Franco *et al*, 2003). Polymerization shrinkage has been regarded as a major factor directly related to marginal leakage and gap formation at the tooth-restoration interface when using resin-based composite restorative materials (Cenci, Demarco, and Carvalho, 2005).

Microleakage is not a significant problem in restorations that have all their margins in enamel, as enamel is a reliable substrate for bonding. However, it is difficult to achieve a complete and long lasting seal if the restoration margins are on dentine or cementum (Yazici, Celik and Ozgunaltay, 2004).

Several new materials such as microhybrids, packable composites and ormocers with different formulations have been developed to overcome the short-comings of resin-based composite restorative materials.

Recently, nanotechnology has gained much interest to produce nanoparticles that are used as nanofillers for the development of nanocomposites. These materials are easy to handle and have excellent physical and aesthetic properties.

Although new materials are subjected to extensive testing by the manufacturers, there is always a need for independent *in vitro* and *in vivo* research. The information obtained will also be useful for comparative assessment of the different materials and for drawing up clinical guidelines for the usage of these materials in the clinics.

CHAPTER 3

AIMS AND OBJECTIVES

3.1 – AIM OF THE STUDY

The aim of this study was to investigate if there was a significant difference in microleakage and surface microhardness between a nanofilled, a nanohybrid and an ormocer based nano-ceramic composite restorative material.

3.2 – OBJECTIVES OF THE STUDY

1. To determine the qualitative microleakage of Class II preparations restored with the above mentioned nanocomposite restorative materials.
2. To determine the surface microhardness of the top and bottom surfaces of each restorative material.

3.3 – NULL HYPOTHESIS

There is no significant difference in microleakage and surface microhardness between a nanofilled, a nanohybrid and an ormocer based nano-ceramic composite restorative material.

CHAPTER 4

MATERIALS & METHODS

4.1- SPECIMEN PREPARATION FOR MICROLEAKAGE

4.1.1- Sample Size and Teeth Selection

Eighty extracted human molar teeth free of visible caries, cracks, and restorations were used in this study. The teeth were cleaned, disinfected in 0.5% chloramine T, and subsequently stored in 0.9% saline solution (Loguercio *et al*, 2004).

4.1.2 – Cavity Preparation

Class II slot cavities were prepared on both the mesial and distal surfaces of each tooth using a FG 110 012 fissure bur (Horico, Germany) in a high speed hand-piece with copious water irrigation. The dimensions of the preparation were 3 mm in width, 4.5-5.5 mm in height, and 2 mm in depth, without any bevels on the occlusal margins (Besnault and Attal, 2003). Half of the sample had the gingival margin located 1 mm above the cementoenamel junction (CEJ), whereas the other half had the gingival margin placed 1 mm below the CEJ (Loguercio *et al*, 2004). Burs were replaced after every eight preparations (Hilton, Schwartz and Ferracane, 1997).

4.1.3 – Restoration Template

A "*restoration template*" was fabricated to simulate the clinical situation during the placement of the restorations (Figure 4.1). Two molar teeth,

approximately 12-14 mm apart, were embedded in dental stone to the level of the cementoenamel junction (CEJ). A test specimen embedded in poly(vinyl siloxane) impression material was placed in the space between the two teeth (Tagtekin *et al*, 2004).

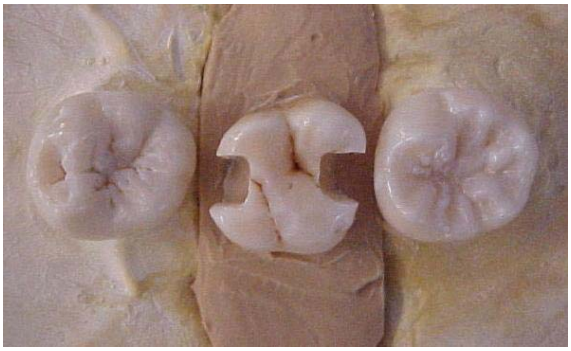


Fig 4.1: Restoration Template

4.1.4 – The Experimental Groups

In order to compare the microleakage of the different restorative materials, the experimental groups were divided as follows:

Table 4.1: Experimental Groups	
Group 1	Ceram X mono (nano-ceramic) + Prime & Bond NT
Group 2	Premise (nanofilled) + OptiBond SoloPlus
Group 3	Grandio (nanohybrid) + Admira Bond
Group 4	Z100 (microhybrid) + Adper Scotchbond

4.1.5 – Restoration Placement Procedure

Teeth were randomly divided into four groups with twenty teeth in each group (Table 4.1). Random division provided each tooth an equal chance to fall in any group. Care was taken that each group had an

equal number of cavities with a gingival margin on enamel and on dentine.

Thus each group had twenty cavities with a gingival margin above the CEJ and twenty cavities with a gingival margin below the CEJ. Each tooth was placed in the restoration template. Metal matrix bands and wooden wedges were used for the placement of each restoration. The restoration of cavities is summarized in Table 4.2. Each restorative material was used with its corresponding adhesive system according to the manufacturer's instructions Table 4.3.

Table 4.2: Division of groups and restoration placement procedure

Groups	No. of Teeth	No. of Cavities with margins on		Bonding Agent	Restorative Material	Technique	Curing Time
		Enamel	Dentine or cementum				
G 1	20	20	20	Prime & Bond NT	Ceram X	Incremental + Metal matrix band	40 Sec per increment
G 2	20	20	20	Optibond Solo Plus	Premise	Incremental + Metal matrix band	40 Sec per increment
G 3	20	20	20	Admira Bond	Grandio	Incremental + Metal matrix band	40 Sec per increment
G 4	20	20	20	Scotchbond	Z100	Incremental + Metal matrix band	40 Sec per increment

The application technique of the adhesive systems and the materials used were as follows:

Group 1: Prime & Bond® NT and Ceram•X™ mono

Acid Conditioning (total etch technique)

After each cavity was cleaned and blot dried, DeTrey® Conditioner 36 (36% phosphoric acid) was applied to the cavity surfaces starting at the

enamel margins. The enamel was conditioned for at least 15 seconds and dentine for 15 seconds or less. The gel was removed by rinsing the cavity with water for 15 seconds. Water was removed from the rinsed cavity with a soft blow of air and blot dried, leaving a moist surface.

Application of Prime & Bond NT and curing

Prime & Bond NT (Figure 4.2), (Lot no. 040804, Expiry date 2006-08) was dispensed directly onto a fresh applicator tip and immediately ample amounts of Prime & Bond NT were applied twice to thoroughly wet all the cavity surfaces for 20 seconds. The cavities were gently air dried for 5 seconds and light cured for 10 seconds with a halogen light curing unit (Demetron LC, sdsKerr, USA).

Application of Ceram•X™

Ceram X mono (Figure 4.3), (Lot no. 0505001618, Expiry date 2006-11) was placed immediately after the application and curing of the Prime & Bond NT. The Ceram X was dispensed directly into the prepared cavity in 2 mm increments. All cavities were filled in three increments. Each increment was cured individually with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 40 seconds. The curing light was held 5 mm away from the filling.

Group 2: OptiBond® Solo Plus™ and Premise

Acid Conditioning (total etch technique)

After each cavity was cleaned and blot dried, etching gel (37.5% phosphoric acid, sdsKerr USA), (Lot no. 5-1003, Expiry date 2008-1) was applied to the cavity surfaces starting at the enamel margins. The enamel was conditioned for at least 15 seconds and dentine for 15 seconds or less. The gel was removed by rinsing the cavity with water for 15 seconds. Water was removed from the rinsed cavity with a soft blow of air and blot dried, leaving a moist surface.

Figure 4.2. Prime & Bond NT and Detrey Conditioner

Figure 4.3. Ceram X

Figure 4.4. Optibond Solo Plus and Kerr etchant

Figure 4.5. Premise

Figure 4.6. Admira Bond and

Figure 4.7. Grandio

Figure 4.8. Adper Scotchbond Primer, Adhesive and 3M etching gel

Figure 4.9. Z100

Application of OptiBond® Solo Plus™ and curing

OptiBond® Solo Plus™ (Figure 4.4), (Lot no. 4-1349, Expiry date 2006-12) was dispensed directly onto a fresh applicator tip and immediately applied to thoroughly wet all cavity surfaces using a light brushing motion for 15 seconds. OptiBond® Solo Plus™ was gently air thinned for 3 seconds and light cured for 20 seconds with a halogen light curing unit (Demetron LC, sdsKerr, USA).

Application of Premise

Premise (Figure 4.5), (Lot no. 014341, Expiry date 2007-06) was placed immediately after the application and curing of the OptiBond® Solo Plus™. The Premise was dispensed from the syringe into the prepared cavity by a flat plastic instrument in 2 mm increments. All cavities were filled in three increments. Each increment was cured individually with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 40 seconds. The curing light was held 5 mm away from the filling.

Group 3: Admira® Bond and Grandio®

Acid Conditioning (total etch technique)

After each cavity was cleaned and blot dried, Vococid etching gel (34.5% phosphoric acid, VOCO Germany), (Lot no. 530221, Expiry date 2007-09) was applied to the cavity surfaces starting at the enamel margins. The enamel was conditioned for at least 20 seconds and dentine for 15 seconds or less. The gel was removed by rinsing the cavity with water for 20 seconds. Water was removed from the rinsed cavity with a soft blow of air and blot dried, leaving a moist surface.

Application of Admira® Bond and curing

Admira® Bond (Figure 4.6), (Lot no. 501929, Expiry date 2007-04) was dispensed directly onto a fresh applicator tip and immediately applied to

thoroughly wet all cavity surfaces for 30 seconds. Cavities were gently air blown for 5 seconds and light cured for 20 seconds with a halogen light curing unit (Demetron LC, sdsKerr, USA).

Application of Grandio®

Grandio® (Figure 4.7), (Lot no. 411433, Expiry date 2006-03) was placed immediately after the application and curing of the Admira® Bond. The Grandio® was dispensed directly into the prepared cavity in 2 mm increments. All cavities were filled in three increments. Each increment was cured individually with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 40 seconds. The curing light was held 5 mm away from the filling.

Group 4: Adper™ , Scotchbond™ and Z100

Acid Conditioning (total etch technique)

After each cavity was cleaned and blot dried, 3M Scotchbond etchant (35% phosphoric acid), (Lot no. 6EB) was applied to the cavity surfaces starting at the enamel margins. The enamel was conditioned for at least 15 seconds and dentine for 15 seconds or less. The gel was removed by rinsing the cavity with water for 15 seconds. Water was removed from the rinsed cavity with a soft blow of air and blot dried, leaving a moist surface.

Application of Adper™ , Scotchbond™ and curing

Adper™ , Scotchbond™ primer (Figure 4.8), (Lot no. 4 AR, Expiry date 2007-09) was dispensed directly onto a fresh applicator tip and immediately applied to thoroughly wet all cavity surfaces and gently air dried for 5 seconds. Adper™ , Scotchbond™ adhesive (Figure 4.8) (Lot no. 5 PC, Expiry date 2008-05) was then dispensed directly onto a fresh applicator tip and immediately applied to thoroughly wet all the cavity

surfaces and light cured for 10 seconds with a halogen light curing unit (Demetron LC, sdsKerr, USA).

Application of Z100

Z100 (Figure 4.9), (Lot no. 20030626, Expiry date 2006-04) was placed immediately after the application and curing of the Adper™, Scotchbond™ adhesive. Z100 was dispensed directly into the prepared cavity in 2 mm increments. All cavities were filled in three increments. Each increment was cured individually with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 40 seconds. The curing light was held 5 mm away from the filling.

A similar shade was used for all materials as described in Table 2.1. The intensity of the light curing unit was measured using an intensity meter before and during the procedure to standardize theses variables as far as possible.

4.1.6 – Finishing & Polishing

All the teeth were stored in distilled water at 37 °C for 7 days. All the restorations were then finished and polished with aluminum oxide-coated flexible discs Sof-Lex, 3M Dental, USA according to the manufacturer's instructions (Loguercio *et al*, 2004).

Table 4.3: Adhesive systems, compositions, and manufacturers' instructions		
Product/manufacturer	**Composition**	**Manufacturer instructions**
Prime & Bond® NT Dentsply, Germany	1- Phosphoric acid: 36% 2- Adhesive: - Di- & trimethacrylate resins - silica - PENTA - Photoinitiators - Stabilisers - Cetylamine hydrofluoride - Acetone	1- Acid etching for 15 seconds 2-Rinse for 15 seconds 3- Blot dry (wet bond technique) 4- application of two adhesive coats (20 seconds) 5- Air dry for 5 seconds 6- Light activation for 10 seconds
OptiBond® Solo Plus™ Kerr, USA	1- Phosphoric acid: 37.5% 2- Adhesive: -Ethanol, -BIS-GMA, GPDM, -HEMA, -silica, barium glass, -sodium hexafluorosilicate	1- Acid etching for 15 seconds 2- Rinse for 15 seconds 3- Dry for 5 seconds 4- Adhesive application 15 seconds 5- Gently air dry 3 seconds 6- Light activation 20 seconds
Admira® Bond Voco, Germany	1- Orthophosphoric acid: 34.5% 2- Adhesive: -Ormocers -BIS-GMA -HEMA -BHT -Acetone -Organic acids	1- Acid etching of enamel for 20 seconds and dentine 15 seconds 2- Rinse for 20 seconds 3- Dry for 5 seconds 4- Adhesive application 30 seconds 5- Gently air dry 5 seconds 6- Light activation 20 seconds
Adper™ , Scotchbond™ 3M ESPE, USA	1- phosphoric acid: 35% 2- Adhesive: -HEMA -BIS-GMA -Photoinitiators -Stabilizers	1- Acid etching for 15 seconds 2- Rinse for 15 seconds 3- Dry for 5 seconds 4- Primer application 5- Dry gently for 5 seconds 6- Adhesive application 7- Light cure 10 seconds
HEMA = Hydroxyethyl methacrylate; BIS-GMA = Dimethacrylate; PENTA = Phosphonate penta-acrylate ester; GPDM = Glycerol Phosphate Dimethacrylate		

4.2- SPECIMEN PREPARATION FOR SURFACE MICROHARDNESS

Sixty four disk shaped specimens 5 mm in diameter and 2 mm thick of all the restorative materials (Ceram X, Premise, Grandio, and Z100) were prepared using a clear resin mold (Figure 4.10) that was prepared from a clear resin block. The resin block of 2mm thickness was cut by a diamond disk cutter (Struers, Minitom Germany). A 5 mm diameter hole was created in the strip using an acrylic bur. A separating media was applied to the mold before the preparation of each specimen.

Transparent cellulose strips were placed on the top and bottom surfaces of the mold before light curing to remove the air-inhibited layer.

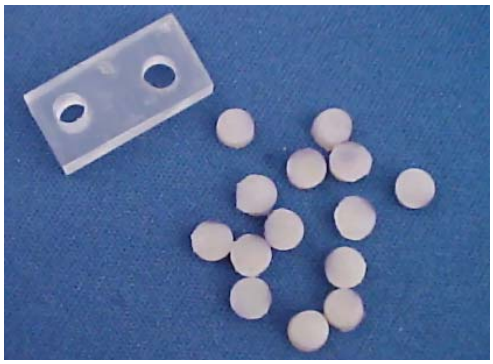


Figure 4.10: Resin mold and specimens for microhardness

Sixteen specimens were prepared for each restorative material. The specimens were divided into four groups (Table 4.4) according to the composite types; nano-ceramic, nano-filled, nanohybrid and microhybrid. The restorative materials were placed in bulk as the mold was only 2 mm thick. All the specimens were cured for 40 seconds using a conventional halogen light curing unit (Demetron LC, sdsKerr USA). The intensity of the light was measured before the start of curing and after every 16 specimen that was cured.

The outer surfaces of all the specimens were polished with a graded series of flexible disks (OptiDisc, KerrHawe SA) according to the manufacturer's instructions. The specimens were stored in 100% relative humidity and in a dark medium at 37°C for 24 hours until the first measurements were recorded.

Table 4.4: Specimen preparation for surface microhardness

Restorative Material Groups	Number of Specimens	Thickness	Curing Time	Finish & Polishing
G1 = Ceram X	16	2mm	40 sec	OptiDisc
G2 = Premise	16	2mm	40 sec	OptiDisc
G3 = Grandio	16	2mm	40 sec	OptiDisc
G4 = Z100	16	2mm	40 sec	OptiDisc

4.3 – MICROLEAKAGE TEST

4.3.1 – Varnishing

The apical 2 mm of each tooth was removed with a model trimmer using water as a coolant. A cavity was prepared in the root apices that were sectioned, with a round carbide bur (C1204008, Horico Germany) in a slow speed hand-piece. Each cavity was rinsed with water, dried with air and conditioned with GC dentine conditioner. The cavities were then filled with resin modified glass ionomer cement (GC Fuji II LC, GC Corporation Tokyo, Japan).

In order to prevent dye penetration into the dentinal tubules or the lateral canals, the teeth were coated with two layers of nail varnish (Charlie, Revlon, New York) except for an area approximately 1 mm around the gingival margin of the restorations (Figure 4.11) (Loguercio *et al*, 2004). The nail varnish was allowed to dry for 12 hours before thermocycling the teeth.

Figure 4.11: nail varnish applied

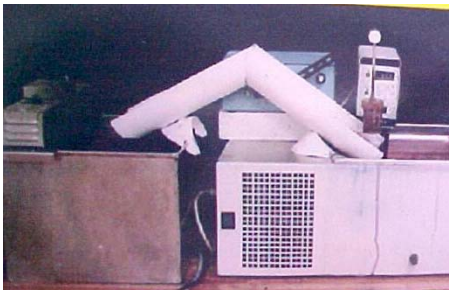


Figure 4.12: cycling unit for thermocycling

Figure 4.13: specimens after thermocycling

4.3.2 – Thermal Cycling

In an attempt to simulate the temperature changes that take place in the oral cavity, the specimens were subjected to thermal cycling (Figure 4.12). All specimens were subjected to thermocycling according to the International Organization for Standardization (ISO) TR11405 standard

of 500 cycles, at 5° to 55 °C, with a 15 second dwell time (Loguercio *et al*, 2004) in a buffered (pH 7) 0.5% methylene blue solution dye.

After removal from the dye (Figure 4.13), the specimens were thoroughly washed under tap water for 10 minutes. The specimens were transferred to specimen bottles containing distilled water until the time of sectioning.

4.3.3 – Sectioning

The nail varnish was removed with acetone solution and all the specimens were again cleaned with water (Figure 4.14). The specimens were embedded in a slow setting epoxy resin and allowed to set overnight. All the teeth were cut buccolingually in the centre and each restoration was then sectioned mesiodistally with a 0.35 mm thick blade in a diamond disk cutter (Struers Minitom, Germany) (Figure 4.15). Three sections per restoration of approximately 0.5 mm thickness provided six surfaces for evaluation (Figure4.16).

Figure 4.14: Specimen after cleansing

Figure 4.15: Struers Minitom

Figure 4.16: Sections for microleakage evaluation

4.3.4 – Microscopy and Scoring

Dye penetration was evaluated at the gingival margin under a stereomicroscope (Wild, Heerbrugg Switzerland) (Figure 4.17) using ten

times magnification by two previously calibrated examiners. Each examiner measured the microleakage of the three sections (six surfaces) of restorations; thus, each section was scored four times and each restoration was scored 12 times by the two examiners.

Any discrepancies between the two examiners were re-evaluated by both until a consensus score was reached.

Figure 4.17: Microscope

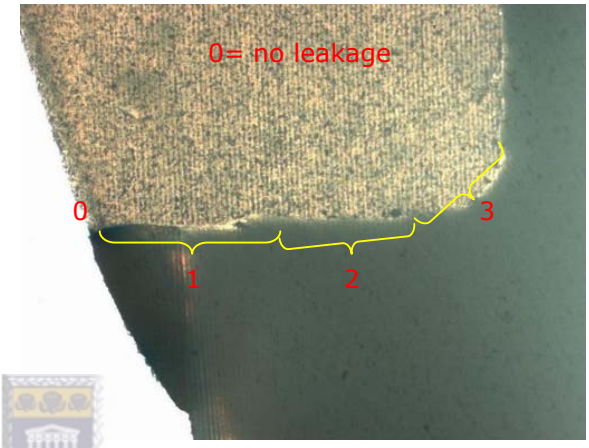


Figure 4.18: Criteria used for microleakage evaluation

The severity of dye penetration was analyzed according to a 0 to 3 scale scoring system (Figure 4.18) used by Loguercio *et al*, (2004), Yazici, Celik and Ozgunaltay, (2004) previously in their study and is reflected in Table 4.5

Table 4.5: Criteria for microleakage evaluation of dye penetration along the tooth – restoration interface.

Score	Details
0	No dye penetration
1	Dye penetration up to less than half the cavity depth
2	Dye penetration up to more than half the cavity depth, but not extending to the axial wall
3	Dye penetration up to the axial wall and beyond

4.4 – SURFACE MICROHARDNESS TEST

The Vickers hardness measurements were carried out using a Vickers microhardness tester (Vickers Instruments Ltd, York, England) (Figure 4.19) under a load of 50 gm for 30 seconds. Two indentations were made on the top surface and two on the bottom surface of each specimen. The measurements were taken from the top and the bottom surfaces of the 2mm thick specimens.

Figure 4.19: Vickers hardness tester

4.5 – DATA ANALYSIS

The scoring from the observers and the final scores were tabulated using an excel spreadsheet.

The data was analyzed using a commercially available statistical software package (SPSS 13.0, SPSS Inc.).

Original scores were supplied to the statistician. For microleakage mean of the 6 dye penetration data measured on each restoration was recorded. The data was analyzed using Kruskal-Wallis one way ANOVA on ranks (significance at $p \leq 0.05$) to find if there were any differences

that were statistically significant between the groups. The Mann-Whitney *U* test was then used for pair-wise comparison between the groups.

Surface microhardness data was analyzed by a one way analysis of variance (ANOVA) and then a Tukey's HSD multiple comparison test was carried out to find which groups differed from the others.

4.6 – Conclusion

The materials that were used in the study included 80 extracted molar teeth, a nano-ceramic composite (Ceram X mono), a nanofilled composite (Premise), a nanohybrid composite (Grandio), a microhybrid composite (Z100), vinyl polysiloxane (President), fissure burs and a light curing unit (Demetron LC). Cavities for the microleakage test were prepared according to a previously used method (Besnault and Attal, 2003) but with a modification for the purpose of this study. After preparation of the Class II slot cavities, the teeth were randomly assigned to receive one of the four materials used to restore the cavities as follows: Group 1 – Ceram X mono (20 teeth); Group 2 – Premise (20 teeth); Group 3 – Grandio (20 teeth); Group 4 – Z100 (20 teeth). The teeth were then placed in a 0.5% methylene blue dye solution and thermocycled. They were then sectioned and the degree of dye penetration was evaluated by two independent observers. Surface microhardness specimens for the above mentioned composite restorative materials were prepared using a resin mold. Two indentations were made on the top and bottom surfaces of the specimens using a Vickers hardness tester at 50 gm load and a 20 second dwell time. The scores were tabulated using an excel spreadsheet. Data analysis was done using a SPSS computer package.

CHAPTER 5

RESULTS

5.1 – INTRODUCTION

A total of 80 extracted molar teeth for microleakage and a total of 64 specimens for surface microhardness were used in this study. The teeth were randomly assigned to receive Ceram X mono, Premise, Grandio and Z100 as a restorative material for Class II slot cavities. The restorations were independently evaluated for the degree of microleakage by two previously calibrated observers. In addition, the observers were blinded as to the restorative materials being used. Where there was disagreement, the observers discussed the results for final determination by consensus. The microleakage was measured by scoring the dye penetration on an ordinal scale ranging from 0 to 3 where 0 represented no evidence of dye penetration and 4 represented dye penetration extending beyond the axial wall.

For surface microhardness 16 specimens were made for each material. Microhardness readings were taken from the top and bottom surfaces of the specimens using a Vickers hardness tester.

In this chapter, the findings from the microscope, microleakage scores, microhardness measurements and the statistical analysis of the results are presented.

5.2 – MICROLEAKAGE

5.2.1 – Microscopic Findings

There was a varying degree of dye penetration along the gingival margin of the restored cavities for all four restorative materials (Ceram x mono, Premise, Grandio and Z100). In some sections the dye penetrated not only along the restorations, but also penetrated into the adjacent dentinal tubules. Dye penetration was more severe in the restorations with dentine or cementum gingival margins as compared to the restorations with enamel gingival margins (Figures 5.1, 5.2 and 5.3). No voids were observed between the different increments of the restorative materials and between the bonding agent and the restorative material.

Figure 5.1: Showing microleakage at enamel margin

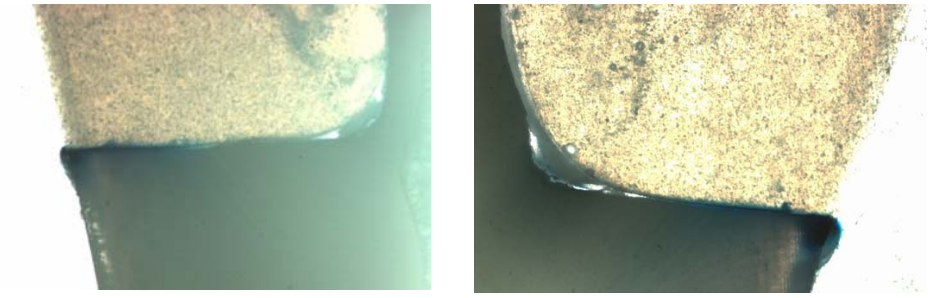


Figure 5.2 and 5.3: Showing microleakage at dentine margin

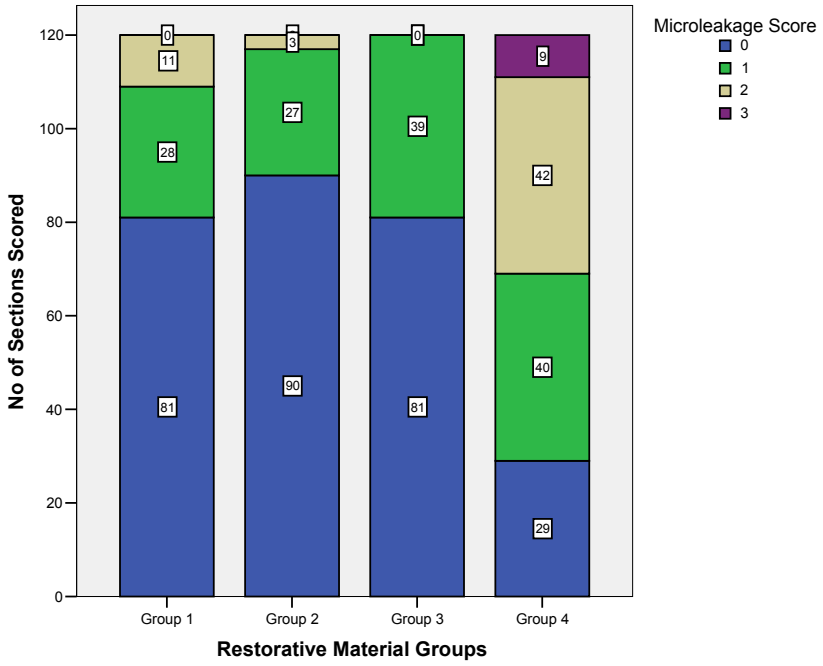
5.2.2 – Microleakage Scoring

The total number of each score in each group was calculated and is represented in Table 5.1 for enamel and in Table 5.2 for dentine.

Table 5.1: Total number of microleakage scores for each group: ENAMEL

Crieteria Depth of dye penetration in cavity 0=none 1= first half 2= second half 3= full depth and beyond	**Group 1** Prime & Bond NT + Ceram X	**Group 2** Optibond Solo Plus + Premise	**Group 3** Admira Bond + Grandio	**Group 4** Adper Scotchbond + Z100
	Score count	**Score count**	**Score count**	**Score count**
0	81	90	81	29
1	28	27	39	40
2	11	3	0	42
3	0	0	0	9

Figure 5.4: Enamel Microleakage Scores for Each Group



From the summarized results, it is evident that Ceram X mono, Premise and Grandio had a higher number of zero scores than that seen with Z100, implying that the sealing ability of these materials was better than that of Z100 at the gingival margin when on enamel. Z100 had the highest number of specimens (9) with a score of 3, implying that these specimens restored with Z100 had leaked to an extent where the dye penetration had reached the axial wall and beyond. Ceram X mono, Premise and Grandio had no specimens with a score of 3. Grandio also had no specimens with a score of 2 while Ceram X had 11 and Premise had 3 specimens with a score of 2. The results are represented by a bar graph in Figure 5.4.

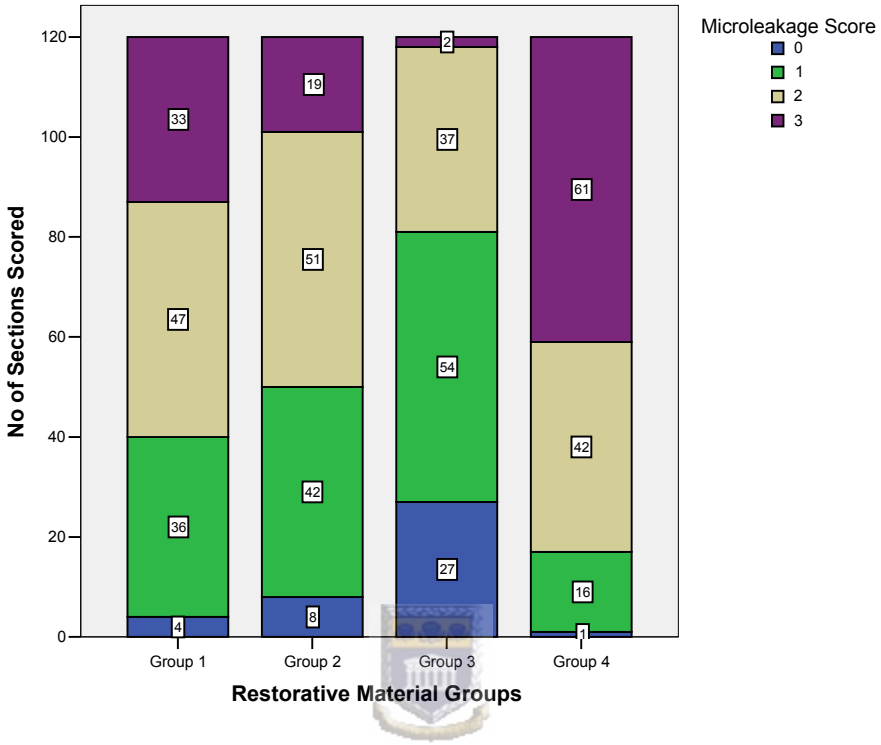
Table 5.2: Total number of microleakage scores for each group: DENTINE/CEMENTUM

Crieteria Depth of dye penetration in cavity 0=none 1= first half 2= second half 3= full depth and beyond	**Group 1** Prime & Bond NT + Ceram X	**Group 2** Optibond Solo Plus + Premise	**Group 3** Admira Bond + Grandio	**Group 4** Adper Scotchbond + Z100
	Score count	**Score count**	**Score count**	**Score count**
0	4	8	27	1
1	36	42	54	16
2	47	51	37	42
3	33	19	2	61

From the summarized results, it is clear that Grandio had the highest count of zero scores (27) amongst the materials tested, implying that these specimens showed no microleakage at all. Z100 had the highest number of specimens (61) with a score of 3, implying that these specimens restored with Z100 leaked to an extent where the dye penetration had reached the axial wall or beyond. This was followed by Ceram X mono and Premise that had 33 and 19 specimens with a score of 3 respectively. Grandio showed the least number of specimens (2) with a score of 3. This indicated the better sealing ability of dentine or

cementum margins by Grandio. The results are represented by a bar graph in Figure 5.5.

Figure 5.5: Dentine Microleakage Scores for Each Group



5.2.3 – Analysis of Results

Statistical analysis of the data was performed using a commercially available statistical software package (SPSS 13.0, SPSS Inc.).

Agreement between the Observers

When observers were compared in their scoring of microleakage, they were found to be in agreement in 89% of the cases with a Kappa value of 0.78. The "benchmark" that is used for the measure of strength of agreement of the Kappa statistics is shown in Table 5.3.

Table 5.3: Showing the labels assigned to the corresponding ranges of Kappa values

Kappa Statistics	Strength of Agreement
<0.00	Poor
0.00 – 0.20	Slight
0.21 – 0.40	Fair
0.41 – 0.60	Moderate
0.61 – 0.80	Substantial
0.81 – 1.00	Almost perfect

Comparison of Materials

A Kruskal-Wallis analysis of variance (ANOVA) test was carried out to investigate if statistically significant differences existed between the experimental groups at a significance level of $p \leq 0.05$. A summary of the means, number of sections scored, the standard deviation, the median as well as the minimum and maximum values for microleakage at an enamel margin is represented in Table 5.4.

Table 5.4: Summary of means, number of sections scored, standard deviation, median, minimum and maximum values for microleakage at an enamel margin.

ENAMEL						
Smallest N for any variable: 480						
	Means	N	Std. Dev.	Minimum	Median	Maximum
Group 1	0.42	120	0.656	0	0	2
Group 2	0.28	120	0.501	0	0	2
Group 3	0.33	120	0.470	0	0	1
Group 4	1.26	120	0.912	0	1	3
All Groups	0.57	480	0.770	0	0	3

Table 4.1: Experimental Groups	
Group 1	Prime & Bond NT + Ceram X mono (nano-ceramic)
Group 2	OptiBond SoloPlus + Premise (nanofilled)
Group 3	Admira Bond + Grandio (nanohybrid)
Group 4	Adper Scotchbond + Z100 (microhybrid)

The results of the Kruskal-Wallis test showed that there was a highly significant difference amongst the restorative material groups tested ($p \leq 0.05$) for both an enamel and dentine or cementum margin Table 5.5. Once it was established that significant differences existed between the groups, a Mann-Whitney *U* test was carried out for a pair-wise comparison to determine which group differed from the others at a significance level of $p \leq 0.05$.

Table 5.5: Summary of Kruskal-Wallis test

	Mean	
	Enamel	**Dentine**
Chi-Square	29.637	25.526
Df	3	3
Asymp. Sig.	0.000	0.000

Table 5.6, represents the statistically significant differences between groups at a 95% confidence level for microleakage at an enamel margin for the mean assessment score of the microleakage. It was found that group 4 differed significantly from groups 1, 2, and 3. However, there was no statistically significant difference between groups 1, 2 and 3 at this level of confidence.

Table 5.6: Statistically significant differences (p≤0.05) between groups using the mean values – ENAMEL

Groups	1	2	3	4
	M=0.42	M=0.28	M=0.33	M=1.26
1		0.718	0.911	0.000
2			0.608	0.000
3				0.000
4				

M=mean score for a group
Shaded areas indicate areas of significance

Table 5.7 represents the mean, number of sections scored, the standard deviation, the median as well as the minimum and maximum values for microleakage in margins located in dentine or cementum.

Table 5.7: Summary of means, number of sections scored, standard deviation, median, minimum and maximum values for microleakage in margins located in dentine or cementum.

DENTINE or CEMENTUM						
Smallest N for any variable: 480						
	Means	N	Std. Dev.	Minimum	Median	Maximum
Group 1	1.91	120	0.840	0	2	3
Group 2	1.68	120	0.822	0	2	3
Group 3	1.12	120	0.769	0	1	3
Group 4	2.36	120	0.742	0	3	3
All Groups	1.76	480	0.910	0	2	3

A Mann-Whitney *U* pair-wise comparison test for microleakage in dentine was performed. The results are summarized in Table 5.8. It was found that group 1 differed statistically significantly from groups 3 and 4. However, there was no statistically significant difference between groups 1 and 2. Group 2 differed statistically significantly from groups 3

and 4 and group 3 differed statistically significantly from groups 2 and 4.

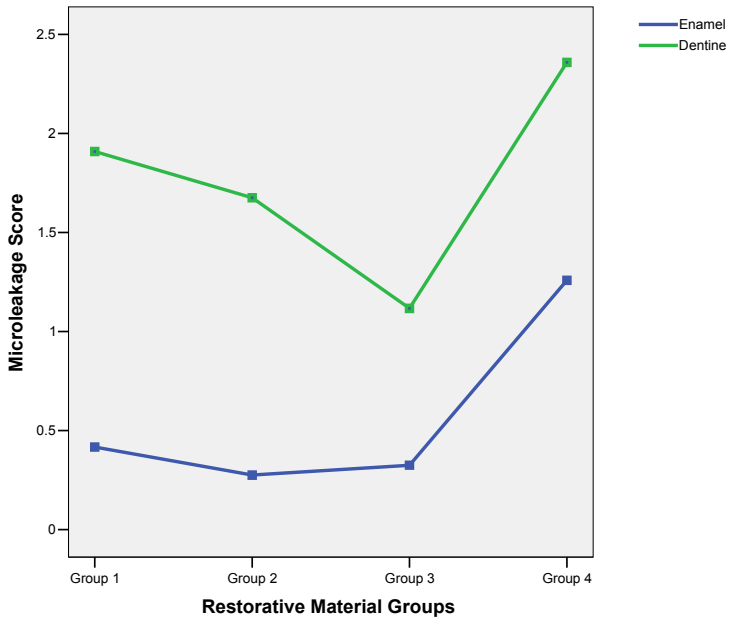
Table 5.8: statistically significant differences (p≤0.05) between groups using the mean values - DENTINE

Groups	1	2	3	4
	M=1.91	M=1.68	M=1.12	M=2.36
1		0.328	0.003	0.05
2			0.019	0.002
3				0.000
4				

M=mean score for a group
Shaded areas indicate areas of significance

A summary of the means for the margins located in enamel and dentine or cementum as regards microleakage, is graphically represented in Figure 5.6

Figure 5.6: Summary of Means



5.2.4 – Box Plot Section

Box plot diagrams were also piled that graphically illustrate the average microleakage of the different experimental groups with their margins in enamel as well as in dentine or cementum. The green line dividing each rectangle indicates the median for each experimental group, while the top and the bottom of each rectangle indicates the top 75% and the bottom 25% of the values observed respectively. The blue lines indicate the 10% and 90% values observed respectively. Abnormally high or low readings are indicated by single blue dots and are regarded as outliers.

Figure 5.7: Box Plot for Enamel Microleakage

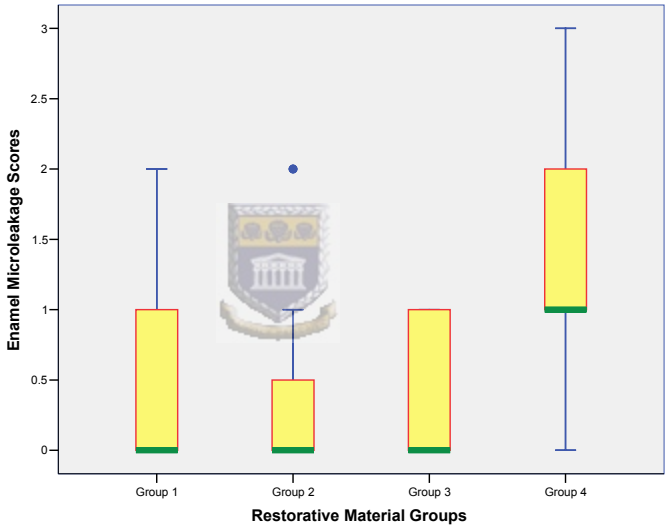
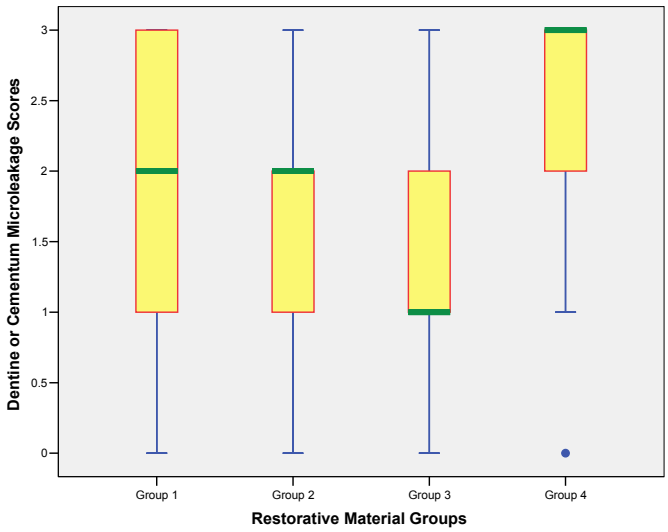


Figure 5.8: Box Plot for Dentine or Cementum Microleakage



5.3 – SURFACE MICROHARDNESS

Statistical analysis of the data was performed using a commercially available statistical software package (SPSS 13.0, SPSS Inc.). A One way analysis of variance (ANOVA) was carried out to investigate if statistically significant differences existed between the experimental groups at a significance level of $p<0.05$. A summary of the means, the standard deviation and the means for hardness ratio (bottom/top) for all groups is represented in Table 5.9

Table 5.9: Vickers surface microhardness numbers

Groups	N	Top Surface	Bottom surface	Hardness Ratio (bottom, top)
		Mean (SD)	Mean (SD)	
Group 1 (Ceram X)	16	94.42 (6.41)	78.16 (7.83)	0.830 (0.087)
Group 2 (Premise)	16	93.25 (9.24)	77.02 (5.88)	0.831 (0.083)
Group 3 (Grandio)	16	113.2 (8.92)	108.83 (14.08)	0.961 (0.097)
Group 4 (Z100)	16	119.68 (11.97)	116.94 (10.72)	0.985 (0.127)

*95% confidence interval.
SD = standard deviation
N = number of specimens tested

A one way analysis of variance (ANOVA) showed statistically significant differences amongst the different materials tested. A Tukey's HSD post-hoc test for multiple comparisons was carried out to find which material differed from the others. The top and the bottom surface readings within a group were also compared using a Paired–Samples T test.

5.3.1 – Top and Bottom Surfaces

For groups 1 and 2 there was a statistically significant difference ($p<0.05$) between the readings taken from the top and the bottom

surfaces respectively however, for groups 3 and 4 there was no statistically significant difference ($p>0.05$) between the readings taken from the top and the bottom surfaces of the specimens respectively Table 5.10.

Table 5.10: Top and bottom surface comparison within each group

	Top Surface Mean	Bottom Surface Mean	Top-bottom Correlation*
Group 1	94.42	78.16	0.000
Group 2	93.25	77.02	0.000
Group 3	113.2	108.83	0.134
Group 4	119.68	116.94	0.471

* 95% confidence interval

5.3.2 – Top Surface Hardness across Groups

The means for groups 1 and 2 were statistically significantly different ($p<0.05$) when compared to those of groups 3 and 4. However, there was no statistically significant difference between groups 1 and 2 and similarly between groups 3 and 4. The results are summarized in Table 5.11.

Table 5.11: Top surface microhardness comparison across groups at 95% confidence interval.

Groups	{1}	{2}	{3}	{4}
	M=94.42	M=93.25	M=113.2	M=119.68
{1}		0.985	0.000	0.000
{2}			0.000	0.000
{3}				0.214
{4}				

M= mean microhardness value for top surface
Shaded areas represent areas of significance

5.3.3 – Bottom Surface Hardness across Groups

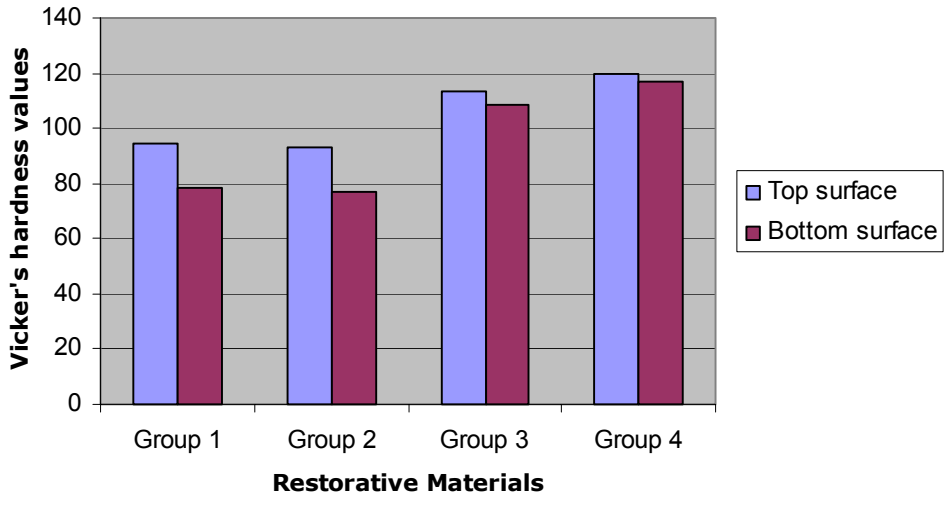
The means for groups 1 and 2 were statistically significantly different ($p<0.05$) when compared to those of groups 3 and 4. However, there was no statistically significant difference between groups 1 and 2 and similarly between groups 3 and 4. The results are summarized in Table 5.12.

Table 5.12: Bottom surface microhardness comparison across groups at 95% confidence interval.

Groups	{1}	{2}	{3}	{4}
	M=78.16	M=77.02	M=108.83	M=116.94
{1}		0.989	0.000	0.000
{2}			0.000	0.000
{3}				0.117
{4}				

M= mean microhardness value for top surface
Shaded areas represent areas of significance

Figure 5.9: Average top and bottom surface hardness values for each group



5.4 – CONCLUSION

A total of 80 extracted molar teeth were used for microleakage analysis in this study. Evaluation of microleakage scores was done using the mean values. In cavities with a gingival margin in enamel Ceram X, Premise and Grandio showed a better sealing ability compared to Z100. In cavities with a gingival margin in dentine or cementum all the materials showed some degree of microleakage. However, there was a highly statistically significant difference in the sealing ability of Ceram X, Premise, Grandio and Z100. Grandio leaked statistically significantly less than all other materials.

For surface microhardness 64 disc shaped specimens were used in this study. The mean microhardness values of Grandio and Z100 were statistically significantly different from those of Ceram X and Premise. However, there was no statistically significant difference between the top and bottom surface microhardness values of Grandio and Z100 whereas the top and bottom surface microhardness values of Ceram X and Premise differed statistically significantly at a confidence level of 95%.

CHAPTER 6

DISCUSSION

6.1 – INTRODUCTION

Recent advances in resin adhesives and restorative materials, as well as an increased demand for esthetics, have stimulated a great increase in the use of resin-based composites in posterior teeth (Cobb *et al*, 2000). Despite the remarkable developments in the technology of the resin composite restorative materials, clinical failures of resin restorations are still reported, particularly when resin composites are placed in stress-bearing areas (Kournetas *et al*, 2004). Poor marginal adaptation along the cervical margins, secondary caries, material fractures and inadequate wear resistance under masticatory loads have been established as the common clinical problems of posterior resin composite restorations (Hickel and Manhart, 2001, Peutzfeldt, 1997).

These problems reflect drawbacks in the resin composite restorative materials and their adhesive systems. Besides the development in the field of adhesive agents, a number of strategies have been introduced to address the short-comings of resin composite restorative materials. The nanocomposites based on nanotechnology are the latest innovations to address the shortcomings that have been identified.

Ceram X by Dentsply (Germany), Premise by Kerr (USA), and Grandio by Voco (Germany) are some of the newly marketed nanocomposite restorative materials.

This study investigated the sealing ability of Ceram X, Premise, Grandio and Z100 when used in Class II restorations with a gingival margin in

enamel and in dentine or cementum. All four materials are biocompatible and have been approved for clinical use, thus making the results of the study clinically relevant.

Dye leakage studies are amongst the most frequently used methods for detecting microleakage (Déjou, Sindres and Camps, 1996). The other methods include the use of color producing micro-organisms, radioactive isotopes including ^{45}Ca, ^{131}I, ^{35}S, ^{22}Na, air pressure method, neutron activation analysis, electrochemical studies, scanning electron microscopy, thermal and mechanical cycling and chemical tracers (Taylor and Lynch, 1992). The dye leakage method was used in this study because it was simple, inexpensive and did not require the use of complex laboratory equipment. A 0.5% methylene blue dye was chosen as the agent of dye penetration to measure microleakage.

The degree of dye penetration supposedly indicates the inert space between the tooth margin (enamel and dentine or cementum) and the restorative material interface that could allow the ingress of bacterial endotoxins and their inflammatory products. In this study, dye penetration was measured after making longitudinal sections to expose the tooth-restoration interface. The limitation of the longitudinal sections was that only the sectioned part of the restored cavity could be examined. The observed section may not necessarily be the best representative of the total leakage distribution (Youngson *et al*, 1998) since dye penetration may vary from one zone to another in the same tooth-restoration interface (Tay *et al*, 1995b and Hilton, Schwartz and Ferracane, 1997). Gale, Darvell and Cheung (1994) reported that microleakage was a three-dimensional phenomenon and that different locations and angles of sectioning might result in completely different dye penetration scores in the interface. This could make it possible for the observers to miss greater dye penetration which could be on the part of the restored cavity that was not exposed, i.e. not in the line of sectioning (Federlin *et al*, 2002).

Multiple-surface scoring methods have been regarded superior to single-surface scoring methods because the results obtained seem to be more representative of the microleakage (Mixon *et al*, 1991). Raskin *et al*, (2003) recommended that at least three sections should be used to avoid an underestimation of the microleakage observed. Based on these findings three sections of each restoration were made to evaluate the microleakage so that the results could be more representative of the actual leakage experienced with these restorative materials.

The restorative materials constantly undergo changes of a thermal nature when placed in the oral environment, due to an intake of food and fluids at varying temperatures (Sidhu, Carrick and McCabe, 2004). Laboratory simulations of the clinical situation are often performed because clinical trials are costly and time consuming (Gale and Darvell, 1999).

Thermal cycling is a widely used method in dental research to simulate temperature changes that take place in the oral environment. It aims at thermally stressing the junction at the tooth-restoration interface by subjecting the restored tooth to extreme temperature changes compatible with temperature changes encountered intraorally (Wahab, Shaini and Morgano, 2003). All the specimens for microleakage evaluation were subjected to thermocycling procedures in a buffered (pH 7) 0.5% methylene blue dye solution at temperature ranges of 5°C to 55°C with a dwell time of 15 seconds for 500 cycles. The temperature range of 5°C to 55°C was used according to the International Organization for Standardization (ISO) TR11405 standard and this is the estimate of the range that has been reported on the surfaces of molar teeth in the mouth of the patient (Gale and Darvell, 1999).

In this study every effort to simulate the clinical situation as closely as possible was undertaken. A restoration template was fabricated. Two third molars were embedded in dental stone to the level of the

cementoenamel junction (CEJ), approximately 12 to 14 mm apart. This space was left between the two molars to place a test specimen embedded in a polyvinyl siloxane impression material. By appositioning the impression material against the root surface, the excellent elasticity of the polyvinyl siloxane impression material supposedly simulated the periodontal ligament. A metal matrix band in a retainer and wooden wedges were used during the restoration procedure that added further in simulating the clinical situation.

A total etch technique was used for all the restorations. The adhesive systems were used strictly according to the manufacturers' recommendations. Each material was placed in three increments and each increment was cured for 40 seconds with a conventional halogen light curing unit to minimize the polymerization shrinkage (Linden and Swift, 1994). All the specimens were stored in distilled water at 37°C for 7 days and the margins were then finished and polished with aluminum coated flexible discs.

All the teeth in the study were completely covered with two layers of nail varnish except for a 1mm area around the gingival margin of the tooth-restoration interface. The apices of all the teeth were sealed with a resin modified glass ionomer filling material to ensure that the only leakage if at all would be through the restoration-tooth interface.

The study also investigated the surface microhardness of the resin-based composite restorative materials being evaluated. Specimens were prepared using a special clear resin mold. The dimensions of the specimens were 5 mm in diameter and 2 mm in thickness. The curing time for each specimen was selected as 40 seconds based on the study of Peutzfeldt, Sahafi and Asmussen, (2000).

6.2 – RESULTS

6.2.1 – Microleakage

The samples were evaluated for the degree of dye penetration by two independent observers. The observers scored the degree of dye penetration on an ordinal scale ranging from 0 to 3. In their scoring they were in agreement 89% of the times (Kappa value = 0.78). According to Landis and Kock (1977) this represented a substantial agreement (Table 5.3).

The analysis of the present study indicated microleakage from the worst leakage to the least leakage in restorations with a gingival margin in enamel in the following sequence Table 6.1:

Table 6.1: Ranking of materials according to the severity of microleakage

Restorative Material Groups	Microleakage
Group 4 = Z100 (microhybrid) + Adper Scotchbond	Worst leakage
Group 1 = Ceram X mono (nano-ceramic) + Prime & Bond NT	↓
Group 2 = Premise (nanofilled) + OptiBond SoloPlus	↓
Group 3 = Grandio (nanohybrid) + Admira Bond	Least leakage

Group 4 differed statistically significantly from all the other groups however, there were no statistically significant difference between groups 1, 2 and 3.

Proper adhesion between the restorative material and the cavity walls results in good marginal sealing with less microleakage and a longer life of the restoration. Enamel has been regarded as a reliable substrate for bonding (Yazici, Celik and Ozgunaltay, 2004). The results of the present microleakage study demonstrate better sealing ability in enamel than in dentine or cementum margins and this is in accordance with previous

findings (Neiva *et al*, 1998, Opdam, Roeters, and Burgersdijk, 1998, Thonemann *et al,* 1999). No material was able to completely eliminate microleakage at the enamel margin. Grandio showed the best results compared to Ceram X, Premise and Z100. Eleven sections of Ceram X and three sections of Premise showed dye penetration for the full depth whereas in the case of Grandio dye penetration was only observed in the first half of the tooth-restoration interface. In most of the sections (75%) these three materials prevented dye penetration at the enamel margin (Figure 5.4, page 56). The results for Z100 were statistically significantly different results when compared to Ceram X, Premise and Grandio.

These results could be attributed to the assessment of dye penetration in serially sectioned specimens using direct magnification that may contribute to a more sensitive detection of small amounts of dye throughout the interface (Cvitko, Denehy and Boyer, 1992). It should also be observed that the mean recorded leakage scores for all the groups except Z100 (1.26) was low (around 0.42), showing positive results with the materials and techniques when sealing ability is of concern clinically.

For the restorations with a gingival margin in dentine or cementum the analysis of the present study indicated microleakage from the worst leakage to the least leakage in the following sequence Table 6.2:

Table 6.2: Ranking of materials according to the severity of microleakage

Restorative Material Groups	**Microleakage**
Group 4 = Z100 (microhybrid) + Adper Scotchbond	Worst leakage
Group 1 = Ceram X mono (nano-ceramic) + Prime & Bond NT	↓
Group 2 = Premise (nanofilled) + OptiBond SoloPlus	↓
Group 3 = Grandio (nanohybrid) + Admira Bond	Least leakage

Group 1 and 2 differed statistically significantly from groups 3 and 4. However, there was no statistically significant difference between groups 1 and 2. Group 3 differed statistically significantly from groups 1, 2 and 4.

The marginal gap formation is a rather complicated phenomenon. It has been suggested that the marginal and internal adaptation of a resin composite restoration is influenced by the bonding ability of the adhesive agent used, the volumetric contraction of the resin composite, the stress induced during polymerization, the stiffness and the rheological properties of the resin composite (Davidson and de Gee, 1984, Retief, 1994). Generally, composite-enamel bonds survive these stresses while failures are observed at the composite-dentin or composite-cementum interfaces (Davidson and de Gee, 1984).

In the present experiment several restorations revealed insufficient sealing performance at the dentine or cementum margin. The inadequate sealing of a restoration permits leakage of bacteria and their products that can result in staining in the borders of the restoration, recurrent caries, pulpal irritation and pathological changes (Kournetas *et al*, 2004).

Bonding to dentine is far more difficult and less predictable than bonding to enamel because dentine is about 75% inorganic in nature as opposed to enamel that is 95% inorganic (Yazici, Celik and Ozgunaltay, 2004). Dentine is regarded as a biologic composite of apatite filler crystallites in a collagen matrix with a fluid-filled tubular structure connecting the pulp to the enamel-dentine junction. Dentine and cementum are complex substrates for bonding. The cementum outer layer is hypomineralized and hyperorganic and that does not provide microretention for the adhesive materials even after acid-etching (Loguercio *et al*, 2004). Difficulty in obtaining good adhesion to dentine or cementum was observed in this study. In the present study no

material was able to completely eliminate microleakage at the dentine or cementum margin. Only one group (Grandio + Admira Bond) showed the best sealing ability when compared to the other groups (Ceram X + Prime & Bond NT, Premise + OptiBond Solo Plus and Z100 + Scotchbond) whereas Z100 showed the worst sealing ability when compared to Ceram X, Premise and Grandio. There was no statistically significant difference between Ceram X and Premise in their sealing ability.

Polymerization shrinkage is one of the most critical properties of resin based composite restorative materials (Chen *et al*, 2001). It is also considered as one of the major problems that still imposes limitations in the application of direct aesthetic restorative techniques (Loguercio *et al*, 2004, Yazici, Celik and Ozgunaltay, 2004, Yap *et al*, 2000). Composite resins shrink during polymerization mainly because the monomeric units of polymer are located closer to one another than they are in the original monomer state. The majority of the shrinkage can be resolved before the polymerization gel point by flow that allows composites to change shape thus reducing the contraction stresses. Following gel formation contraction stress build-up occurs since subsequent shrinkage is obstructed because the material is too rigid to allow plastic flow to compensate for the original volume (Chen *et al*, 2001). While restoring an adhesive cavity, the resin composite is restricted from changing shape except at the free surface because it is bonded to the walls and floor of the rigid tooth structure. This causes further stress development and increases the possibility of microleakage.

The polymerization shrinkage values of the tested materials (Table 2.1) imply that this may be one of the factors responsible for the different behavior evident in the study. According to the manufacturers' Z100 and Ceram X have volumetric polymerization shrinkage values of 2.8% and 2.3% respectively, while Premise and Grandio have shrinkage

values of 1.6% and 1.57%. Premise (84% by weight) and Grandio (87% by weight) are highly filled resin composites with a nominal resin matrix content that results in low shrinkage values and contraction stresses. The poor sealing ability of Z100 and Ceram X may be the result of the higher polymerization shrinkage values but it does not seem to be responsible for the poor sealing ability of Premise.

The use of different dentine bonding systems introduces an additional factor of influence to the material factor. All the tested materials were used with their corresponding adhesive systems according to the manufacturers' instructions. The difference in sealing ability of tested materials may be due to the different formulations of the adhesive systems used.

Prime & Bond NT is an acetone based bonding agent that contains silica nanofiller. OptiBond SoloPlus is an ethanol based adhesive system containing silica and barium glass filler particles. Admira Bond is also an acetone based bonding agent containing ormocers filler particles. Adper Scotchbond is an acetone based unfilled resin. In this study Admira Bond filled with ormocer nano-particles appeared to produce better marginal sealing both at the enamel and dentine or cementum margins when compared to the other materials. That is the bond strength of Admira Bond was sufficient to resist polymerization contraction stresses and to maintain the integrity of the tooth-restoration interface. Van Meerbeek *et al*, (1993) reported that the flexibility and elasticity of the bonding layer provides a gradient of elasticity between the resin-bonding areas that may absorb the stresses induced during polymerization. Tay, Moulding and Pashley (1999) found that nanofillers from Prime & Bond NT were congested around patent tubular orifices but were not found within the interfibrillar spaces of the hybrid layer. They concluded that the aggregation of the nanofillers within the adhesive resulted in filler clusters that were too large to infiltrate the

interfibrillar space of the hybrid layer. This may have resulted in the poor bonding seen with Prime & Bond NT.

Effective bonding requires removal of the smear layer with acids followed by rinsing and drying of the preparation for a clean, adequately moist surface before application of the bonding agent (Owens, 2002). Hashimoto *et al*, (2000b) reported that after prolonged acid conditioning there was an increase in the demineralized dentine zone without resin impregnation, within the hybrid layer. This resulted in lower bond strength values. The increase in the demineralized dentine zone without resin impregnation can be the reason for the increased microleakage evident in the etched dentine in this study. Perdigao, Swift and Cloe, (1993), Santini, Plasschaert and Mitchell, (2000) reported that dentine should not be dehydrated or desiccated following conditioning and that the presence of excessive surface moisture could result in voids. All manufacturers recommended the wet bonding technique for dentine that was followed in this study.

The results of a comparative *in vitro* study of the marginal sealing of restorations depends on many parameters such as the selection of teeth, the method of restoring the cavities, the type of tracer or dye and the thermal cycling (Chohayeb, 1988, Soderholm, 1991, Taylor and Lynch, 1992). In this study every possible effort was made to treat all the groups equally. After restoration of the cavities the teeth were stored at 37°C for one week. Finishing, polishing and thermocycling was carried out for all the groups in a similar way. Class II cavities were standardized and their depths were controlled. Before sectioning the teeth were embedded in clear resin in order to accurately identify the bucco-lingual plane. The length of the interface was equal in each section, so the extent of dye penetration in each group may be compared.

Multiple-surface scoring methods have been regarded to be superior to single-surface scoring method because the results obtained seem to be more representative of the microleakage (Mixon *et al*, 1991). In the present study three sections of each restoration were made to evaluate microleakage so that the results could be more representative of the actual leakage present. Scoring of both surfaces of each section resulted in several measurements being available for analysis. In these cases, various methods have been proposed to optimize the use of the data (Déjou, Sindres and Camps, 1996). Each measurement may be considered as a statistical criterion or a criterion can be used that is calculated summarizing all the data from each restoration (Swift and Hansen, 1989).

The mean of the dye penetration data measured for each restoration was recorded in the present study. The mean describes a statistical distribution of data but under two conditions: 1- normality of distribution of data measured on each restoration and 2- no aberrant data. In the opposite case, the median situated in the centre of the series of data and not influenced by the shape of the distribution of the data or aberrant data is preferable (Déjou, Sindres and Camps, 1996).

6.2.2 – Surface Microhardness

The surface hardness test has been used as an indicator of the degree of polymerization (Tagtekin *et al*, 2004). For the resin-based composite materials evaluated in this study statistically significant differences in microhardness values between the materials were found. Based on these findings the null hypothesis was rejected.

The surface microhardness measurements were taken from the top and bottom surfaces of all the specimens in each group. The mean for each material was calculated and subjected to statistical analysis.

The results of the present study demonstrated microhardness values from the hardest to the least hard material in the following sequence Table 6.3:

Table 6.3: Ranking of materials according to surface microhardness values

Restorative Material Groups	Microhardness
G4 = Z100	Hardest
G3 = Grandio	↓
G1 = Ceram X	↓
G2 = Premise	Least hard

Z100 and Grandio differed statistically significantly from Ceram X and Premise. However, there was no statistically significant difference between Z100 and Grandio and between Ceram X and Premise.

The top surfaces of all the materials showed higher microhardness values as compared to the bottom surfaces of the specimens. There was no statistically significant difference between the top and bottom surface values for Z100 and Grandio whereas the top and bottom surfaces of Ceram X and Premise showed a statistically significant difference.

Statistically significant differences in the microhardness readings may be due to the different composite formulations that can influence the degree of polymerization in the deepest layer (Tagtekin *et al*, 2004). It may also be due to the fact that when light passes through the material it is dispersed and the efficacy of polymerization in the deepest layer is compromised (Ruyter and Oysæd, 1982)

The shade of a resin composite material is another factor that can have an influence on the degree of polymerization (Shortall, Wilson and Harrington, 1995). Several studies have demonstrated that darker

shades show less polymerization when compared to the lighter shades (Onose *et al*, 1985, Swartz, Phillips and Rhodes, 1983).

It was also noted that the different materials studied reacted differently to the same light intensity and the same period of exposure, even when similar shades were used. A study by Davidson-Kaban *et al*, in 1997 demonstrated a tendency towards the need for greater power density to achieve better polymerization in the deepest portions of the restorations made with darker shades as compared to the more translucent shades. However, another study that used hardness test and infra-red analysis showed that dark-shaded composites presented a depth of polymerization similar to the light-shaded composites (Ferracane *et al*, 1986).

Therefore, it seems that the depth of polymerization is much more dependent on the opacity or translucency of a resin composite rather than the shade itself (Poskus, Placido and Cardoso, 2004b). Light transmission in darker shades is reduced due to their opacity, but the white pigments in very light shades also tend to turn the material more opaque, thus possibly dispersing light and limiting its penetration into the composite (Sakaguchi, Douglas and Peters, 1992). In the present study an A3 shade was used for all the materials tested.

All the materials showed a reduction in the hardness values when moving away from the top surface. In the present study Ceram X and Premise showed the greatest reduction compared to Z100 and Grandio. The large content of micro particles with a 0.04 μm diameter has been reported to increase the light dispersion thus favoring a lower degree of polymerization in deeper parts of the restoration (Kawaguchi, Fukushima and Miyazaki, 1994, Kanca, 1985). It has also been reported that when particle size reaches one-half the wavelength of activating light, that is, approximately 0.23 μm, than light dispersion is greater (Poskus, Placido and Cardoso, 2004b). Small particle size and high filler

content may have caused greater light dispersion in Ceram X and Premise resulting in low hardness values.

Although differences were detected amongst the resin composites tested in this study, it must be noted that a specific value of hardness cannot be related to a certain degree of polymerization when different formulations of resin composites are compared because their mechanical properties also depend on other factors related to the resin composition (Poskus, Placido and Cardoso, 2004b).

Hardness tests consist of an indentation of a static diamond tip under load into the tested material over a certain period of time. After removal of the load, the microscopic impression obtained from this procedure is evaluated. For a Vickers' hardness test the diamond is pyramidal in shape and hence a square shaped impression is obtained in the material being tested. Measurements are made on both diagonals and mean values are obtained. The polymeric materials such as resin composites show the phenomenon of elastic recovery that could affect both diagonals in the Vickers' impression, thus masking the results. Errors may occur due to the elastic recuperation, difficulty in focusing the two diagonals at the same time, duration of the contact period and impact of the load on the samples due to any vibration.

Microhardness is defined as the resistance to permanent deformation only caused by indentation after load. There will be an overestimation of the hardness due to the elastic recovery when Vickers' hardness tests are used for resin-based composite restorative materials. Microhardness values may also differ depending upon the response of the materials after load is removed (Van Meerbeek *et al*, 1993 and Willens *et al* 1993).

CHAPTER 7

LIMITATIONS OF THE STUDY

Laboratory studies attempt to reproduce clinical situations but do not entirely reflect variables encountered with the *in vivo* performance of the materials. The main limitation of this study relates to the relevance of *in vitro* studies in predicting the clinical performance of the materials being tested. Extrapolating the data of *in vitro* observations to the clinical situation is often unreliable and should be done with caution for the following reasons according to Swift, Perdigao and Heymann, 1995:

- Tests of this type do not take into account the three-dimensional nature of tooth preparations, and thus underestimate the effects of polymerization shrinkage;

- Other factors that can affect the results may include age and storage conditions of specimens, location and depth of the dentine, thermocycling procedures and the type and duration of the loading forces.

Pashley, (1990) reported that the results of an *in vitro* microleakage study should be viewed as a theoretical maximum level of leakage that may be expected *in vivo*.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 – CONCLUSIONS

This *in vitro* study evaluated the microleakage of nanocomposite restorative materials in Class II restorations and also the surface microhardness of these materials. The null hypothesis was rejected because the results showed statistically significant difference in the microleakage and surface microhardness of the different materials.

Under the conditions set up for this study, several conclusions can be drawn:

The Ceram X mono, Premise and Grandio groups were able to prevent microleakage in restorations with a gingival margin in enamel whereas the Z100 group showed significant microleakage when the margins were in enamel.

No material was able to totally eliminate microleakage in restorations with a gingival margin in dentine or cementum. The materials can be rated in their sealing ability from severe to least leakage as follows:

Z100 > Ceram X > Premise > Grandio

Amongst all the materials tested Grandio showed the best sealing ability when margins were placed both in enamel and dentine or cementum.

Surface microhardness values of Ceram X mono, Premise, Grandio and Z100 were statistically significantly different. Materials can be rated from the hardest to the softest as follows:

Z100 > Grandio > Ceram X mono > Premise

There was no statistically significant difference between the surface microhardness values for Z100 and Grandio, and between Ceram X mono and Premise.

The surface hardness values for the top and the bottom surfaces of Z100 and Grandio were not statistically significantly different and that shows good curing light penetration and optimal polymerization of these materials.

The surface hardness values for the top and the bottom surfaces of Ceram X and Premise were significantly different indicating poor curing light transmission and inadequate polymerization in the deeper layers.

8.2 – RECOMMENDATIONS

The present results are *in vitro* data and definite conclusions should not be drawn until long term *in vivo* studies are completed. More research is needed in the future, especially concerning the nanocompoiste materials, dentin surface moisture and the adhesive systems used.

Concerning microleakage tests, it is also needed to determine their real importance and ability to predict the clinical performance of the material. If this importance is confirmed, it is necessary to clarify the mechanism of dye penetration in the adhesive interface, and to improve the methodology to avoid the great variability of results.

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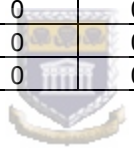
APPENDIX-I

MICROLEAKAGE SCORES

Enamel

Tooth #	GroupMat	Sec1a	Sec1b	Sec2a	Sec2b	Sec3a	Sec3b
1	***G1E***	2	2	2	2	2	2
2	***G1E***	0	0	1	0	1	1
3	***G1E***	0	0	1	1	2	2
4	***G1E***	0	0	1	1	2	2
5	***G1E***	0	0	0	0	0	0
6	***G1E***	1	1	0	0	0	0
7	***G1E***	0	0	0	0	0	0
8	***G1E***	0	0	0	0	0	0
9	***G1E***	0	0	1	1	1	1
10	***G1E***	0	0	0	0	0	0
11	***G1E***	1	1	1	1	1	1
12	***G1E***	0	0	0	1	0	1
13	***G1E***	0	0	0	0	0	0
14	***G1E***	0	0	0	0	0	0
15	***G1E***	1	2	0	0	0	1
16	***G1E***	0	0	0	0	0	0
17	***G1E***	0	1	1	1	0	0
18	***G1E***	0	0	0	0	0	0
19	***G1E***	0	0	0	1	0	0
20	***G1E***	0	0	0	1	0	0



Dentine

Tooth #	GroupMat	Sec1a	Sec1b	Sec2a	Sec2b	Sec3a	Sec3b
1	***G1D***	2	2	2	2	2	2
2	***G1D***	1	1	2	1	2	1
3	***G1D***	1	1	2	2	2	2
4	***G1D***	1	1	1	1	1	1
5	***G1D***	2	3	2	2	2	2
6	***G1D***	1	1	2	2	2	2
7	***G1D***	2	2	2	2	3	3
8	***G1D***	2	2	1	1	2	1
9	***G1D***	2	2	2	2	2	2
10	***G1D***	1	1	1	1	1	1
11	***G1D***	2	2	2	3	3	3
12	***G1D***	3	3	2	3	3	3
13	***G1D***	2	1	2	1	0	2
14	***G1D***	2	0	2	1	2	2
15	***G1D***	3	3	3	3	3	3
16	***G1D***	3	3	3	3	3	3
17	***G1D***	3	3	3	3	2	2
18	***G1D***	1	1	1	1	0	0
19	***G1D***	3	3	3	3	3	3
20	***G1D***	1	1	1	1	1	1

APPENDIX-II

MICROHARDNESS VALUES

Ceram X mono

Spec#	GroupMat	TopSur	Botsurf
1	GI	89.75	83
2	GI	97	68.5
3	GI	89.5	80.25
4	GI	85.25	73
5	GI	95.75	82
6	GI	98.5	89.75
7	GI	107	85.5
8	GI	96	81.25
9	GI	104.75	80.25
10	GI	101.25	88.75
11	GI	95.25	64.25
12	GI	88.5	83.5
13	GI	90.5	74
14	GI	85.5	78
15	GI	90.5	74
16	GI	95.75	64.5

Premise

1	GII	86	75.5
2	GII	123	75.5
3	GII	90.5	84.5
4	GII	90	72.5
5	GII	91.5	62.5
6	GII	95	83.25
7	GII	85.25	80
8	GII	90.5	72.5
9	GII	87	70.5
10	GII	91.25	75
11	GII	95.25	80
12	GII	100.75	83.75
13	GII	86.5	76.5
14	GII	101.25	84.5
15	GII	88.75	77.5
16	GII	89.5	78.25

APPENDIX-III

MICROLEAKAGE

Kruskal-Wallis Test

Ranks

	GroupMat	N	Mean Rank
ScoreEMed	Group 1	20	35.38
	Group 2	20	27.75
	Group 3	20	35.95
	Group 4	20	62.93
	Total	80	
ScoreDMed	Group 1	20	44.68
	Group 2	20	35.80
	Group 3	20	22.80
	Group 4	20	58.73
	Total	80	

Test Statistics(a,b)

	ScoreEMed	ScoreDMed
Chi-Square	30.692	27.115
df	3	3
Asymp. Sig.	.000	.000

a Kruskal Wallis Test
b Grouping Variable: GroupMat

NPar Tests
Mann-Whitney Test

Ranks

	GroupMat	N	Mean Rank	Sum of Ranks
ScoreEMed	Group 1	20	22.45	449.00
	Group 2	20	18.55	371.00
	Total	40		
ScoreDMed	Group 1	20	22.88	457.50
	Group 2	20	18.13	362.50
	Total	40		

Test Statistics(b)

	ScoreEMed	ScoreDMed
Mann-Whitney U	161.000	152.500
Wilcoxon W	371.000	362.500
Z	-1.274	-1.337
Asymp. Sig. (2-tailed)	.203	.181
Exact Sig. [2*(1-tailed Sig.)]	.301(a)	.201(a)

a Not corrected for ties.
b Grouping Variable: GroupMat

NPar Tests
Mann-Whitney Test

Ranks

	GroupMat	N	Mean Rank	Sum of Ranks
ScoreEMed	Group 1	20	20.25	405.00
	Group 3	20	20.75	415.00
	Total	40		
ScoreDMed	Group 1	20	26.10	522.00
	Group 3	20	14.90	298.00
	Total	40		

Test Statistics(b)

	ScoreEMed	ScoreDMed
Mann-Whitney U	195.000	88.000
Wilcoxon W	405.000	298.000
Z	-.152	-3.122
Asymp. Sig. (2-tailed)	.879	.002
Exact Sig. [2*(1-tailed Sig.)]	.904(a)	.002(a)

a Not corrected for ties.
b Grouping Variable: GroupMat

NPar Tests
Mann-Whitney Test

Ranks

	GroupMat	N	Mean Rank	Sum of Ranks
ScoreEMed	Group 1	20	13.68	273.50
	Group 4	20	27.33	546.50
	Total	40		
ScoreDMed	Group 1	20	16.70	334.00
	Group 4	20	24.30	486.00
	Total	40		

Test Statistics(b)

	ScoreEMed	ScoreDMed
Mann-Whitney U	63.500	124.000
Wilcoxon W	273.500	334.000
Z	-3.841	-2.177
Asymp. Sig. (2-tailed)	.000	.029
Exact Sig. [2*(1-tailed Sig.)]	.000(a)	.040(a)

a Not corrected for ties.
b Grouping Variable: GroupMat

APPENDIX-IV

MICROHARDNESS

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
TopSur	Between Groups	8526.305	3	2842.102	32.520	.000
	Within Groups	5243.680	60	87.395		
	Total	13769.984	63			
Botsurf	Between Groups	20470.414	3	6823.471	66.696	.000
	Within Groups	6138.445	60	102.307		
	Total	26608.859	63			

Post Hoc Tests

Multiple Comparisons
Tukey HSD

Dependent Variable	(I) GroupMat	(J) GroupMat	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
TopSur	CeramX	Premise	1.17188	3.30520	.985	-7.5622	9.9059
		Grandio	-18.78125(*)	3.30520	.000	-27.5153	-10.0472
		Z100	-25.26563(*)	3.30520	.000	-33.9997	-16.5316
	Premise	CeramX	-1.17188	3.30520	.985	-9.9059	7.5622
		Grandio	-19.95313(*)	3.30520	.000	-28.6872	-11.2191
		Z100	-26.43750(*)	3.30520	.000	-35.1716	-17.7034
	Grandio	CeramX	18.78125(*)	3.30520	.000	10.0472	27.5153
		Premise	19.95313(*)	3.30520	.000	11.2191	28.6872
		Z100	-6.48438	3.30520	.214	-15.2184	2.2497
	Z100	CeramX	25.26563(*)	3.30520	.000	16.5316	33.9997
		Premise	26.43750(*)	3.30520	.000	17.7034	35.1716
		Grandio	6.48438	3.30520	.214	-2.2497	15.2184
Botsurf	CeramX	Premise	1.141	3.576	.989	-8.31	10.59
		Grandio	-30.672(*)	3.576	.000	-40.12	-21.22
		Z100	-38.781(*)	3.576	.000	-48.23	-29.33
	Premise	CeramX	-1.141	3.576	.989	-10.59	8.31

		Grandio	-31.813(*)	3.576	.000	-41.26	-22.36
		Z100	-39.922(*)	3.576	.000	-49.37	-30.47
	Grandio	CeramX	30.672(*)	3.576	.000	21.22	40.12
		Premise	31.813(*)	3.576	.000	22.36	41.26
		Z100	-8.109	3.576	.117	-17.56	1.34
	Z100	CeramX	38.781(*)	3.576	.000	29.33	48.23
		Premise	39.922(*)	3.576	.000	30.47	49.37
		Grandio	8.109	3.576	.117	-1.34	17.56
* The mean difference is significant at the .05 level.							

Homogeneous Subsets

TopSur Tukey HSD			
GroupMat	N	Subset for alpha = .05	
		1	2
Premise	16	93.2500	
CeramX	16	94.4219	
Grandio	16		113.2031
Z100	16		119.6875
Sig.		.985	.214
Means for groups in homogeneous subsets are displayed.			
a Uses Harmonic Mean Sample Size = 16.000.			

Botsurf Tukey HSD			
GroupMat	N	Subset for alpha = .05	
		1	2
Premise	16	77.02	
CeramX	16	78.16	
Grandio	16		108.83
Z100	16		116.94
Sig.		.989	.117
Means for groups in homogeneous subsets are displayed.			
a Uses Harmonic Mean Sample Size = 16.000.			