# CHAPTER ONE

## **INTRODUCTION AND OBJECTIVES OF STUDY**

# **1.1 Introduction**

The increased demand for aesthetics and concerns about toxic and allergic reactions to dental alloys has led to the development of all-ceramic crowns because of their highly aesthetic results and biocompatibility (Snyder and Hogg, 2005; Cha et al., 2001).

Many alternatives have been suggested for restoring lost tooth structure in the posterior region. In the 20<sup>th</sup> century, porcelain-fused-to-metal (PFM) restorations have accounted for a significant proportion of posterior tooth restorations (Okutan et al., 2006).

The patient and clinician alike have an interest in aesthetic restorations that are not limited to just the anterior teeth. Aside from poor aesthetics, metal-based crowns have some other disadvantages such as galvanic and corrosive side effects (Moller, 2002) as well as causing gingival discoloration (Christensen, 1994). As a result, posterior toothcolored adhesive restorative techniques have grown considerably over the last decade (Magne, 2006). All-ceramic crowns were routinely placed not only in the anterior aesthetic zone but also in the posterior where they were subjected to greater occlusal forces and stress from cyclic loading (Snyder and Hogg, 2005).

As the demand for more natural-looking crowns has increased, dentists and porcelain manufacturers have investigated a number of methods to help reinforce ceramics with the goal of fabricating an all-ceramic restoration that delivers excellent aesthetics and good biocompatibility. In addition, these restorations need to have sufficient strength to allow its use anywhere in the mouth (Leite el al., 2005; Yilmaz et al., 2007).

Silica-based ceramics such as feldspathic porcelain and glass ceramic are frequently used to veneer metal frameworks or high-strength ceramic copings for all-ceramic restorations (Andersson and Oden, 1993). Their excellent aesthetic properties make them the material of choice for ceramic laminate veneers and inlays/onlays (McLaughlin, 1998; and Blatz, 2002). Inspite of the inherent brittleness and limited flexural strength of silica-based ceramics, final adhesive cementation with composite increases the fracture resistance of the ceramic restoration (Pagniano et al., 2005).

Leucite-reinforced feldspathic porcelain (egs: IPS Empress; Ivoclar-Vivadent, Schaan, Liechtenstein) achieves significantly higher fracture strength and provides the restorative team with the ability to fabricate full-coverage all-ceramic restorations for both anterior and posterior teeth if resin bonding techniques are properly applied (Blatz, 2002).

Several new all-ceramic systems, which offer comparable stability to PFM, good aesthetics, and simplified fabrication procedures, have been introduced (Okutan et al., 2006). Recently, new dental materials and techniques have been introduced to fabricate aesthetic ceramic restorations with improved strength and marginal adaptation. This becomes more important for posterior areas in the mouth, where the forces are much higher than for the anterior region and can reach 522 N in the average individual (Bakke et al., 1990; Pallis et al., 2004).

In order to provide satisfactory posterior all-ceramic restorations, strong alumina cores have been produced. Turkom-Cera<sup>TM</sup> All-ceramic material (Turkom-Ceramic (M), Puchong, Malaysia), Procera AllCeram (Nobel Biocare, Göteborg, Sweden) and In-Ceram (Vita Zahnfabrik, Bad Sackingen, Germany) are three ceramic systems that incorporate a high alumina core. These cores differ in their manufacturing process and chemical structure. Procera AllCeram contains a densely sintered alumina core (> 99.9%), whereas Turkom-Cera<sup>TM</sup> and In-Ceram are made of a high alumina core which is subsequently crystal hardened or glass infiltrated. The alumina content of the Turkom-Cera<sup>TM</sup> and In-Ceram cores is 99.98% and 80-82% respectively (Turkom-Cera booklet, 2007; Andersson and Oden, 1993; Haselton et al., 2000; Conrad et al., 2007). However, alumina cores tend to be opaque and require the use of veneer porcelain to mask the core and provide the desired contours (Webber et al., 2003).

The Procera AllCeram system was first described by Andersson & Oden in 1993. The die for the core is scanned using CAD/CAM technology and the data is sent via the internet to the factory in Sweden where a second (enlarged) die is milled. High purity aluminium oxide is then compacted onto the enlarged die and the external shape of the coping is milled before sintering at 1600 °C. The coping is then sent back to the dental lab for veneering with aluminous porcelain in the traditional way.

In-Ceram system has been commercially available for around 20 years and the manufacturer claims that it is suitable for anterior and posterior crowns and short span bridgework. The conventional In-Ceram uses the slip casting technique. Through this technique, the alumina slip is applied onto a refractory die and sintered in a special furnace at 1120 °C for 10 hours. This produces a porous structure of alumina particles,

which is infiltrated by capillary action with lanthanum oxide-based glass in a second firing at 1100 °C for 4 hours (Yeo et al., 2003).

The Turkom-Cera<sup>™</sup> system uses a conventional way to fabricate all-ceramic crowns. This system based on the plastic foil technique. The fabrication of the Turkom-Cera core requires two steps. In the first step, the aluminium oxide gel is applied onto the stone die, covered with the red plastic foil, and sintered in an ordinary furnace at 1150 °C for 5 minutes. In the second step, the crystal powder is applied onto the pre-sintered aluminium oxide core and fired in the same furnace for 30 minutes. The process of making Turkom-Cera core does not take more than one hour (Appendix). However, approximately 14 hours (10 hours of sintering and 4 hours of glass infiltration) are needed to make the In-Ceram core (Yeo et al., 2003).

According to Diegoa et al., (2007) the particle size of the In-Ceram glass powder varies from 1 to 200  $\mu$ m. This large particles size distribution will make the glass powder infiltration into the porous pre-sintered ceramic difficult during the glass infiltration process. This characteristic of the In-Ceram glass powder justifies the high infiltration time (3 to 6 hours) required during the fabrication of In-Ceram dental crowns. Therefore, it can be expected that a reduction of the particles size of the Turkom-Cera crystal powder, facilitate the infiltration of theses crystals during melting process.

Aluminium oxide serves as reinforcement of the glassy matrix, comparable to leucite crystals. In general, ceramics containing less than 15 wt% silica are not regarded as silica-based or silicate ceramics. In high-strength alumina or zirconia based ceramics, the aluminium oxide or zirconium oxide is not a reinforcement; it forms the matrix (Andersson and Oden, 1993).

The clinical success of ceramic restorations depends on the cementation process, which varies according to the composition of the ceramic material (Borges et al. 2003). In dentistry, two approaches are possible to establishment a reliable and durable bond between dental ceramic and resin luting cements: micromechanical bonding to porosities created from hydrofluoric acid etching and/or sandblasting and pure chemical bond by using silane coupling agent (Matinlinna and Vallittu, 2007a).

Acid etching of conventional silica-based dental ceramic materials has been used primarily as a means of developing controlled surface microroughness to aid in the retention of the cementation medium to the ceramic surface (Saraçoğlu et al., 2004; Filho et al., 2004).

Acid etchants used for silica-based dental ceramics do not sufficiently roughen the surface of aluminium oxide ceramics (Awliya et al., 1998; Soares et al., 2005). Airborne particle abrasion with Al<sub>2</sub>O<sub>3</sub> is effective and practical for creating an activated and roughened surface on glass-infiltrated alumina ceramic (Kern and Thompson, 1994; Borges et al., 2003; Derand et al., 2006). Densely-sintered high-purity aluminium oxide ceramic does not contain any silica. Similar to glass-infiltrated alumina ceramic, the surface of pure aluminium oxide ceramic cannot be altered through conventional acid etching.

Awliya et al., (1998), in their study found that airborne particle abrasion with a micro abrader (50  $\mu$ m Al<sub>2</sub>O<sub>3</sub> at 2.5 bar) revealed significantly higher bond strengths than acid etching with either 9.6% HF or 37% phosphoric acid, grinding with a diamond, or no treatment (control).

The presence of surface flaws created by acid etching or airborne-particle abrasion is often the site of fracture initiation and, therefore, weakens ceramic materials. However, it has been shown that resin luting agents proven to provide durable bonds, have the ability to heal minor surface flaws and, therefore, significantly strengthen ceramic materials (Blatz, 2002; Burke et al., 2002; Blatz et al., 2004).

Slip casting is technique derived from industrial technology and adopted for dental use (Probster and Diehl, 1992). The slip cast alumina is first partially sintered in a furnace, producing a porous framework that is then infiltrated with liquid glass in a second firing process. Crack propagation during failure is limited because of the densely packed alumina particles. Glass infiltration eliminates almost all porosities, which are potential sites for crack initiation. The difference in the coefficients of thermal expansion between alumina and glass produces compressive stresses at the alumina-glass interface that further enhances strength (Xiao-ping et al., 2002).

Many factors may be associated with crack initiation and propagation within a dental ceramic. These include the shape and thickness of the restoration, microstructural inhomogeneities, size and distribution of surface flaws, residual processing stresses, the magnitude, direction, and frequency of the applied load, the restoration-cement interfacial defects, the elastic modulus of the restoration components, and environmental effects (Thompson et al., 1994; Kelly, 1995).

Wagner & Chu (1996), in their study on biaxial strength of three all ceramic materials, found a significant difference between Procera AllCeram (687 MPa), In-Ceram (352 MPa) and Empress ceramic core (134 MPa). They also found that the flexural strength of In-Ceram core material varied greatly, whereas the Procera core material was found to be more uniform. Similarly, previous studies have indicated that the Procera core (469 MPa) has a higher failure stress than In-Ceram core (301 MPa) (Zeng et al., 1998). However Chai et al., (2000), found no difference in fracture strength between Procera and In-Ceram crowns that were cemented with resin cement (Panavia F), which suggests that the choice of luting agent may affect the strength of the ceramic restoration.

Resin bonding of ceramic restorations to the supporting tooth structure increases retention (EL-Mowafy, 2001), marginal adaptation (Sorensen et al., 1991) and fracture resistance of the restoration (Burke, 1996; Pagniano et al., 2005).

Strub & Beschnidt (1998), in their in vitro study on extracted incisors showed that the fracture strengths of five different types of all-ceramic crowns to be similar to that of metal-ceramic crowns when cemented with resin cement. However, ageing through chewing and thermocycling devices have resulted in a significant decrease in fracture strength, suggesting that prolonged exposure of the cement to simulated oral conditions may lead to the deterioration of its integrity. Cement breakdown may lead to ingress of fluids and microorganisms along the tooth-restoration interface, causing marginal discoloration, pulpal irritation, and secondary caries.

The role of cement in fixed prosthodontic is to preserve the integrity and health of the prepared tooth structure by providing a seal against microleakage at tooth crown interface (Toman et al., 2007). Different types of luting agents vary considerably in solubility, strength, and ability to adhere to tooth structure. Thompson et al., (1998) cited that the clinical failure rate for resin bonded ceramic restorations had been found to be lower than when traditional cements were used.

Gu & Kern (2003), have evaluated microleakage of IPS Empress-2 all-ceramic crowns cemented with three different types of luting cements. They concluded that the adhesive composite resin cement showed an excellent ability to minimize leakage of all-ceramic crowns and was far superior to zinc-phosphate and compomer luting cements. The IPS Empress-2 crowns had extensive microleakage when zinc phosphate cement was used.

Adhesive composite resin luting systems are recommended for cementation of allceramic systems (Blatz et al., 2003a, Hill, 2007; Pegoraro et al., 2007). It has been shown that full coverage densely sintered alumina crowns can achieve long-term clinical success with conventional luting agents (Oden et al., 1998; Odman and Andersson, 2001). However, adhesive cementation may be beneficial for high-strength ceramic full coverage restorations, especially in situations of compromised retention or high occlusal load.

Prior to time-consuming and costly clinical investigations, in vitro studies should be carried out to evaluate the clinically relevant properties for newly developed dental materials and products (Gu and Kern, 2003). There are no in vivo or in vitro studies up to date on the clinical and mechanical performance of Turkom-Cera<sup>TM</sup> all-ceramic material. Different luting agents are available for luting all-ceramic materials. However,

the effect of these materials on the strength of the new all-ceramic material Turkom-Cera<sup>TM</sup> has also not investigated.

Durable bonding to fixed prosthodontic restorations is desirable; however, no information is available on the strength of the bond between different cements and the new fixed prosthodontic all-ceramic restorative material Turkom-Cera<sup>TM</sup>.

A new all-ceramic material Turkom-Cera<sup>™</sup> (Turkom-Ceramic (M) Sdn. Bhd., Puchong, Malaysia), particularly with aluminium oxide, has been introduced in an attempt to provide high-quality, cost effective copings and to improve clinical success with all ceramic restorations. Independent studies of basic comparative data are necessary to characterize this new material in relation to its mechanical properties.

Therefore, this study was aimed to evaluate the clinical performance and mechanical properties of Turkom-Cera<sup>TM</sup> all-ceramic material.

### **Objectives of the study**

- To compare the bi-axial flexural strength and hardness of Turkom-Cera, In-Ceram and Vitadur-N all-ceramic systems.
- 2. To determine the shear bond strength of Turkom-Cera luted with different cements.
- To compare the effect of various surface treatment on the shear bond strength of Turkom-Cera when luted with resin-based cement.
- To determine the occlusal fracture resistance of Turkom-Cera copings compared to In-Ceram and Procera AllCeram copings.

- 5. To compare the effect of different luting cements on the occlusal fracture resistance of Turkom-Cera copings.
- 6. To determine the occlusal fracture resistance of Turkom-Cera copings compared to In-Ceram and Procera All-Ceram copings cemented to natural teeth.
- 7. To examine the effect of finish line design and artificial ageing on the occlusal fracture resistance of Turkom-Cera copings.
- 8. To examine the marginal adaptation of Turkom-Cera, In-Ceram and Procera AllCeram copings.
- To determine the influence of the finish line on the marginal adaptation of Turkom-Cera copings.
- To evaluate the clinical performance of Turkom-Cera all-ceramic crowns over a two year period.

CHAPTER TWO

**REVIEW OF LITERATURE** 

# 2.1 Background

Since the first use of porcelain to make a complete denture by Alexis Duchateau in 1774, numerous dental porcelain compositions have been developed. One of the most serious drawbacks with the early dental porcelains was their lack of strength and large shrinkage, which limited their use (McCabe and Walls, 2008).

Dental ceramics were first used in dentistry in the late 1700s. The term ceramic is defined as any product made essentially from a nonmetallic material by firing at a high temperature to achieve desirable properties. The term porcelain refers to a family of ceramic materials composed essentially of kaolin, quartz, and feldspar, also fired at high temperature. Dental ceramics for ceramic-metal restorations belong to this family and are commonly referred to as dental porcelains (Craig, 2002).

Presently, metal-ceramic crowns are the most common complete coverage system. With proper tooth preparation, the retention and strength of metal-ceramic crowns are superior to veneer restorations. There are, however, some disadvantages to metalceramics. Depth of translucency can be achieved only if there is significant tooth reduction. Compromised aesthetics because of a gray line at the gingival margin is another drawback.

In 1808, individually formed porcelain teeth that contain embedded platinum pins were introduced in Paris by Giuseppangelo Fonzi. Although probably not involving feldspathic porcelains, the enamelling of metal denture bases was described in 1723 by Pierre Fauchard who was credited with recognizing the potential of porcelain enamels and initiating research with porcelains to imitate colour of the teeth and gingival tissues (Jones, 1985). The first all-ceramic crowns introduced by Land in 1903 were relatively weak materials with limited clinical use (Rosenblum and Schulman, 1997; Burke et al., 2002). Porcelain jacket crowns (alumina-reinforced feldspathic core) were first introduced in 1965 by McLean and Hughes by means of infiltration of dental porcelain with aluminium oxide. They consisted of feldspathic or aluminous porcelain baked on a thin platinum foil. However, porcelain jacket crowns were limited to anterior teeth because of their low strength (Raigrodski, 2004). A core porcelain was made containing 50% (by weight) fused alumina crystals to which a matched-expansion veneer porcelain was baked. The flexural strength is approximately 125 MPa (McLean and Kedge, 1987). In the forthcoming years, research resulted in different methods aimed to increase the fracture resistance, and stronger dental ceramics was developed and manufactured for the market.

The use of all-ceramic restorations has increased in recent years. The reason is that allceramic systems can be more aesthetic than metal-ceramic systems because of the lack of a metal core. A number of all-ceramic techniques have been introduced in restorative dentistry since the early 1980s. The first was a castable glass-ceramic system in which the restoration was cast using the lost-wax technique and later heat-treated to promote its transformation into a glass-ceramic. This castable system was later abandoned due to processing difficulties and the high incidence of fractures (Denry, 1996; Craig, 2002).

The injection-moulded, IPS Empress 2, is a lithium disilicate-reinforced glass ceramic that is primarily a glass matrix with crystalline lithium disilicate that strengthens the ceramic without significantly diminishing its translucency and aesthetics. This core material possesses a high flexural strength in the range of 352 to 452 MPa (Albakry et al., 2003).

Two other contemporary all- ceramic approaches have emerged with greater promise. They are Procera AllCeram, which has a reported 96.9 % success rate (Odén et al., 1998), and In-Ceram aluminium, which has a reported 98.4 % success rate (Scotti et al., 1995). Procera AllCeram cores are 99.9 % aluminium oxide, whereas In-Ceram aluminium copings are 80-82 % sintered aluminium oxide saturated with lanthanum glass (Conrad et al., 2007).

Procera All Ceram cores can only be fabricated with a computer-aided design/ computer-aided manufacturing (CAD/CAM) technique, whereas In-Ceram aluminium copings can be built up on special plaster dies or milled by a CAD/CAM technique.

An increasing interest in all-ceramic restorations has followed improvements in strength, aesthetics and ease of processing. Such advances include introduction of Turkom-Cera<sup>™</sup> all-ceramic material for dental use. Turkom-Ceramic SDN. BHD. set out to produce a pure aluminium oxide (99.98 %) all-ceramic material, which would achieve greater strength than other all-ceramic materials in the market. This material would also be a perfect compliment to all-ceramic materials. However, in vitro and in vivo investigations of new all-ceramic systems should be undertaken before introducing them into routine clinical use.

# 2.2 Classification of all-ceramic systems

The following general types of all-ceramic systems are currently available:

### 2.2.1 Conventional Powder-Slurry Ceramics

Stronger all-ceramic systems were developed by increasing the crystalline content of conventional feldspathic porcelain. These products are supplied as powders to which the technician adds water to produce a slurry. The ceramic mass is built up in layers on a platinum foil or a refractory die material to form the restoration. The powders are available in various shades and translucencies and are supplied with characterizing stains and glazes (Kelly, 2004).

Vitadur N was one of the first feldspar ceramic systems used for the fabrication of veneers (McLean and Sced, 1987; Rucker et al., 1990; Hui et al., 1991). According to the technique described by McLean & Sced (1987), a platinum foil of 0.02 mm is placed and burnished to adapt to the die. The ceramic mass is then layered onto the foil. During sintering, microporosities are formed on the surface of the veneer, which decrease the strength of the restoration. Thus, the clinical use of this material is nowadays very limited (Raigrodski, 2004).

Optec HSP is leucite reinforced feldspar ceramic that condenses like an alumina ceramic and is sintered like a traditional feldspar ceramic. The leucite concentration was reported to be 50.6 wt% (Kelly et al., 1996). Optec HSP has greater strength than conventional feldspathic porcelain because it contains an increased amount of leucite. The leucite and glassy components are fused together during the baking process at 1020°-1035°C. The buildup and contouring of the crown is accomplished using the powder-slurry technique on a special semipermeable die material (Rosenblum and Schulman, 1997).

### 2.2.2 Cast glass and polycrystalline ceramics

Castable ceramics are supplied as solid ceramic ingots, which are used for fabrication of cores or full-contour restorations using a lost wax and centrifugal casting technique. Generally, one shade of material is available, which is covered by conventional feldspathic porcelain or is stained to obtain proper shading and characterization of the final restoration (Qualtrough and Piddock, 2002; McCabe and Walls, 2008).

Dicor was released to the dental community in 1982, and was the first commercially available castable ceramic material for dental use. It is a polycrystalline glass-ceramic material, initially formed as a glass and subsequently heat treated under controlled crystallization conditions to produce a glass-ceramic material. The crystalline phase of Dicor comprised tetrasilicic fluoromica ( $K_2Mg_5$  Si<sub>8</sub> O<sub>20</sub> F<sub>4</sub>), which provides fracture resistance and strength from the generation of compressive stress around the crystals (Giordano, 1996; Krishna et al., 2009). Dicor is highly translucent, and this may be due to the fineness of crystals formed and the fact that the refractive index of the glass is close to that of precipitated crystals (Tzeng and Duh, 1993; Heffernan et al., 2002a; Heffernan et al., 2002b). However, its low strength value limits its use for single crowns (Giordano, 1996, McCabe and Walls, 2008).

Recently, another core ceramic, yttrium tetragonal zirconia polycrystals (Y-TZP), has been introduced to dentistry. Yttrium oxide is added to pure zirconium oxide at room temperature to generate a multiphase material known as partially stabilized zirconia. These restorations may be processed using casting procedures or may be milled from monolithic blocks of partially or fully sintered material (Raigrodski et al., 2004 and McCabe and Walls, 2008). The high values of flexural strength (900–1200 MPa) and fracture toughness (9-10 MPa) reported for these Y-TZP ceramics are due to their polycrystalline structure and to a process of transformation toughening caused by changes in crystal structure initiated by internal stresses. The transformations result in localized increases of 3 % to 5 % in volume which can cause compressive stresses to be set up around and at the tip of the cracks. This will help to reduce the likelihood of further propagation of the cracks (Christel et al., 1989; Raigrodski, 2004; McCabe and Walls, 2008).

# 2.2.3 Pressable Ceramics

Pressable ceramics are also supplied as ceramic ingot products. These products are melted at high temperatures (1150°C) and pressed into a mould created using the lost-wax technique. The pressed form can be made to full contour, or can be used as a substrate for conventional feldspathic porcelain buildup (Rosenblum and Schulman, 1997).

IPS Empress and IPS Empress 2 are two types of pressable ceramics with different chemical composition, crystallinity, strength and opacity. IPS Empress is a leucite reinforced glass-ceramic supplied in ingot form. The leucite (KAlSi<sub>2</sub>O<sub>6</sub>) crystals serve to reinforce the glassy matrix and prevent crack propagation. Although the crystals serve to strengthen the ceramic core, the more crystallinity it presents the more opaque is the core. With IPS Empress, 30-40 % crystal content can be introduced before the aesthetic of the core and resulting restoration are compromised. The crystalline content has been limited to provide strength improvement over conventional feldspathic porcelain without a change in the level of translucency that would compromise aesthetics (Heffernan et al., 2002a; and Heffernan et al., 2002b). Therefore, the IPS Empress system is designed for the fabrication of single unit crowns, inlays, onlays and veneers (Raigrodski, 2004).

The IPS Empress 2 is a lithium disilicate ( $Li_2Si_2O_5$ ) reinforced glass-ceramic. In IPS Empress 2, controlled crystallization production of a lithium disilicate glass ceramic enable the creation of a 60 % crystal content (by volume) without loss of translucency, as the refractive index of the crystals is similar to that of the glassy matrix (Qualtrough and Piddock, 2002). The increased crystalline content forms a tighter, interlocking structure that significantly increases the strength and fracture toughness and only

slightly increases the opacity of the material (Heffernan et al., 2002a; and Heffernan et al., 2002b). Albakry et al., (2004), in their study on the biaxial flexural strength of two recycled pressable glass ceramics found that IPS Empress can withstand 148 MPa, whereas IPS Empress 2 can withstand 340 MPa. Therefore, this material is recommended for the fabrication of fixed partial dentures in the anterior or premolar areas (McCabe and Walls, 2008).

# 2.2.4 Glass Infiltrated ceramics

The high failure rates for molar crowns, the need for improved fracture strength of allceramic restorations, and the potential to extend all-ceramic restorations to fixed prostheses have led to the development of ceramics with increased alumina content (McLean and Hughes, 1965; Thompson and Rekow, 2004). The aluminium oxide serves as reinforcement of the glassy matrix, comparable to leucite crystals.

High-strength aluminium-oxide ceramics are indicated in all areas of the mouth for copings and frameworks of full-coverage crowns and fixed partial dentures. Such copings and frameworks are veneered with feldspathic porcelain to combine superior physical strength with optimal aesthetic properties (Blatz, 2002).

In-Ceram Alumina (Vita Zahnfabrik, Bad Säckingen, Germany) and Turkom-Cera<sup>™</sup> Fused Alumina (Turkom-Ceramic (M), Puchong, Selangor, Malaysia) are two representatives of this group. In-Ceram consists of two components: an alumina powder, which is fabricated into a porous substrate, and glass, which is infiltrated at high temperature into the porous substrate. Turkom-Cera<sup>™</sup> consists of two components: an alumina gel, which is sintered into a porous substrate, and crystal powder, which is used for the hardening process of the porous substrate. The infiltrated/hardened ceramic is then veneered, using the conventional feldspathic porcelain technique.

### 2.2.4.1 In-Ceram

In-Ceram alumina (Vita Zahnfabrik, Bad Säckingen, Germany) incorporates a drysintered aluminium oxide core that is infused with molten glass. The all-ceramic core offers a flexural strength of 450 MPa after glass infiltration and is veneered with feldspathic porcelain for enhanced aesthetics (Seghi and Sorensen, 1995).

In-Ceram Spinell is a glass-infiltrated spinel ceramic, a composition containing aluminium oxide and magnesium oxide (MgAl<sub>2</sub>O<sub>4</sub>), and is slightly weaker than In-Ceram Alumina (Seghi and Sorensen, 1995; Conrad et al., 2007). It offers improved optical properties and translucency partly because of the crystalline habit of the spinel and lower index of refraction compared with alumina (Hwang and Yang, 2001). It is more suited for aesthetically critical areas and inlays (Kelly et al., 1996; van Dijken, 1999). Nevertheless, some strength has been sacrificed for the translucency. An in vitro study by Seghi & Sorensen (1995), reported that the flexural strength of spinel was about 75% of the alumina core.

The sintered In-Ceram core is porous and made from fine insoluble particles, either aluminium oxide or spinel, that are mixed with water to form a suspension referred to as slip. The slip mass is placed on a refractory die and heat-treated at 1120°C for 10 hours to produce the opaque, porous core. After choosing an appropriate shade, the porous, partially sintered alumina (or spinel) core is then infiltrated with a low-viscosity glass (lanthanum glass) by baking again at 1120°C for 4 hours to yield a ceramic coping of high density and strength. The finished In-Ceram coping contains at least 70 % aluminum oxide or spinell, and is currently one of the strongest ceramics available with a flexural strength between 236 and 446 MPa depending on the test method used (Seghi and Sorensen, 1995, Giordano et al., 1995). The densely stacked particles and the different thermal expansion coefficients of glass and alumina (or spinel) contribute to the high strength of the material. The In-Ceram material is then veneered with a feldspathic ceramic for the final aesthetics (Conrad et al., 2007).

In an in vitro study by Hwang & Yang (2001), they found that In-Ceram Alumina showed a significantly higher fracture strength (876.19 N) than In-Ceram Spinell (687.90 N). The higher fracture strength was a result of the higher flexural strength of the alumina core.

In-Ceram alumina has been used extensively for a number of years with excellent success. In a retrospective study for 6 years, which evaluated 546 In-Ceram alumina anterior and posterior crowns, it was reported that the overall success rate was 99.1%. The success to failure ratio for anterior all-ceramic crowns was 98.9% to 1.1%; the posterior crown ratio was 99.2% to 0.8% (Segal, 2001).

Copy-milling technologies are used for making a Celay In-Ceram crown (Rinke et al., 1995). The copy-milled Celay In-Ceram crowns have shown a biologically acceptable marginal gap (Beschnidt and Strub, 1999; Groten et al., 1997). This system has two advantages over conventional In-Ceram. The Celay In-Ceram system has higher fracture strength (about 10%) than slip casting In-Ceram (Hwang and Yang, 2001). In addition, the Celay system uses a pre-sintered alumina blanks, eliminating the 10-hour sintering procedure that is necessary for the conventional technique. The Celay core

requires 40 minutes for glass infiltration, and therefore this system saves time (Rinke et al., 1995; Yeo et al., 2003).

### 2.2.4.2 Turkom-Cera<sup>™</sup> Fused Alumina

An increasing interest in all-ceramic restorations has followed improvements in strength, aesthetics and ease of processing. Such advances include introduction of Turkom-Cera<sup>TM</sup> (Turkom-Ceramic (M) SDN. BHD, Puchong, Selangor, Malaysia) all-ceramic material for dental use. Turkom-Ceramic (M) SDN. BHD. set out to produce a pure aluminium oxide (99.98 %) all-ceramic material, which would achieve greater strength than other all-ceramic materials in the market. Turkom-Cera<sup>TM</sup> Fused Alumina incorporates a pure aluminium oxide core that is infused with molten crystals. The Turkom-Cera<sup>TM</sup> all-ceramic system comprises a conventional procedure for the fabrication of high strength aluminium oxide all-ceramic inlays, onlays, crowns and bridges for anterior and posterior mouth applications. The framework ceramic consists of an aluminium oxide core supplemented by a specially designed veneer ceramic.

According to the manufacturer, the Turkom-Cera alumina gel is a pure aluminium oxide (99.9%) whitish in colour with a bulk density, boiling point and melting point of 0.17 g/ml, 2800 °C, and 2050 °C, respectively. The Turkom-Cera crystal powder is a lanthanum oxide-based glass which is white or white to yellowish in colour with a bulk density of 0.27 g/ml and a melting point of more than 1000 °C. The finished Turkom-Cera core is translucent and during porcelain build-up there is no need to use the opaque, according to the manufacturer's recommendation. The detailed step-by-step fabrications of dental restorations using Turkom-Cera<sup>TM</sup> system are in the Appendix I.

# 2.2.5 Machinable (CAD/CAM) Ceramics

Machinable ceramics are supplied as ceramic ingots in various shades. Nowadays, machining has become a viable option as a forming method in the fabrication of all-ceramic restorations. Both CAD/CAM systems and the precision copy-milling machine are commercially available (Kelly et al., 1996). The ceramic ingots do not require further high-temperature processing. They are placed in a machining apparatus to produce the desired contours. The machined restoration may be stained and glazed to obtain the desired characterization.

The term 'CAD/CAM' in dental technology is currently used as a synonym for prostheses produced by milling technology. This is not entirely correct. CAD-CAM is the abbreviation for computer-aided design/computer aided manufacturing. Therefore, the term 'CAD/ CAM' does not provide any information on the method of fabrication (Beuer et al., 2008a).

All CAD/CAM systems utilise a process chain consisting of scanning, designing and milling phases. Depending on the location of the components of the CAD/CAM systems, in dentistry three different production concepts are available: chairside production, laboratory production, and centralised fabrication in a production centre (Freedman et al., 2007; Beuer et al., 2008a).

### 2.2.5.1 CAD/CAM systems

The introduction of CAD/CAM systems to restorative dentistry represents a major technological breakthrough. This technology is implemented in the Cerec system (Sirona, Bensheim, Germany), which was developed in 1984. This system is a computer aided design/computer aided manufacture (CAD/CAM) system designed for the

fabrication of indirect restorations (Mörmann and Bindl, 2000). The system mills ceramic restorations from industrial blocks of ceramic material which are prefabricated under optimum and controlled conditions (Martin and Jedynakiewicz, 1999).

Since its development in 1984, the Cerec system has undergone several technical modifications. The first generation system, Cerec 1, was designed for chairside fabrication of intracoronal restorations such as inlays, onlays, and/or veneers, whereas the Cerec 2 was introduced in 1994 with redesigned software and hardware to fabricate complete crowns in addition to intracoronal restorations. In 2003, Cerec 3 made its debut and has several improvements over the Cerec 2 system. These improvements include: an enhanced intraoral optical camera able to reproduce finer detail and depth of scale and improved software capable of recording the preparation much faster. Additionally, the Cerec 3 system allows more flexible and more true-to-detail grinding than the Cerec 2, which in turn should lead to a better fitting crown with improved occlusal morphology and design (Apholt et al., 2001; Qualtrough and Piddock, 2002; Giordano, 2006). It is possible with this system to generate a restoration without taking conventional impression, to fabricate a temporary restoration, and to eliminate any laboratory induced errors. The entire procedure can be performed in one appointment (David and LoPresti, 1994).

Technically, the cavity is coated with a light-reflecting powder and mapped using a mini hand-held 3-dimensional intraoral video camera. This procedure is called optical impression. The obtained information is fed to a computer that stores the 3-dimensional pattern depicted on the screen. The video display serves as a format for the necessary manual construction via an electrical signal. Then the integrated microprocessor develops the final 3-dimensional restoration from the 2-dimensional construction. After

that the electronic information is transferred numerically to the linked miniature 3-axis milling device. The milling unit driven by a water turbine device generates a restoration from the standard ceramic block. The blocks are then cast and cerammed (Leinfelder et al., 1989; David and LoPresti, 1994; Mörmann and Bindl, 2000; Freedman et al., 2007).

#### 2.2.5.1.1 The ceramic materials available for the Cerec System

A wide range of block materials are available for use with CAD/CAM systems to produce dental restorations. These blocks are fabricated continuously in a reproducible manner. The reliability of the block materials may be enhanced due to the reproducibility of the manufacturing process, which is a constant. Conventionally processed restorations fabricated by hand are also with high quality, but this may affect their reliability with respect to mechanical and aesthetic properties. The electron micrographs demonstrated numerous pores in the cross-sections of the pressed and hand-built restorations compared to CAD/CAM made block materials (Giordano, 2006; Beuer et al., 2008a).

Vitablocs Mark I (Vita Zahnfabrik, Bad Säckingen, Germany) is a feldspathic porcelain and was the first composition used with the Cerec system. It is similar in composition, strength and wear properties to feldspathic porcelain used for porcelain fused to metal restorations (Fasbinder, 2002). Vitablocs Mark II (Vita Zahnfabrik, Bad Säckingen, Germany) was introduced in 1991 to replace the original Vitablocs Mark I material, which had been developed for the Cerec-1 system. Vitablocs Mark II is a fine-grained feldspathic porcelain with a homogeneous structure. The average particle size is 4  $\mu$ m (Conrad et al., 2007). Dicor machinable glass ceramic (MGC, Dentsply Int. Inc., York, PA, USA) is composed of fluorosilicic mica crystals in a glass matrix (Rosenblum and Schulman, 1997; Krishna et al., 2009). The low fracture toughness of these materials limits the scope of restorative procedures for which they can be safely used. Currently, only inlays and conservative onlays are recommended for use with Vitablocs Mark II and Dicor MGC materials with the Cerec system (Thompson and Heymann, 1996).

ProCAD (Ivoclar Vivadent, Schaan, Lichtenstein) blocks were introduced in 1998 for the Cerec system. ProCAD is a leucite reinforced ceramic similar to Empress I, but with finer particle size. The ProCAD material can be etched to create a retentive surface for adhesive luting with resin cement (Fasbinder, 2002).

Chen et al., (1999) evaluated the fracture resistance of CAD/CAM restorations and pressed ceramic crowns. All-ceramic crowns of Vitablocs Mark II, ProCAD and Empress I were cemented on composite resin dies with Variolink II and loaded to fracture. The ProCAD crowns were not significantly different in fracture load to the Empress I crowns but were significantly stronger than the Vitablocs Mark II crowns. Although there have been few published clinical studies to date involving ProCAD. It is similar to Empress 1 in physical structure, so a similar clinical performance is anticipated.

Although in vitro studies are useful in initial trial evaluation of restorative materials, a clinical trial is the best evaluation of restorative longevity. For Cerec materials, the greatest numbers of clinical studies published were conducted on Vitablocs Mark II restorations. The material was introduced with the development of the Cerec System. Martin & Jedynakiewicz (1999), completed a systemic review of 29 clinical studies of

Cerec ceramic inlays between the years 1986 and 1997. Of the 29 studies considered, 15 were included in the systemic review with a total of 2862 restorations. The authors reported a survival rate of 97.4 % over a period of 4.2 years. The fracture was the most common reason of failure of the restorations. The studies used Vitablocs Mark I, Vitablocs Mark II, and Dicor MGC (the Vitablocs Mark and Dicor MGC are no longer marketed).

### 2.2.5.2 Copy-milling technique

The Celay system is a variation of the direct-indirect restoration concept but without the need for a laboratory technician. After the tooth preparation is completed, a precision imprint composite material is molded directly in the patient's mouth or indirectly in the dental laboratory, where it is adjusted for occlusion, contact relations, and marginal integrity. The material then undergoes a light or chemical curing process. Afterwards, it is removed from the cavity, mounted on one side of the Ce1ay (the scanning Side), and it serves as a prototype model to be copied and reproduced in ceramics using the milling system.

The model is fixed in a special attachment unit and the scanning tools are used to trace the surface of the restoration while a corresponding milling tool removes the ceramic. The system uses a sequential milling procedure ranging from coarse to fine milling burs and can mill a typical inlay restoration in about 15 to 20 minutes. The internal and occlusal surfaces are fully formed with this technique. The same type of ceramic blocks that are available for the CAD/CAM systems can be used with this technique (van Dijken, 1999). In-Ceram alumina and Spinell blocks can also be used to fabricate single- and multiple-unit In-Ceram cores for production of all-ceramic crowns and bridges. The In-Ceram porous alumina is milled with the Celay and subsequently infused with glass before application of the overlying porcelain. The milling procedure for In-Ceram dramatically decreases the time needed to produce an In-Ceram restoration by eliminating the slip fabrication and long sintering cycle. Glass infusion time is also decreased. The milled copings are veneered with aluminous porcelain (Giordano, 1996; Eidenbenz et al., 1994; van Dijken, 1999).

Two advantages are provided by the availability of CAD/CAM or other machining routes to all-ceramic restorations. Firstly, these systems remove the processing of ceramics, and hence microstructural control, from the dental laboratory and place it within jurisdiction of the manufacturer. Secondly, the manufacturer merely provides a few sizes of simple blocks; complex shaping is controlled by the machining process (Kelly et al., 1996; Beuer et al., 2008a).

Hwang & Yang (2001), evaluated the fracture strength of copy-milled and conventional In-Ceram crowns and found that the copy-milled In-Ceram Alumina crowns showed significantly higher fracture strength (984.81 N) than the conventionally fabricated crowns (876.19 N). In the case of crowns having spinel core, Celay In-Ceram Spinell had slightly higher fracture strength (706.32 N) than conventional In-Ceram Spinell (687.90 N), no significant difference in fracture strength between the two methods was observed (P > 0.05). In-Ceram crowns having alumina core showed significantly higher fracture strength than those having spinel core in both methods.

### 2.2.5.3 Procera AllCeram CAD/CAM system

The Procera AllCeram (Nobel Biocare, Göteborg, Sweden) crown was developed by Andersson & Oden in 1993. The Procera system utilizes the concept of CAD/CAM to fabricate all-ceramic restorations. The restoration is composed of a densely sintered, high purity aluminium oxide coping combined with compatible low-fusing AllCeram veneering porcelain (Andersson et al., 1998). The ceramic coping is composed of more than 99.5 % pure alumina ( $Al_2O_3$ ) that is sintered to produce a pore-free crown substrate (Prestipino et al., 1998). The fabrication of the coping takes into account the sintering shrinkage, approximately 20 %, by enlarging a model of the preparation that is used in the manufacturing process. The scanner, which is located in a local dental laboratory, digitizes a conventional die. The digital image is sent by the modem to the central production unit, where the computer-controlled milling machine fabricate refractory dies compensating for the sintering shrinkage. The high-purity aluminium oxide powder (> 99.5 %) is compacted against the enlarged preparation model, milled, and sintered to full density (Andersson et al., 1998).

The high-purity aluminum oxide system promotes the densification of alumina during melting and solidification, hence eliminating most porosity and increasing the strength of the material (Andersson and Oden, 1993). Wagner & Chu (1996), evaluated the strength of aluminum oxide ceramics using a biaxial flexural test. These authors reported significant differences in flexural strength for the Procera AllCeram material (687 MPa) when compared to In-Ceram at 352 MPa and IPS Empress at 134 MPa. The fractured toughness of Procera AllCeram (4.48 MPa) and In-Ceram (4.49 MPa) was significantly higher than the fractured toughness of the IPS Empress ceramic (1.74 MPa).

In a study of in vitro fracture resistance of Procera AllCeram crowns by Abed et al., (1997) it was concluded that differences in the coping thickness of 0.5 mm and 0.7 mm produced no significant differences in the fracture resistances of the crowns tested.

Oden & co-workers, (1998) evaluated the clinical performance of Procera Allceram crowns for five years. Of the 97 crowns remaining in the study after five years, only three crowns had experienced a fracture through the veneering porcelain and the aluminium oxide coping material. Two additional crowns were replaced as a result of fractures of only the veneering porcelain. One crown was replaced as a result of recurrent caries. All remaining crowns were ranked as either excellent or acceptable for surface/colour, anatomic form and marginal integrity.

Chai et al., (2000) compared the probability of fracture of four systems of all-ceramic crowns fabricated on maxillary central incisors. The mean strength of the crown systems were 1,005 N for In-Ceram; 865 N for In-Ceram CAD/CAM; 1,111 N for Empress and 902 N for Procera. There were no significant differences in the probability of fracture among the four systems studied.

#### **2.3** Strength of all-ceramic materials

Ceramics are brittle and have limited tensile strength because of the presence of inherent flaws within the material and their inability to reduce the tensile stresses at the tip of the cracks by deforming. This also explains why dental restorations normally fail in areas of tensile stresses (Anusavice, 1996). Therefore, tensile strength is generally considered as the more meaningful property for these brittle materials for assessment of the failure potential of dental restorations, especially in the presence of critical surface flaws.

Strength is an important mechanical property that controls the clinical success of dental restorations. Usually, complex stress distributions that are induced by compressive, tensile, and shear stresses are present in most specimens under practical conditions. It is

extremely difficult to induce a pure stress of a single type in a body. In general, tensile strength is easily determined for ductile materials such as metals. For convenience, compressive strength is often measured for brittle materials such as porcelains, cements, amalgams, and resin composites (Ban and Anusavice, 1990).

Extrapolation of strength data of clinical performance, considered alone, must be approached cautiously. Proper use of strength data for predicting the life expectancy of restorations in clinical situations requires that; (1) critical flaw in the test specimen is the same as the one involved in clinical failure; (2) environmental influences have been replicated in the laboratory; (3) failure parameters describing the flaw size, distribution and crack growth rates have been measured; and (4) stress distribution in the clinical structure is well characterized (Kelly, 1995).

#### **2.3.1** In vitro strength tests for modern dental ceramics

Before starting time-consuming and costly clinical investigations, preclinical in vitro studies should be carried out to evaluate the clinically relevant properties for newly developed dental materials and products (Gu and Kern, 2003).

Numerous test methods have been used to evaluate the strength of ceramics such as tensile strength test, diametral tensile strength test, compressive strength test and flexural strength test (Wang et al., 2003; Sadighpour et al., 2006). However due to complex geometry of restorations no standard method exists for measuring the strength of these configurations. For this reason, discrepancies exist amongst the published values of mechanical properties for a given material. Sometimes, researchers use devices that try to simulate dental morphology. However, the experimental variables can become extremely complex and difficult to reproduce in this type of testing (Craig,

2002). Hence, strength values which are obtained from a well-defined shape specimen like a bar or disk, could be often used as indicators of structural performance for dental materials. Strength data are however meaningful when placed into context via knowledge of the material microstructure, processing history, testing methodology, testing environment and failure mechanisms (Kelly, 1995).

Fracture strength values are often relied upon as indicators of structural performance for brittle dental materials. However, strength is more of a conditional than an inherent material property, and strength data alone cannot be directly extrapolated to predict structural performance (Kelly, 2004).

Typically, the fracture strength of ceramics is checked by using bend bars with three or four point loading and/or discs tested in biaxial flexure (Campbell, 1989; Giordano et al., 1995; Seghi and Sorensen, 1995). Measured strengths vary as a function of specimen preparation and testing methodology, including surface condition, three-point versus four-point bending and different stress rates (Craig, 2002).

Homogeneous all-ceramic restorations consist of a layer of ceramic (approximately 1.0-2.0 mm thick) atop a layer of cement (approximately 30 to 120 µm thick) supported by a layer of dentine. This structure is not well represented by simple bar-shaped specimens, such as those used in 3-point or 4-point bending tests (Kelly, 1999). Furthermore, the surface of a restoration is sophisticated and not represented by the typical test methods. Therefore, in order for strength testing to be relevant, it is generally recommended that the mode of loading be chosen to closely simulate the actual component in service (Ritter, 1995; Kelly, 1999).

The diametral tensile strength test provides a simple experimental method for measurement of the tensile strength of brittle materials. However, the complex stress distribution developed in the specimen can lead to various modes of fracture. If the specimen deforms significantly before failure, the data may not be valid (Ban and Anusavice, 1990). A study by Zidan et al., (1980) suggested that the diametral tensile test cannot be considered reliable for dental resinous materials.

The traditional tensile test, which is commonly used to evaluate the strength of metals, has rarely been used for brittle materials because of the difficulties associated with gripping and aligning the specimens and designing the mounting unit in the testing machine (Ban and Anusavice, 1990; Sadighpour et al., 2006).

#### 2.3.1.1 Flexural strength test

The strengths of brittle materials are usually measured in flexure (bending) because this test is generally easier to conduct than a pure tensile test. In bending, tensile stress reaches a maximum on one surface and compressive stress reaches a maximum on the opposite surface. In the flexural strength tests, the stress at the point of mechanical failure is defined as the failure stress (Zeng et al., 1998). The uniaxial flexural strength tests, including three-point, and four-point bending tests, and bi-axial bending tests are the most commonly applied methods for evaluating the strength of dental restorations (Zeng et al., 1996; Zeng et al., 1998; Tinschert et al., 2000).

The estimate of the uniaxial tensile strength for brittle materials is obtained from diametral tensile or three or four point bend flexure testing. The main advantage of the flexure test is that a state of pure tension can be established on one side of the specimen. However, the stress state under in-vivo conditions in the oral environments is not purely uniaxial, but biaxial or triaxial in nature (De Groot et al., 1987).

In uni-axial flexural strength tests, the principal stress on the lower surfaces of the specimens is tensile, and it is usually responsible for crack initiation in brittle materials. However, undesirable edge fracture (which can increase the variance of the failure stress value) can occur. Furthermore, these methods were designed for engineering materials that are usually associated with relatively large specimens.

The bi-axial flexural strength test has been used frequently for the evaluation of fracture characteristics of brittle materials (Wagner and Chu, 1996; Albakry et al., 2004; Pagniano et al., 2005). The measurement of the strength of brittle materials under bi-axial flexural strength conditions rather than uni-axial flexural strength is often considered more reliable because the maximum tensile stresses occur within the central loading area and spurious edge failures are eliminated.

The ease of sample preparation, the elimination of edge effects, the similarity to clinical size scale and intra oral loading conditions are the main advantages of biaxial flexural strength testing compared to uniaxial flexural strength testing. Furthermore, the evaluation of slightly warped specimens and the possibility of estimation of uniaxial flexural strength testing (Ban and Anusavice, 1990; Wagner and Chu, 1996; Wen et al., 1999).

According to ISO (ISO-6872, 1995(E)), the bi-axial flexural strength is determined by support of a disc specimen on three metal spheres positioned at equal distances from each other and from the center of the disc. The load is applied to the center of the opposite surface by a flat piston. The disc specimens can be easily made under typical restorative conditions. Furthermore, the flat surface of the test specimen can be easily controlled by conventional metallographic polishing methods and typical dental finishing techniques.

# 2.4 Ideal properties of a luting agent

The selection of the appropriate dental cement when delivering an indirect restoration is vital to the success of the treatment. This task has become a challenge, considering the different types of luting agents available and the increasing number of different restorative options. The following are the desirable properties of a dental luting agent:

### 2.4.1 Adhesion

The main function of the luting agent is to provide a reliable bond to retain the restoration on the tooth, to seal the exposed dentine and to fill the unavoidable gap between them (Davidson, 2001; Hatrick et al., 2003). This would occur if the cement would biomechanically or biochemically adhere to the restoration or prepared tooth. Zinc phosphate cement does not chemically bond either to the tooth structure or the restorative material. Polycarboxylate cements exhibit chemical adhesion to the tooth through interaction of free carboxylic acid groups with calcium. It is reasonable to assume that because of this adhesion, polycarboxylate cements would exhibit less microleakage. However, microleakage studies demonstrate that they leak just as much as zinc phosphate (Jivraj et al., 2006).

The glass ionomer cements form an ionic bond to the tooth as a result of chelation of the carboxyl groups in the acid with the calcium and phosphate ions in the apatite of dentine and enamel (Wilson et al., 1983). The adhesion of resin-modified glass-ionomer cements to enamel and dentine is thought to be through a mechanism similar to conventional glass ionomers. However, the bond strengths to dentine are higher and these luting agents also bond to composite resin (Kim et al., 1998; Diaz-Arnold et al., 1999).

With the advent of predictable dentine bonding, the resin cements can bond to both tooth structure and restorative material. Recently, dual-cured, self-etching, self-adhesive resin cements that do not require bonding agents have been introduced. In addition, resin cements bond chemically to resin composite restorative materials and to silanated porcelain (Diaz-Arnold et al., 1999).

It is reasonable to assume that luting agents that present stronger bonds to tooth structure will also demonstrate less microleakage. This has been verified by both in vitro (Albert and El-Mowafy, 2004) and in vivo studies (White et al., 1994).

#### 2.4.2 Working and setting time

The working and setting time are considerations in the choice of luting agent. The setting characteristics should allow sufficient time for mixing the material, applying to the restoration and/or tooth preparation and for seating the restoration in place in the mouth. With conventional luting agents, the working time can be varied by utilizing - techniques such as slaking, incremental mixing, use of a chilled slab, and mixing over a wide area to dissipate the heat of the exothermic reaction (Hill, 2007; McCabe and Walls, 2008).

Working time for glass-ionomer cement is shorter than that for zinc phosphate or polycarboxylate. With resin cements there is a choice between dual-cured and lightcured resins. The light-cured resins have some purported advantages in that working time is increased, the ability to remove excess and reduced finishing time (Anusavice, 1996).
#### 2.4.3 Compressive and tensile strength

Cements used for permanent and high-strength bases need good compressive and tensile strengths. Cements are brittle materials with good compressive strength but more limited tensile strength. Testing has revealed that zinc phosphate has the lowest compressive and diametral tensile strength while resin cements have values which are much higher (Hatrick et al., 2003). The cement strength is almost linearly dependent on the powder/liquid ratio; thus, the more the powder the better the strength (Diaz-Arnold et al., 1999).

Increased strength of cements will not increase retention of castings cemented on prepared teeth. However, crown retention is a function of resistance and retention form coupled with accuracy of fit of the casting. Increasing the strength of the cement will not compensate for lack of retention and resistance form (Donovan and Cho, 1999).

#### 2.4.4 Solubility

An ideal luting agent must not dissolve or wash out in oral fluids over the lifetime of the restoration (Hill, 2007). Most of the cements used in dentistry will disintegrate in the oral environment over time. Solubility is important whenever the cement is expected to remain exposed to mouth fluids for prolonged periods of time. Significant differences in solubility exist between the different luting agents. Zinc phosphate cements show relatively low solubility in water (0.06%) when they are tested in accordance with the American Dental Association (ADA) specification. Glass ionomer cement is quite soluble within the first 24 hours (1.25%) and performs poorly in a 24-hour test. However after the initial 24 hours, glass ionomers are quite resistant to dissolution and hence perform very well in a long-term clinical test. The latter is more clinically significant (Anusavice, 1996; Donovan and Cho, 1999; Hatrick et al., 2003). Excellent

fitting of restorations reduce the solubility of cement in oral fluids. Therefore, the problem should not be the solubility of the cement but rather the fit of the restoration (Jivraj et al., 2006).

Recent study conducted by Kuybulu et al., (2007) found that water based cements showed greater erosion in acid storage medium, whereas resin-based cements did not experience a loss of depth, but rather expanded following hygroscopic expansion caused by water sorption.

#### 2.4.5 Low-film thickness (low viscosity)

Ideally, the material should be of low initial viscosity to allow flow of the luting cement so that proper seating of the restoration can occur (McCabe and Walls, 2008). The film thickness of the luting agent is influenced by a number of factors including particle size of the powder, cementation force and technique, viscosity and the use of specific techniques such as die spacing, venting, or placement of escape channels (Hill, 2007). The luting space should be kept to a minimum to improve the fit of the restoration, exposing minimum of luting material to oral fluids and minimizing any polymerization contraction stress (De la Macorra and Pradíes, 2002). According to American Dental Association (ADA) specifications, effective luting agent should be able to flow into a film thickness of 25 µm or less (Diaz-Arnold et al., 1999).

#### 2.4.6 Biocompatibility

An ideal luting agent should not be harmful to the dental tissues. Many types of cement are a combination of a powder of zinc oxide or powdered glass and acid. The pH of the acid both at placement and after complete setting is a concern. Careful attention to powder/liquid ratios, dispensing technique and mixing recommendations can minimize this concern (Hatrick et al., 2003). It was long thought that cements containing phosphoric acid cause pulpal inflammation as a result of their low pH. Research has challenged this belief and it is likely that all commonly used dental cements (eg. Zinc phosphate, silicate cement, glass ionomer and resin) elicit no long-term pulpal response and hence meet the criteria for biocompatibility (Cox et al., 1987; Felton et al., 1991a).

Clinical symptoms such as sensitivity after crown cementation are more likely due to microleakage rather than pulpal inflammation resulting from insult caused by the luting agent. Therefore, it appears to be a perfectly rational way to seal and protect the dentino-pulp complex, prevent sensitivity, and bacterial leakage during the cementation phase (Jivraj et al., 2006).

#### 2.4.7 Anticariogenic properties

It has been proven that fluoride is released from certain dental materials, although at different rates and with different durations, depending on the material tested (Helvatjoglu et al., 2001). Many luting agents have been described as having anticariogenic properties and a number of these have been marketed on this premise.

The fact that a material contains fluoride does not necessarily endow it with anticariogenic properties. Sufficient concentrations of fluoride must be released over a period of time, and the material itself should not suffer from any significant degradation (Jivraj et al., 2006). Glass ionomer cements have been reported to have long-term fluoride release and cariostatic activity of these cements has been proposed. However, even if fluoride is released, it is important to know how much fluoride is released from the margins of a well-fitting restoration, and whether this amount of fluoride has any significant impact. Nevertheless, a gap-free interface seems more important in preventing secondary caries than the release of fluoride or other substances (Imazato et al., 1998; Imazato et al., 1994).

#### 2.4.8 Radiopaque

An ideal luting agent should be radio-opaque to enable the practitioner to distinguish between the cement, the tooth, and the restoration. Presently, there is no specification for radiopacity of luting materials, but it is important that dental cements have greater opacity than dentine. Proper radiopacity of a dental material allows differentiation between tooth and restoration to detect eventual gaps, secondary caries, overfillings or underfillings. As the radiopacity of the luting agent increases, the detection threshold for marginal overhangs decreases; thus, a luting agent should be chosen that is as radiopaque as possible (Rosenstiel et al., 1999). Triphenyl bismuth is an example of an additive to biomedical resin that increases radiopacity. The tooth-colored formulations have recently been established to impart radiopacity with a zirconium additive (Rosenstiel et al., 1999).

It is impossible radiographically to detect excess luting agent if the material is radiolucent. In practice, luting agents come in a wide range of radiopacities. A study conducted by Attar et al., (2003) showed that zinc phosphate has the highest radiopacity of all materials tested. The dual polymerized resin and conventional glass ionomer showed radiopacity essentially the same as human enamel. The radiopacity of RMGI was intermediate between enamel and dentine. The autopolymerizing luting agents showed similar radiopacity to dentine and had the lowest radiopacity of all materials tested.

#### 2.4.9 Ease of manipulation

One of the most important attributes of any dental material is that it should be relatively easy to use and manipulate. Amongst the conventional luting agents, zinc phosphate appears to be the least technique-sensitive. A specific protocol is required with the use of zinc phosphate, and as long as these recommendations are followed long-term success will be achieved (Donovan and Cho, 1999; Hill, 2007).

Resin cements are extremely technique-sensitive because of their inherent polymerization shrinkage and their sensitivity to moisture. Resin-modified glass ionomer cements are less technique-sensitive than the resin cements and in auto-mix cartridges, can prove to be an extremely efficient way of delivering cast restorations (Hill, 2007). Another category of luting agents that has recently been introduced is the auto-adhesive group. This category of resin cement is becoming very popular with practitioners because of the reduced chairtime and a simpler application protocol. It combines the adhesive and resin in one product eliminating the need for pretreatment of both tooth and restorative material prior to cementation (Abo-Hamar et al., 2005).

#### 2.4.10 Aesthetic

The aesthetic properties of luting agents are of considerable significance with the increasing use of translucent ceramic restorations, especially for anterior restorations (Rosenstiel et al., 1999; Hill, 2007). Presently, an aesthetic appearance of luting materials is virtually a must in almost all non-metallic restorations, particularly when margins are visible. In such regions, colour-matched resin-based luting materials are clearly superior to any other type, mainly due to their translucency and excellent colour match to dentine and enamel. Ionomer-based luting materials may also have a good

colour match, but their translucency is somewhat inferior to resin-based luting materials (De la Macorra and Pradíes, 2002).

Colour stability over time should be considered. The amine accelerator necessary for dual polymerization can cause the colour of the luting agent to change over time (Brauer et al., 1979). Therefore, many practitioners prefer light-cured resin cements for luting porcelain veneers and other aesthetic restorations because it is thought that they are more colour stable. Noie et al., (1995) have shown that although measurable colour changes of dual resin cements were detected under accelerated ageing, they were not found to be visually perceptible. Their findings suggest that the practitioners can use dual-cure resin cements in aesthetic areas with confidence.

# 2.5 Dental Luting Agents

Dental luting cements form the link between indirectly fabricated restorations and the prepared tooth structure (Ergin and Gemalmaz, 2002). Their major function is to retain the restoration on the tooth, to seal the exposed dentine and to fill the unavoidable gap between them (De la Macorra and Pradíes, 2002; Burke, 2005).

Three main types of luting cements are commonly in use, zinc phosphate, glass ionomer cements and composite resin cements. Zinc phosphate cement is considered the gold standard against which all other luting agents are compared because of its long clinical history of successful use. Glass ionomer cements are classified as either conventional glass ionomer cements which are water-based without any resin or resin modified glass ionomer cements that have some resin added to the formula. The purpose of adding resin was to enhance the physical properties and to reduce the sensitivity to water balance of the conventional GICs. Composite resin cements have gained considerable

popularity in recent years. These include the adhesive composite resin cements that require a separate adhesive application and self-adhesive composite resin cements. The resin-based cement category comprises light-cured, dual-cured and chemically cured agents (Tyas and Burrow, 2004; Jivraj et al., 2006; Pegoraro et al., 2007; Monticelli et al., 2008).

#### 2.5.1 Zinc phosphate cement

Zinc phosphate cement is the oldest of the currently available luting cements, having been available, unchanged for 100 years (Burke, 2005). It is supplied in several brands as a powder-liquid system. These cements are primarily used as permanent luting agent, under indirect restorations and for cementation of orthodontic bands (Hatrick et al., 2003).

The principle components of the zinc phosphate cement powder are zinc oxide (90%) and magnesium oxide (8%). Silicone oxide, bismuth trioxide and other minor ingredients are used in some products to alter the working characteristic and final properties of the mixed cement. The liquid contains phosphoric acid (67%), water, aluminium phosphate and in some instances zinc phosphate (Anusavice, 1996; McCabe and Walls, 2008). Aluminium (1%–3%) in the liquid is needed for the cement-forming reaction. The water content (33%) is significant because it controls the ionization of the acid, which in turn influences the rate of the setting reaction (Craig, 2002; Hill, 2007).

Zinc phosphate cement does not chemically bond to any substrate and provides a retentive seal by mechanical means only. It possesses adequate compressive and tensile strengths, low film thickness of about 25  $\mu$ m for cementation, pH of 3.5 at time of cementation and also it has a reasonable working time (Craig, 2002; Burke, 2005). Ayad

et al., (1995) reported that the greatest retentive strength of zinc phosphate cement was achieved by increasing the surface roughness of tooth preparation.

Apart from the strong rationale for its use, disadvantages of zinc phosphate cement include the negative biologic effects (pulp irritation), the lack of antibacterial action, the lack of adhesion and the elevated solubility in oral fluids (Davidson, 2001).

A recent study by Johnson et al., (2009) has evaluated the retention of metal-ceramic crowns to human dentine using different luting cements. The results of the study showed that conventional resin cement, resin-modified glass ionomer cements and self-adhesive resin cements demonstrated greater casting retention (4.0-8.0 MPa) than zinc phosphate cement (2.3 MPa).

#### 2.5.2 Glass ionomer and resin-modified glass ionomer cements

Glass ionomer cement was developed by Wilson from the desire to have a luting agent with the fluoride release/translucency of dental silicate cement and the adhesion to tooth of polycarboxylate cement (Wilson et al., 1977; Hill, 2007).

The powder is composed mainly of a sodium alumino-silicate glass, and contains fluoride to lower the temperature of the glass fusion, improve the handling properties of the cement mix and modify properties (Hewlett and Mount, 2003; McCabe and Walls, 2008). The liquid consists of copolymers of relatively weak polyalkenoic acids, including itaconic, maleic, tartaric, plus other minor organic acids. These acids can also be freeze-dried and incorporated into the powder component, which is then mixed with water to reconstitute the acid (Diaz-Arnold et al., 1999; McCabe and Walls, 2008).

When the powder and liquid are mixed, the polyacid attacks the glass to release fluoride ions (Mount, 2002).

These cements bond chemically to tooth structures, forming an ionic bond between the carboxyl groups of the polyalkenoic acid and calcium of hydroxyapatite, and this may contribute to the retention of the restoration (Tyas and Burrow, 2004). This is a critical point, because bonding using products with separate etch and rinse steps are very technique sensitive. Over-etching, under-etching, too much moisture in the dentine, too little moisture, over-or under-drying of the dentine bonding agent all contribute to reduced bond strength and seal (Van Meerbeek et al., 2003).

Glass ionomer cements have superior compressive strength ranging from 93-226 MPa and is greater than that of polycarboxylate and phosphate cements (Diaz-Arnold et al., 1999; Craig, 2002; McCabe and Walls, 2008). The compressive strength of the glass ionomer cements increases between 24 hours and one year. Moreover, the strength improves more rapidly when the cement is isolated from moisture during its early life (Craig, 2002).

Values of solubility and disintegration of the glass ionomer cements as measured in water by an ADA test are substantially higher than those measured for other cements. However, when these cements are tested under ideal laboratory conditions, the values are quite low compared to values for polycarboxylates, and most of the other cement products (Craig, 2002, Kuybulu et al., 2007; McCabe and Walls, 2008).

Glass ionomer cements are biologically compatible with the pulp when manipulated properly. Early concerns and reports of post-cementation sensitivity when using glassionomer cements have largely been dismissed as being multifactorial in origin. Over drying of the preparation and moisture contamination during the first 24 hours of setting have been indicated as possible sources of this sensitivity (Hatrick et al., 2003). However, mild to moderate sensitivity has been reported, particularly if mixed to a low powder-liquid ratio (Burke, 2005).

Chemical adhesion to tooth structure by chelation with calcium and phosphate ions in dentine and enamel, good translucency and slow, long-term fluoride release are all factors that have made glass-ionomer an extremely popular definitive luting agent (Diaz-Arnold et al., 1999). Fluoride release has been shown to be pH-dependent (being greater at lower pH values). In addition glass-ionomer cement displays fluoride uptake when exposed to topical fluoride (Preston et al., 2003). Selection of this material as a luting agent may be an important issue for the patient who has high caries potential (Gandolfi et al., 2006).

Resin-modified glass ionomer cements were developed in order to overcome some of the shortcomings of traditional glass ionomer cements, such as poor tensile strength. In addition to the components of glass ionomer materials (fluoroaluminosilicate glass and polyalkenolic acid), these materials also contain a monomer such as 2-hydroxyethyl methacrylate (HEMA) or BisGMA (Burke, 2005). This modification in the chemistry provides the advantages of the conventional glass-ionomer systems and resin technology (Terry, 2005; Nicholson, 2007).

Resin-ionomer cements exhibit compressive and diametral tensile strengths that are greater than zinc phosphate, polycarboxylate, and several conventional glass ionomer cements, but less than composite resin luting cement (Terry, 2005).

Clinical features of resin-modified glass-ionomer cements include; fluoride release and cariostatic potential, resistance to marginal microleakage, adhesion to enamel and dentine, improved fracture resistance and wear characteristics, and more resistant to moisture and less soluble than conventional glass-ionomer cements (Diaz-Arnold et al., 1999; Mitchell et al., 2000; Burke, 2005; Terry, 2005).

The adhesion of resin-modified glass ionomer luting cements to enamel and dentine is thought to be through a mechanism similar to conventional glass ionomer cements, however, the bond strengths to dentine are higher and these luting agents also bond to composite resin (Ngo et al., 1997; Ergin and Gemalmaz, 2002).

Resin-modified glass ionomer cements can release fluoride and other ions. The amount released is greater in acid solutions than in neutral solutions. The matrix-forming ions released are determined in part by the chemical composition of the glass component and in part by the medium in which the material is stored. Storage in an acidic medium increases the release of ions. Conventional glass-ionomers can buffer storage solutions, changing the pH of lactic acid solutions, for example, from 2.7 to 4.5 (Nicholson et al, 2002; Nicholson, 2007).

Like their conventional counterparts, resin-modified glass-ionomer cements have been found to buffer their storage media and release ions. It was concluded that the presence of the resin phase in these materials makes little or no difference to their overall interaction with aqueous media (Czarneka and Nicholson, 2006). Disadvantages (compared to conventional glass-ionomer cements) include higher shrinkage at setting and sensitivity to dehydration, resulting in significant shrinkage. Degradation from long-term exposure to moisture may result in an increased water sorption and subsequent plasticity and hygroscopic expansion (Diaz-Arnold et al., 1999).

### 2.5.3 Resin-based cements

Resin luting cements have become popular clinically owing to their ability to bond to both the tooth structure and restoration. In general, resin-based cements are the material of choice for adhesive luting of all-ceramic restorations (Kramer et al., 2000; Hill, 2007). A variety of resin-based cements have now become available because of the development of the direct-filling resins with improved properties, the acid-etch technique for attaching resins to enamel and molecules with a potential to bond to dentine conditioned with organic acid (Pegoraro et al., 2007). These luting materials are generally supplied in a range of shades and may therefore be used with aesthetic restorations such as tooth-coloured crowns, veneers and inlays. They are also indicated for resin-retained bridges or, indeed, for any type of indirect restoration (Burke, 2005).

The early resin cements were primarily poly-methyl methacrylate powder with various inorganic filler and methyl methacrylate liquid. Resin cements are methyl methacrylate-, Bis-GMA dimethacrylate-, or urethane dimethacrylate-based, with fillers of colloidal silica or barium glass 20% to 80% by weight (Hill, 2007). The composition and characteristics of most modern resin-based cement are similar to conventional composites and consist of inorganic fillers embedded in an organic matrix such as Bis-GMA, TEGDMA and UDMA (Blatz et al., 2003a). Filler particle size is kept very small, similar to microfills or microhybrids. Initiators of polymerization are added to change the setting mechanism. Pigments are added to aid in tooth colour matching (Hatrick et al., 2003).

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Resin bonding to enamel is by micromechanical interlocking into an acid etched surface. Bonding to dentine is also micromechanical, but is much more complex, usually requiring multiple steps that include removal of the smear layer and surface demineralization, then application of an unfilled resin bonding agent or primer, to which the resin chemically bonds (Hill, 2007). The practice of total etching, which frequently resulted in postoperative sensitivity, has been deemed not necessary and has been replaced by less invasive self-etching methods (Christensen, 2006). Residual eugenol from provisional cement can interfere with the setting reaction of the bonding agent, so non-eugenol provisional cement is recommended when resin is used for the definitive restoration (O'Brien, 2002). Polymerization shrinkage of the luting resin (depending on the bulk) may be significant enough to generate stresses that can form small gaps at the cement/tooth interface (Diaz-Arnold et al., 1999).

Resin luting cements chemically bond to etched, silane-treated porcelain (Diaz-Arnold et al., 1999). Based on multiple laboratory and clinical studies looking at fracture resistance and sealing, resin cements are considered the best choice for luting all-ceramic restorations. It has been postulated that resin cement bonded to conditioned tooth on one side and etched/silane coated porcelain on the other helps diffuse stresses across the tooth (Burke et al., 2002).

Because of the diversity of products and their ingredients, physical properties for resin cements vary, but certain generalizations can be made (O'Brien, 2002). Resin luting cements possess high compressive strength, increase the fracture resistance of ceramic materials that can be etched or sandblasted and silanated, and resist tensile fatigue (Attar et al., 2003; and AL-Makramani et al., 2008a). They have good aesthetic qualities, ability to adhere to multiple substrates, increased retention, low solubility, improved

marginal wear resistance and less microleakage in comparison to conventional luting cements (Piwowarczyk et al., 2004; Albert and El-Mowafy, 2004; Terry, 2005; Kuybulu et al., 2007). Conversely, resin luting cement offers no fluoride release or uptake, short working time, greater film thickness and postoperative sensitivity from polymerization shrinkage. They require more complicated clinical procedures that involve multiple steps that are technique sensitive and more expensive. Removal of a restoration may require total destruction and more time consuming than zinc phosphate, conventional glass ionomer, or resin-ionomer cements (Mitchell et al., 2000; Hill, 2007).

### 2.5.4 Self-adhesive resin luting cements

Currently, all resin cements are based on the use of an etch-and-rinse or self-etch adhesive along with a low-viscosity resin composite to bond aesthetic restorations to dental structures (Van-Meerbeek et al., 2003). These adhesives are both somewhat acidic and hydrophilic in nature. During cementation, the acidic groups in the uncured layer of simplified adhesive agents (due to the presence of oxygen) compete with peroxides for aromatic tertiary amines of the luting agent, resulting in an acid-base reaction between the adhesive and the resin cement. This reaction minimizes appropriate co-polymerization between the adhesive and the resin cement (Sanares et al., 2001; Suh et al., 2003). Additionally, the hydrophilic feature of such self-etch adhesive systems functions as a permeable membrane. This hydrophilic behaviour permits the flux of water through the adhesive after polymerization. The presence of water droplets at the interface between the adhesive-cement interface (Tay et al., 2002; Carvalho et al., 2004).

This multi-step application technique is complex, and consequently may compromise bonding effectiveness (Van-Meerbeek et al., 2003). Recently, new self-adhesive resin cements without surface pre-treatment have been marketed and combines the use of adhesive and cement in one single application (Abo-Hamar et al., 2005).

These cements consist of multifunctional phosphoric acid dimethacrylate–modified monomers, such as Bis-GMA, and inorganic fillers of fine glass and silica. The phosphoric acidic methacrylates can react with the basic fillers in the luting cement and the hydroxyapatite of the hard tooth tissue (Hikita et al., 2007).

Abo-Hamar & co-workers, (2005) have stated that the use of self-adhesive resin cement (RelyX Unicem; 3M ESPE, Seefeld, Germany) with its simplified application procedure may be considered an alternative to the currently used systems for luting conventional ceramics, high-strength ceramics and metal-based restorations, when no or little enamel is left.

Hikita et al., (2007) have evaluated the bond strength of adhesive luting agents to enamel and dentine. The etch-and-rinse, self-etch and self-adhesive resin luting agents are equally effective in bonding to enamel and dentine, on the condition that a correct adhesive procedure was carried out.

According to Lin-hu et al., (2007), phosphoric acid etching has been shown to improve the bonding strength of self-adhesive luting agents to enamel, but is unnecessary for dentine. Komine & co-workers, (2004) have found that these cements are capable of successfully luting aluminum oxide all-ceramic crowns. The incompatibility and technique sensitivity issues of older resin luting materials appear to have been overcomed with the introduction of a novel self-adhesive resin luting material, for which initial laboratory and clinical testing shows promise (Burke, 2005). The results of a laboratory study investigating the retention of complete crowns prepared with three different tapers and luted with four different cements have indicated that the retention of the adhesive resins investigated were 20% higher at 24° taper than the retentive values of conventional cements at 6° taper (Zidan and Ferguson, 2003). As the resin luting materials provided retention that was double the values of zinc phosphate or conventional cements, this result provides an overwhelming indication for the use of adhesive luting.

# 2.6 Factors affecting bonding to ceramics

All ceramic restorations are widely used in multiple clinical situations because of their excellent aesthetic qualities and good biocompatibility (Begazo et al., 2004). All-ceramic restorations rely on adequate bonding because of their brittle nature. Resin bonding of ceramic restorations to the supporting tooth structure increases the fracture resistance of the tooth and the restoration itself; it also minimizes microleakage, which may be the determining factor in the success or failure of the treatment (Burke, 1996; Pagniano et al., 2005; Toman et al., 2007).

The clinical success of all-ceramic restorations depends in part on the use of appropriate cementation procedures. A great number of luting cements and various surface treatments have been proposed to improve bond strengths to ceramics (Blatz etal., 2003a). These surface treatments are strongly dependent on the type and the microstructure of the ceramic surface (Kern and Thompson, 1995; Della Bona et al., 2000; Borges et al., 2003).

A durable and predicable bond between resin luting cements and ceramic is usually created by two mechanisms: micromechanical attachment to porosities originated from hydrofluoric acid etching and/or gritblasting and chemical bond by a silane-coupling agent (Filho et al., 2004). The common surface treatments are acid etching, airborne particle abrasion, silane-coupling agent, and combinations of these methods (Awliya et al., 1998; Özcana and Vallittu, 2003; Zhang et al., 2004; Nagayassu et al., 2006).

#### 2.6.1 Acid etching

The composition and physical properties of high-strength ceramic materials, such as aluminum oxide-based (Al<sub>2</sub>O<sub>3</sub>) and zirconium oxide-based (ZrO<sub>2</sub>) ceramics, differ substantially from silica-based ceramics (Blatz et al., 2003a; Soares et al., 2005). Although hydrofluoric acid is effective in roughening feldspathic ceramic for bonding resin luting cements, acid etching has no positive effect on high-strength ceramics since such ceramics do not contain a silicon oxide phase (Janda et al., 2003; Valandro et al., 2006). Therefore they require alternative bonding techniques to achieve a strong, long-term and durable resin bond (Awliya et al., 1998; Della Bona et al., 2000; Borges et al., 2003; Derand et al., 2006).

Hydrofluoric acid attacks the glass phase of conventional ceramic materials producing a retentive surface for micromechanical bonding with composite resin (Filho et al., 2004). According to Della Bona et al., (2004) the bond strength of composite cements increases with increasing ceramic surface roughness caused by acid-etching.

Hydrofluoric acid solutions between 2.5 percent and 10 percent applied for one to four minutes seem to be most successful (Chen et al., 1998; Canay et al., 2001). Nagayassu et al., (2006) found that hydrofluoric acid etching for two minutes produced a favorable micromechanical retention that enhanced resin cement bond strength to porcelain. They have also reported that a four minutes etching time was significantly less efficient than the two minutes etching time, which suggests that an over-etching may lead to stress concentration in the adhesive interface and weaken the ceramic surface (Canay et al., 2001).

Hydrofluoric acid is a toxic chemical which causes, in the case of skin contact, severe damage (Meldrum, 1999). As alternatives, to avoid the hazardous hydrofluoric acid, acidulated phosphate fluoride or phosphoric acid were also used to condition the ceramic surfaces, however, their effectiveness on the enhancement of the bond strength is still doubtful (Kato et al., 2000). According to Della Bona et al., (2003) IPS Empress I and II ceramic surfaces have shown greater adhesion values when conditioned by 9.5% hydrofluoric acid compared with the value obtained with 4 % acidulated phosphate fluoride.

#### 2.6.2 Silane coupling agents

Silane coupling agents function as mediators and promote adhesion between inorganic and organic matrices through dual reactivity. They are bi-functional molecules that bond silicon dioxide with the OH groups on the ceramic surface, and copolymerize with the organic matrix of the resin (Kim et al., 2006; Matinlinna and Vallittu, 2007a; Matinlinna and Vallittu, 2007b). Della Bona et al., (2003) have demonstrated an increase in adhesive resistance when using silane with ceramics reinforced with feldspar, leucite, or lithium disilicate. They have also concluded that the application of silane over non-treated ceramics presents a low resistant adhesive interface. Whilst, Hooshmand et al., (2002) discovered that a durable and reliable bond between a resin composite and leucite-reinforced feldspathic ceramic can be obtained by an improvement in the silane bonding procedure without the need for a micro-mechanical bond. The use of micro-roughening by gritblasting with aluminium oxide, followed by the improved silane treatment may be sufficient. They have found that a 15-second washing using 80°C water prior to a 30-second drying using a 50°C air jet promotes a reduction of the number of adhesive flaws. The association of silanization process with heat application helps to eliminate water, alcohol, and other solvents, and thus promotes the condensation reaction and the silicasilane covalent bonding.

The use of silanes is recommended for ceramics containing silicon with which a chemical reaction may occur and a siloxane (-Si-O-Si-O-) network with covalent bonding and perhaps an element of hydrogen bonding is established with the ceramic surface. That is not the case with ceramics with high alumina content. Although one report showed doubling in bond strength after silanization of high alumina content ceramic, but no explanation was given as to why this occurred (Blatz et al., 2003b). The application of silane has been controversial with regard to the effect of the adherence between composite materials and these types of ceramic surfaces (Blatz et al., 2003c). However it has also been suggested that silanization may improve the wetting of the surface, resulting in higher bond strength values even though very small (Madani et al., 2000; Özcan et al., 2001).

An alternative is to use phosphate-monomer-containing composite resin cements such as Panavia F, which seem to provide strong and long-term durable resin bonds to air particle-abraded glass-infiltrated alumina and densely sintered high alumina ceramics (Kern and Strub, 1998; Madani et al., 2000; Blatz et al., 2003a). The adhesive functional phosphate monomer 10-methacryloyloxy-decyldihydrogen phosphate bonds chemically to metal oxides such as aluminum oxides (Derand et al., 2006). Some authors recommend the use of these cements without a silane or bonding agent (Kern and Strub, 1998), whereas others suggest a silane coupling agent to increase wettability of the ceramic substrate (Özcan et al., 2001; Madani et al., 2000).

#### 2.6.3 Air abrasion (sandblasting)

Creating mechanical roughness with bur is obviously nonconsistent and arbitrary. An appropriate treatment with several material surfaces is airborne particle abrasion. Airborne particle abrasion cleans any greasy substances from ceramic surfaces and creates micromechanical bonding (Matinlinna and Vallittu, 2007a). In dental laboratories the procedure can be carried out by using alumina particles (e.g. with 50  $\mu$ m diameter) in pressurized air, typical working parameters being the perpendicular distance of the nozzle from the surface of 10 mm, with an air pressure of 2.5 bars, and application time of 10–15 s (Sadan et al., 2003).

Hydrofluoric acid surface treatment promotes shallow surface micromechanical retentions in alumina-based ceramic restorations due to its low silica content (Awliya et al., 1998; Özcan et al., 2001; Borges et al., 2003). Based on chemical facts, hydrofluoric acid etching is only possible with silica-based ceramics or glasses according to the reaction equation:

$$6H_2F_2 + 2SiO_2 \rightarrow 2H_2SiF_6 + 4H_2O$$

This reaction certainly cannot occur with alumina or zirconia. Therefore, other bonding techniques are required in order to lute these materials adhesively (Janda et al., 2003).

According to Sen et al., (2000) hydrofluoric acid chemical conditioning did not produce good results for alumina-based ceramics, and surface sandblasting can be considered a good alternative for creating a micromechanical adhesion-favorable surface.

Awliya et al., (1998) & Kern and Thompson (1994), found significantly positive results with the adhesion of the composite cement when submitting In-Ceram alumina and Procera AllCeram ceramics to the shear test after sandblasting with 50  $\mu$ m aluminum oxide particles compared with hydrofluoric acid chemical etching, diamond abrasion plus phosphoric acid, or control (no treatment). In spite of this fact, Borges et al., (2003) showed that sandblasting with 50  $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles was not effective in increasing irregularities on the surface of these ceramics, which could mean an unreliable surface treatment to improve adhesion.

As an alternative treatment, silica coating and silane application with the Rocatec System (Rocatec System, 3M ESPE, USA) seems to provide a durable resin bond to glass-infiltrated aluminum oxide ceramic with bisphenol A glycidyl methacrylate (BIS-GMA) composite cements (Kern and Strub, 1998; Madani et al., 2000, Özcan, 2002).

Although silica coating systems were developed for coating of metals, they can improve bonding of resin to glass-infiltrated aluminum oxide ceramic, and densely sintered alumina ceramic (Kern and Thompson, 1994; Valandro et al., 2005). The silica coating systems create a silica layer on the ceramic surface because of the high-speed surface impact of the alumina particles modified by silica. The tribochemical silica coating system, which include sandblasting and formation of silica layer, increases tensile bond strength of resin luting cement (Panavia F), and shear bond strength of luting cements (zinc phosphate, glass ionomer, resin-modified glass ionomer, and dual-cured resin cement) on Procera AllCeram (Valandro et al., 2005; Blixt et al., 2000).

Kern & Thompson (1995), compared the effects of silica coating on bond strength of resin cements with In-Ceram cement with Rocatec and Silicoater systems and concluded durable bond strength was achieved with the Rocatec system.

# 2.7 Marginal integrity of all-ceramic crowns

In addition to the inherent properties of all-ceramic crown systems and their design and fabrication techniques, luting agents are considered one of the main factors contributing to marginal discrepancies of crown restorations because of crown elevation after cementation (Behr et al., 2003; Gu and Kern, 2003; Albert and El-Mowafy, 2004; Quintas et al., 2004).

Marginal fit is one of the important criteria used in the clinical evaluation of fixed restorations. The presence of marginal discrepancies in the restoration exposes the luting agent to the oral environment. The larger the marginal discrepancy and subsequent exposure of the luting agent to oral fluids conditions, the more rapid is the rate of cement dissolution (Sulaiman et al., 1997; Jacobs and Windeler, 1991). As a consequence, microleakage occurs and permits the ingress of food, oral debris and other substances that are potential irritants to the dental pulp tissues (Bergenholtz et al., 1982; Eick and Welch, 1986). Microleakage has also been related to the longevity of the tooth, periodontitis and secondary caries, which is considered the most common reason for replacing restorations (Gardner, 1982; Felton et al., 1991b; Mjör and Toffenetti, 2000).

Holmes et al., (1989) have established several gap definitions according to the contour differences between the crown and tooth margin. According to their classification, the marginal gap is defined as the perpendicular measurement from the internal surface of the casting to the axial wall of the preparation at the margin.

Great variations or complex gap morphology complicate attempts to measure marginal gaps; thus, it was of practical interest to determine the number of measurements necessary for determining gap sizes (Groten et al., 2000). The number of sites measured per crown varies considerably. It ranges from 4 (Sulaiman et al., 1997) to 8 (Albert and El-Mowafy, 2004), 12 (Anusavice and Carroll, 1987), 54 (Rinke et al., 1995), 60 (Akbar et al., 2006), 64 (Okutan et al., 2006) and more than 100 sites per crown (Groten et al., 1997).

Groten et al., (2000) conducted a study to estimate the minimum number of gap measurements on margins of single crowns to produce relevant results for gap analysis. They concluded that, approximately 50 measurements along the margin of a crown yielded clinically relevant information and a consistent estimate for the gap size.

Marginal fit is one of the most important criteria for long term success of all-ceramic crowns. Marginal fit of cemented restorations that range from 25 to 40  $\mu$ m has been suggested as a clinical goal, but marginal openings in the range of these dimensions clinically are seldom achieved (May et al., 1998). Marginal opening of  $\leq$ 120  $\mu$ m is considered clinically acceptable with regard to longevity (Fransson et al., 1985; Karlsson, 1993; McLean and von Fraunhofer, 1971; Boening et al., 1992; Boening et al., 2000; Suarez et al., 2003).

Lack of marginal fitting in all-ceramic crowns can affect fracture strength and thus reduce longevity. This is in addition to other known adverse effects of poor fit such as damage to the adjacent tissues and increased dissolution of the cementing medium (Tuntiprawon and Wilson, 1995; Karlsson, 1986).

Tuntiprawon & Wilson (1995), found that all-ceramic crowns with smaller gap dimensions at the axial wall and marginal opening demonstrated the best compressive strengths.

Sulaiman & co-workers, (1997) compared the marginal fit of three all-ceramic crown systems (In-Ceram, Procera and IPS Empress). The mean marginal discrepancy of the all-ceramic crowns was, in descending order: In-Ceram (161 ± 46  $\mu$ m), Procera (83 ± 41  $\mu$ m) and IPS Empress (63 ± 37  $\mu$ m). Both Procera and IPS Empress met the criterion for acceptable marginal discrepancy of 120  $\mu$ m.

Yeo et al., (2003) have found that IPS Empress 2 system ( $46 \pm 16 \mu m$ ) showed the smallest and most homogeneous gap dimension, whereas conventional In-Ceram system presented the largest and more variable dimension compared with the control group metal-ceramic crowns ( $87 \pm 34 \mu m$ ).

An in vitro study conducted by Albert & El-Mowafy (2004), found that the mean marginal gap of Procera (54  $\mu$ m) was significantly larger than the mean marginal gap of the control group metal-ceramic crowns (29  $\mu$ m).

**CHAPTER THREE** 

# FLEXURAL STRENGTH AND HARDNESS OF TURKOM-CERA COMPARED TO TWO OTHER ALL-CERAMIC MATERIALS

# 3.1 Introduction

Advances in all-ceramic systems have established predictable means of delivering aesthetic and biocompatible materials that are metal free. These metal-free materials must have sufficient strength to be a viable treatment alternative for the fabrication of crowns and fixed partial dentures (Chong et al., 2002).

The all-ceramic materials that are currently used in dentistry consist of alumina, zirconia, pressed, castable or machinable glass ceramics. Several developments have taken place in these areas resulting in the production of ceramic materials for clinical use. These include the aluminous porcelain crown (Vitadur), the non shrink ceramic crown (Cerestore), the castable mica glass-ceramic crown (Dicor) and the leucite reinforced glass ceramics (IPS Empress) (Qualtrough and Piddock, 2002; Santos et al., 2004; Conrad et al., 2007; Yilmaz et al., 2007). All these all-ceramic systems exhibit low flexural strengths (100 - 150 MPa) which make them at risk of failure when used for the construction of either posterior crowns or fixed partial dentures (Seghi et al., 1990; Giordano et al., 1995; Rizkalla and Jones, 2004).

The popularity of high-strength ceramic systems is increasing, and the range of their clinical indications is expanding constantly. Lithium disilicate ceramics (eg. IPS Empress 2), infiltrated alumina ceramic (eg. In-Ceram Alumina; and Turkom-Cera Fused Alumina), densely sintered aluminum oxide ceramic (eg, Procera), and zirconium oxide ceramic (eg, Procera AllZirkon; and Lava) are popular high-strength ceramic materials that offer favorable esthetic characteristics, mechanical properties and biocompatibility (Blatz et al., 2004; Della Bona et al., 2007).

Hardness is one of the frequently measured properties of a ceramic. It is usually measured with conventional microhardness instruments such as Knoop or Vickers diamond indenters (Yilmaz et al., 2007). These instruments make impressions for which the diagonal size is measured with an attached optical microscope (Quinn, 1998).

The hardness of a material is a relative measure of its resistance to wear and permanent deformation caused by indentation after a specific, constant load is applied (Anusavice, 1996; Miranda et al., 2003; Poskus et al., 2004). Thus, hardness may be described as a measure of the ability of a material to resist indentation or scratching (Mandikos et al., 2001). However, surface hardness alone is not an indicator of overall rigidity and strength and cannot predict the clinical behavior of long-span prostheses. Mechanical properties such as hardness, compressive and flexural strength of the new dental materials were claimed to be sufficient for its use (Diaz-Arnold et al., 1999).

Although long-term clinical studies constitute the ultimate basis on which to reliably predict the long-term performance of such restorations, several physical and mechanical properties are essential to support the correct indication of these materials (Wang et al., 2003). Because of different compositions and manufacturing techniques, dental ceramics vary in their physical and mechanical properties. The maximum biting forces that may occur in the posterior area vary between 300 and 880 N (Gibbs et al., 1986; Bakke, 1990; Braun et al., 1995; Okiyama et al., 2003; Ferrario et al., 2004). Therefore, it is important for the posterior restorations to be able to withstand the maximum biting forces created in this region. Therefore, the objectives of this study were:

 To compare the biaxial flexural strength of Turkom-Cera<sup>™</sup>, In-Ceram and Vitadur-N all-ceramic systems.  To compare the hardness of Turkom-Cera<sup>™</sup>, In-Ceram and Vitadur-N allceramic systems.

#### Null Hypotheses

- There is no difference in the biaxial flexural strength of Turkom-Cera<sup>™</sup>, In-Ceram and Vitadur-N all-ceramic systems.
- There is no difference in the hardness of Turkom-Cera<sup>™</sup>, In-Ceram and Vitadur-N all-ceramic systems.

# 3.2 Materials and methods

## **3.2.1** Materials used

Three different types of ceramic materials, Turkom-Cera<sup>™</sup> (Turkom-Ceramic (M) Sdn. Bhd., Puchong, Malaysia), In-Ceram (Vita Zahnfabrik, Bad Säckingen, Germany) and Vitadur-N (Vita Zahnfabrik, Bad Säckingen, Germany) were used in this study.

# 3.2.2 Methods

### **3.2.2.1** Preparation of the disc specimens

### **3.2.2.1.1** Preparation of Turkom-Cera discs

Perspex split mould with five circular openings of 16 mm diameter and 2 mm thickness was used for the preparation of the Turkom-Cera disc specimens (Figure 3.1). The Turkom-Cera Alumina Gel was mixed to an optimum consistency and placed into the disc-shaped perspex mould. The Turkom-Cera Alumina Gel was left in the mould for 24 hours (Figure 3.1A&B). After drying of the alumina gel, the discs were taken from the mould and sintered in the furnace (Programat p300, Ivoclar Vivadent AG, Schaan, Liechtenstein) for 5 minutes at 1150 °C (Figure 3.2A & B).



**Figure 3.1A & B:** The perspex mould used (A), and the perspex mould after 24 hours of placing Turkom-Cera alumina gel (B).



**Figure 3.2A & B:** The dried Turkom-Cera non sintered discs (A), and sintering using the Programat p300 furnace (B).

The Turkom-Cera crystal powder was mixed with water and the sintered discs were crystal hardened in a second firing process in the same furnace for 30 minutes at 1150 °C (Figure 3.3A & B). After firing, the excess crystals were removed with a diamond bur (Figure 3.4A & B). A total of 15 Turkom-Cera discs with 16 mm diameter and 2 mm thickness were fabricated.



**Figure 3.3A & B:** Mixing the Turkom-Cera crystal powder (A) and the sintered Turkom-Cera discs with crystal powder on top (B).



**Figure 3.4A & B:** Turkom-Cera disc before removal of excess crystals (A) and the finished Turkom-Cera disc after removal of excess crystals (B).

## **3.2.2.1.2** Preparation of In-Ceram discs

Perspex split mould with an open top and bottom five circular openings of 16 mm diameter and 2 mm thickness was used for the preparation of the In-Ceram disc specimens (Figure 3.5). The mould was rested and secured on a base made from gypsum die material (Densite, Shofu Inc., Kyoto, Japan).



Figure 3.5: The perspex mould used for In-Ceram discs preparation.

The In-Ceram alumina slip was prepared by mixing In-Ceram alumina powder with In-Ceram mixing fluid and additive supplied by the manufacturer. The slip was formed by sequential addition of alumina powder into the glass beaker containing In-Ceram mixing fluid and additive and vibration of the alumina and liquid mixture using In-Ceram Vitasonic unit (Figure 3.6).



**Figure 3.6:** Preparation of the In-Ceram alumina slip and vibration using the In-Ceram Vitasonic unit.

The slip was poured into the mould and dried for 24 hours. After drying, the In-Ceram alumina discs were taken from the mould and fired using the In-Ceramat furnace (Vita Zahnfabrik, Bad Säckingen, Germany) for 6 h at 120 °C and 4 h at 1120 °C (Figure 3.7).

The In-Ceram Glass Powder was mixed with water and the sintered In-Ceram alumina discs were glass infiltrated in a second firing process in the same furnace for 30 minutes at 200 °C and 4 hours at 1100 °C. Excess glass was removed with a diamond bur. A total of 15 In-Ceram discs of 16 mm diameter and 2 mm thickness were fabricated.



Figure 3.7: Vita In-Ceramat furnace used for the firing of In-Ceram discs.

### **3.2.2.1.3** Preparation of Vitadur-N discs

According to Abu-Hassan (1998) and the results of the preliminary study, Vitadur-Alpha and Vitadur-N porcelain discs of initial diameter 18 mm shrunk to 15.5-16 mm in diameter when fired. Therefore, a brass split mould with five circular opening of 18 mm diameter was used for the preparation of the Vitadur-N disc specimens (Figure 3.8).



**Figure 3.8:** The brass split mould and compactor used for the preparation of the Vitadur-N disc specimens.

Vitadur-N aluminous core porcelain powder (Vita, Bad Säckingen, Germany) was mixed with Vita modeling liquid P (Lot 10070) to an optimum slurry consistency. The slurry was placed into the brass mould and vibrated to reduce air bubbles (Figure 3.9). A brass compactor (Figure 3.8) was also machined and used to condense the slurry into the mould in order to obtain a flat surface.



Figure 3.9: The brass split mould with the condensed slurry of Vitadur-N.

The condensed slurry was left in the mould for 30 minutes and any excess liquid were blotted away with absorbent tissue. A layer of Vita Modisol separating medium was applied to the mould before the porcelain mixture was poured to facilitate removal of the set porcelain without any distortion. The disc specimens were then fired according to the manufacturer's recommendation in a vacuum furnace (Multimat-Touch & Press, Dentsply, Dreieich, Germany) (Figure 3.10). The furnace was programmed to give a temperature of 1120 °C for 60s under vacuum followed by a further 60s at atmospheric pressure. The discs were allowed to cool to room temperature, and any discs with visible surface imperfections were discarded. A total of 15 Vitadur-N aluminous core porcelain discs with 15.5-16 mm diameter were fabricated.



Figure 3.10: Multimat-Touch & Press furnace used for the firing of Vitadur-N discs.

### **3.2.2.2** Grinding and polishing of the specimens

# **3.2.2.1** Preparing the specimens for biaxial flexural strength testing

In order to meet the exact requirements of the biaxial testing protocol recommended by ISO 6872-1995, all specimens were subsequently grinded to a parallel shape with diamond discs using the grinder/polisher machine (Metaserv<sup>®</sup> 2000, Buehler, UK) (Figure 3.11A & B). A custom made specimen holder made from aluminum was designed and used for the grinding purpose. Eight specimens were fixed into the specimen holder using modeling wax (Figure 3.12A & B). The initial grinding was performed under running water using a diamond grinding disc with a grain size of 70  $\mu$ m, followed by fine- grinding using a grain size of 30  $\mu$ m. Once the specimens were grinded on one side, they were turned upside down and grinded in the same way on the other side. This yielded plane-parallel specimens with better precision.



**Figure 3.11A & B:** The grinding machine used (A) and the diamond disc fixed in the grinding machine (B).



**Figure 3.12:** The ceramic discs fixed to the specimen holder (A) and the specimen holder during grinding (B).

After that, the specimens were polished using a 15  $\mu$ m diamond polishing paste (DIAMAT<sup>TM</sup>, PACE Technologies, Tucson, USA) with lubricant (DIALUBE LAPPING LUBE (32 oz), PACE Technologies, Tucson, USA) on a polishing cloth for two minutes (Figure 3.13). The specimens were then cleaned in an ultrasonic bath (Delta Ultrasonic Cleaner D150, Taiwan Delta New Instrument Co..ltd., Dongguan, China) (Figure 3.14) containing distilled water for 3 minutes and then air dried.



Figure 3.13: The diamond paste, lubricant and polishing cloth used.


Figure 3.14: Ultrasonic cleaner machine used.

The ceramic discs were fabricated following the International Standards Organization specification for the testing of dental ceramic materials (ISO 6872-1995). According to International Standard Organization specification for dental ceramic (ISO 6872-1995), the specimens were trimmed to  $1.2 \pm 0.2$ mm in thickness with parallelism of  $\pm 0.05$ mm measured with the digital caliper (Mitutoyo Corp, Tokyo, Japan) (Figure 3.15).



Figure 3.15: The Mitotuyo digital caliper used.

### **3.2.2.2 Preparing the specimens for microhardness testing**

The specimens for microhardness testing were prepared using the following procedure:

• The specimens were mounted inside epoxy resin using plastic mould (Figure 3.16).



Figure 3.16: Specimens for microhardness testing mounted inside the epoxy resin.

- Specimens were wet-ground to a flat and smooth surface using a Metaserv® 2000 Grinder (Figure 3.11) at 300 revolutions per minute (rpm) with a series of silicon carbide (SiC) abrasive papers in sequence (No. 320, 400, 600, 800 and 1000 grit, Buehler Ltd., Lake Bluff, IL).
- Then the specimens were polished with 15 μm diamond polishing paste on polishing cloth using a Metaserv® 2000 Grinder/Polishing machine.
- Finally the specimens were cleaned in ultrasonic bath (Delta Ultrasonic Cleaner D150) containing distilled water for 3 minutes and air-dried.

### 3.2.2.3 Testing procedure

### 3.2.2.3.1 Biaxial flexural strength testing

Ten ceramic discs of each ceramic system (Turkom-Cera, In-Ceram and Vitadur-N) were subjected to biaxial flexural strength testing using the Instron Universal Testing Machine (Instron 4302, Instron Corporation, England) (Figure 3.17).



Figure 3.17: Instron Universal Testing Machine.

Piston-on-three-ball test was used for the testing. In order to carry out the test, a loading pin and mounting jig were designed and used with the Instron Testing Machine (Figure 3.18A & B).



Figure 3.18A & B: The 1.6 mm loading pin used (A) and the specimen's mounting jig used (B).

The loading pin was cylindrical in shape with a diameter of 1.6 mm. The mounting jig had a circular opening 16 mm in diameter with three depressions positioned at equal distances from each other (120° apart) and 5 mm from the center forming a tripod. These depressions were the sites for the 3.2 mm stainless steel ball bearing supports. The loading pin and the jig were mounted on the Instron Testing Machine and the discs were placed in the jig which ensured the same relation between the supports and the applied load for all specimens.

After 24 hours storage in distilled water at 37 °C, each specimen was dried and placed in the mounting jig which ensured the same relation between the supports and the applied load for all specimens. Then, the 1.6 mm diameter loading pin was mounted to the crosshead of the Instron Testing Machine and applied the load at the center of each specimen. The test was carried out at a crosshead speed of 0.5 mm/min. The definitive fracture load was recorded for each specimen and the biaxial flexural strength was calculated from the following equation (ISO-6872, 1995):

Biaxial flexural strength = $-0.238 7P(X - Y)/d^2$
$\mathbf{X} = (1+\nu)\ln(r_2/r_3)^2 + [(1-\nu)/2](r_2/r_3)^2$
$\mathbf{Y} = (1+\nu)[1+\ln(r_1/r_3)^2] + (1-\nu)(r_1/r_3)^2$
Where
<b>P</b> is the total load causing fracture (N)
<b>v</b> is Poisson's ratio (0.25)
$\mathbf{r_1}$ is the radius of the support circle (5.0mm)
$\mathbf{r_2}$ is the radius of the loaded area (0.8mm)
$\mathbf{r}_3$ is the radius of the specimen (8mm)
<b>d</b> is the specimen thickness at the origin of fracture (mm)

#### 3.2.2.3.2 Hardness testing

Five ceramic discs of each ceramic system (Turkom-Cera, In-Ceram and Vitadur-N) were selected to measure Vickers microhardness. The surface hardness of the specimens was determined using a Vickers microhardness (VHN) indenter (HMV, Shimadzu Corp., Tokyo, Japan) connected to a computer (Figure 3.19).

After 24 hours storage in distilled water at 37 °C, each specimen was mounted horizontally on the stage of the Vickers microhardness tester and a higher magnification objective of 40X was used to adjust and bring into focus the centre of the ceramic disc. Then, auto fine focusing was performed using the software provided with the HMV Micro Hardness Tester.

Indentations were conducted on the polished surface of the specimens using the Vickers diamond pyramid under a contact load of 9.8 N (1 kg) for 15 seconds (Figure 3.20 & Figure 3.21). For each load cycle three different indentations were carried out, and the mean Vickers hardness value was then calculated in VHN for each specimen. These indentations were placed at equal distance from each other and not closer than 1 mm to the adjacent ones or to the margin of the specimens.



Figure 3.19: HMV Micro Hardness Tester used.



Figure 3.20: Vickers diamond pyramid indenter while indent is being placed.



Figure 3.21: Micrograph of Vickers indentation in one of the ceramic discs tested.

### 3.2.2.4 Statistical analysis

Descriptive statistics were computed for biaxial flexural strength and Vickers microhardness. In order to compare biaxial flexural strength or Vickers microhardness between Turkom-Cera, In-Ceram and Vitadur N all-ceramic materials, One Way ANOVA test should be conducted provided that the assumptions will be met.

The assumption of normality will be tested by histogram and Shapiro-Wilk test and the assumption of equal variances of biaxial flexural strength or Vickers microhardness between groups will be tested using Levene's test. Whenever significant differences found, a post hoc test using Tukey's HSD test will be performed to test which pair of groups differ from each other significantly.

Then, in a case where assumptions were not met, an equivalent nonparametric Kruskal-Wallis test will be conducted. Whenever significant differences found, Mann-Whitney U tests will be performed on each pair of groups and the *p* value will be adjusted with the Bonferroni method. The SPSS software package (SPSS Inc., Chicago, IL) was used to perform the statistical analysis. Statistical significance will be set at  $\alpha$ = 0.05.

# 3.3 Results

### 3.3.1 Biaxial flexural strength

Specimens were divided into three groups according to ceramic system used; Group 1 (Turkom-Cera), Group 2 (In-Ceram) and Group 3 (Vitadur N). Descriptive analysis was performed and recorded in Table 3.1.

					95% Confidence Interval		
n	n	Mean (SD)	Median (IQR)	Lower Bound	Upper Bound		
Turkom-Cera	10	506.8 (87.01)	501.46 (141.91)	444.59	569.08		
In-Ceram	10	347.4 (28.83)	348.45 (56.10)	326.73	367.98		
Vitadur N	10	128.7 (12.72)	127.91 (16.41)	119.64	137.84		

**Table 3.1:** The mean and median biaxial flexural strength (MPa) of Turkom-Cera, In-<br/>Ceram and Vitadur N

Due to unmet assumption where the histogram and Shapiro-Wilk test (Appendix II) showed a non-normal distribution of biaxial flexural strength among the 3 groups, nonparametric Kruskal-Wallis Test was then used to compare the biaxial flexural strength of Turkom-Cera, In-Ceram and Vitadur N. Results were shown in Table 3.2. There was a significant difference between biaxial flexural strengths of the 3 tested groups (p<0.001).

Ceramic	n	Mean (SD)	Median (IQR)	Chi-Square ( <i>df</i> )	<i>P</i> value <sup>a</sup>	
Turkom-Cera	10	506.8 (87.01)	501.46 (141.91)	25.301	<0.001	
In-Ceram	10	347.4 (28.83)	348.45 (56.10)	(2)		
Vitadur N	10	128.7 (12.72)	127.91 (16.41)			
<sup>a</sup> Kruskal Wallis Test was used.						
Significant level wa	Significant level was set at 0.05.					

**Table 3.2:** Comparison of biaxial flexural strength (MPa) between Turkom-Cera, In-<br/>Ceram and Vitadur N by Kruskal Wallis Test

A Post hoc test using Mann-Whitney tests with Bonferroni correction as multiple pairwise comparisons (Appendix II) revealed that there were significant differences in biaxial flexural strength between Turkom-Cera and In-Ceram (p<0.001), Turkom-Cera and Vitadur-N (p<0.001) and also between In-Ceram and Vitadur-N (p<0.001). Results are summarized in Table 3.3. Turkom-Cera has the highest mean biaxial flexural strength (506.8 ± 87.0 MPa), whereas, Vitadur-N has the lowest mean biaxial flexural strength (128.7±12.7 MPa) and In-Ceram was in between (347.4 ± 28.8 MPa).

<b>D</b> · · · · ·			Р
Pairewise comparison	Mean (SD)	Median (IQR)	value <sup>a</sup>
Turkom-Cera	506.8 (87.01)	501.46 (141.91)	< 0.001 v
vs In-Ceram	347.4 (28.83)	348.45 (56.10)	< 0.001*
Turkom-Cera	506.8 (87.01)	501.46 (141.91)	< 0.001*
vs Vitadur N	128.7 (12.72)	127.91 (16.41)	< 0.001*
In-Ceram	347.4 (28.83)	348.45 (56.10)	< 0.001*
vs Vitadur N	128.7 (12.72)	127.91 (16.41)	< 0.001*
<sup>a</sup> Mann-Whitney Test was used	ł.		
* Bonferroni correction was us	sed.		

 Table 3.3: Multiple pairwise comparisons of bi-axial flexural strength (MPa) using Mann-Whitney Test with Bonferroni correction

### 3.3.2 Vickers microhardness

Specimens were divided into three groups according to ceramic system used; Group 1 (Turkom-Cera), Group 2 (In-Ceram) and Group 3 (Vitadr-N). The mean Vickers microhardness and standard deviation for each group are presented in Table 3.4.

Table 3.4:	The mean Vickers microhardness (VHN) of Turkom-Cera, In-Ceram and
	Vitadur N

		Mean			95% Confidence Interval for	
Ceramic	n	(VHN)	SD	Me	an	
			, 	Lower Bound	<b>Upper Bound</b>	
Turkom-Cera	5	1002.1	55.17	933.61	1070.63	
In-Ceram	5	1116.2	72.25	1026.51	1205.92	
Vitadur-N	5	812.8	41.88	760.81	864.80	

Since the distribution of Vickers microhardness was normally distributed as indicated by histogram and Shapiro-Wilk test (Appendix II) and the variances were equal by Levene's test (p=0.287, Appendix II), thus, One Way ANOVA was performed to compare Vickers microhardness between Turkom-Cera, In-Ceram and Vitadur N allceramic systems. The results were tabled in table 3.5. There was a significant difference in Vickers microhardness between the three groups (p<0.001).

Ceramic	n	Mean (VHN)	SD	F Statistics (df)	<i>P</i> value <sup>a</sup>		
Turkom-Cera	5	1002.1	55.17				
In-Ceram	5	1116.2	72.25	35.166	< 0.001		
Vitadur-N	5	812.8	41.88	(2,12)			
<sup>a</sup> One Way ANOVA was used.							
Significant level was	Significant level was set at 0.05.						

**Table 3.5:**Comparison of Vickers microhardness (VHN) between Turkom-Cera, In-<br/>Ceram and Vitadur N by One Way ANOVA

Further analysis using Tukey's HSD test was done to determine the pair of means that differ significantly (Table 3.6). The test indicates that all three group means differ from each other significantly. Based on these findings, it is concluded that In-Ceram has the highest mean Vickers microhardness ( $1116.2 \pm 72.3$  VHN), whereas, Vitadur-N has the lowest mean Vickers microhardness ( $812.8 \pm 41.9$  VHN) and Turkom-Cera was in between ( $1002.1 \pm 55.2$  VHN).

Pairewise comparison	Mean (SD)	Mean Difference	<i>P</i> value				
Turkom-Cera	1002.1 (55.17)	-114.1	0.022*				
vs In-Ceram	1116.2 (72.25)	114.1	0.022*				
Turkom-Cera	1002.1 (55.17)	189.3	0.001*				
vs Vitadur N	812.8 (41.88)	-189.3	0.001*				
In-Ceram	1116.2 (72.25)	303.4	<0.001*				
vs Vitadur N	812.8 (41.88)	-303.4	<0.001*				
* 2 pairs of means are signified	* 2 pairs of means are significantly different by Tukey's HSD Test						

 Table 3.6:
 Multiple pairwise comparisons of microhardness (VHN) by Tukey's HSD

### 3.4 Discussion

Strength is an important mechanical property that can assist in predicting the performance of brittle materials (Zeng et al., 1998).

The uniaxial flexural strength tests, including three-point, and four-point bending tests, and biaxial bending tests are the most commonly applied methods for evaluating the strength of dental restorations (Zeng et al., 1996; Zeng et al., 1998; Tinschert et al., 2000, Yilmaz et al., 2007). For uni-axial flexural strength tests, the principal stress on the lower surfaces of the specimens is tensile, and it is usually responsible for crack initiation in brittle materials. Therefore, undesirable edge fracture (which can increase the variance of the failure stress value) can occur. Since the principal stress for uni-axial flexural strength tests concentrated on the edges of the specimen, this will facilitate crack initiation and reduce specimen strength (Ban and Anusavice, 1990; Zeng et al., 1996; Jin et al., 2004). In a biaxial flexural strength test, the influence of edge flaws are eliminated, as the disc edges are located in a low stress area (Abu-Hassan et al., 1998).

The method adapted in this study was the one recommended by ISO [6872, 1995 (E)] since the test standardizes specimen thickness, diameter, shape and roughness. In addition, the measurement of the strength of brittle materials under biaxial flexural strength conditions rather than uni-axial flexural strength is often considered more reliable because the maximum tensile stresses occur within the central loading area and edge failures are eliminated (Anusavice et al., 2007). Therefore, the biaxial flexural test should produce less variation in the strength data (Wagner and Chu, 1996; Wen et al., 1999).

According to ISO [6872, 1995 (E)], the biaxial flexural strength is determined by supporting a disc specimen on three metal spheres positioned at equal distances from each other and from the center of the disc. The load is applied to the center of the opposite surface by a flat piston. The disc specimens can be easily made under typical restorative conditions. Furthermore, the flat surface of the test specimen can be easily controlled by conventional metallographic polishing methods and typical dental finishing techniques.

Due to the relatively low strength of the conventional porcelain jacket crowns, an alumina-reinforced porcelain core material was developed by McLean for the fabrication of jacket crowns (McLean and Hughes, 1965). These alumina reinforced crowns were regarded as providing better esthetics for anterior teeth than metal ceramic crowns, but they exhibited a lower flexural strength, which limit their use for posterior teeth (Rizkalla and Jones, 2004).

In-Ceram Alumina (VITA Zahnfabrik) has a high strength ceramic core fabricated through the slip-casting technique (Haselton et al., 2000). A slurry of densely packed  $Al_2 O_3$  (80-82 wt%) is applied and sintered to a refractory die at 1120°C for 10 hours (Haselton et al., 2000; Conrad et al., 2007). This produces a porous skeleton of alumina particles which is infiltrated with a low-viscosity glass in a second firing at 1100°C for 4 hours.

Advances in dental ceramics include the introduction of a high-strength all-ceramic core material (Turkom-Cera), particularly with aluminium oxide. The preparation of Turkom-Cera all-ceramic copings in the dental laboratory does not need more than standard laboratory furnace, propane gas flame, standard laboratory micromotor and Turkom-Cera all-ceramic kit. The stone die is covered by a red plastic foil 0.1 mm thick and dipped in the Turkom-Cera Alumina Gel (99.98%) following the manufacturer's instructions. After drying of the alumina gel, the coping with the red plastic foil is removed from the stone die and sintered for 5 minutes at 1150 °C. Then, the sintered coping is crystal hardened in a second firing process for 30 minutes at 1150 °C.

According to results of the biaxial flexural strength test, the Turkom-Cera core material had the highest strength value and the Vitadur-N core material had the lowest strength value. Statistical analysis carried out using Dunnett T3 post-hoc test at a pre-set significance level of 5 % revealed that Turkom-Cera has a significantly higher biaxial flexural strength (506.8±87 MPa) than In-Ceram (347.4±29 MPa) and Vitdur-N (128.7±13 MPa). Furthermore, the biaxial flexural strength of Turkom-Cera and In-Ceram was significantly higher than Vitadur-N.

Different researchers have studied the biaxial flexural strength of In-Ceram core using the same methods as the current study. The biaxial flexural strength of In-Ceram has been found to be in the average of 337.5 MPa (Wagner and Chu, 1996; Wen et al., 1999; Yilmaz et al., 2007). The mean biaxial flexural strength value for In-Ceram (347.4 MPa) achieved in the current study is in agreement with these results. The biaxial flexural strength of Vitadur-N core material has been investigated using the same methods as the current study and found to vary from 123.5 to 155 MPa (Seghi et al., 1990; Fleming et al., 1999; Fleming and Narayan, 2003). The mean biaxial flexural strength value for Vitadur-N (128.7 MPa) achieved in the current study is in agreement with these results.

The higher flexural strength achieved with Turkom-Cera and In-Ceram may be attributed to the following (Giordano et al., 1995; Wagner and Chu, 1996; Wei and Becher, 1984; Taya et al., 1990; Clarke, 1992):

- 1. Decreasing of the total porosity by initial firing (sintering) of Turkom-Cera alumina gel and In-Ceram alumina slip.
- 2. The alumina particles increase the strength of the material and limit potential sites for crack propagation.
- Prevention of the growth of cracks by crack bridging. The crystals and glass powders in combination with alumina may bridge the opening created by a crack after the crack front passes.
- 4. Compressive stresses which further improve the strength are also introduced due to the differences in the coefficient of thermal expansion of the alumina and crystals/glass.

The Turkom-Cera and In-Ceram are all-ceramic systems that incorporate a high alumina core. The difference in the strength of Turkom-Cera and In-Ceram shown in this study may be related the type of crystal powder and alumina used in Turkom-Cera. Turkom-Cera used alumina in the gel form whereas In-Ceram used alumina powder to be mixed with fluid and additive supplied by the manufacturer. Manual mixing of alumina powder may not produce the same consistent mixture as alumina gel. According to manufacturer, the maximum particles size of Turkom-Cera crystal powder is 80 micron compared to 200 micron for In-Ceram glass powder. Therefore, larger particles size will make the glass powder infiltration into the porous pre-sintered ceramic difficult, which might affect the final strength of the material.

This study also investigated the surface microhardness of all-ceramic materials being evaluated. The microindentation 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 (Poskus et al., 2004). The Vickers microindentation hardness test uses a square-based pyramidal-shaped diamond indenter with face angles of 136° and hence a square shaped impression is obtained in the material being tested (ASTM C1327-03). Measurements are then made on both diagonals and the mean values are obtained (Quinn, 1998; Sakar-Deliormanli and Güden, 2006).

The indentation load used in this study was 9.8 N, which is in accordance with the standards of the American Society for Testing and Materials (ASTM C1327-03).

In this study, all specimens for microhardness testing were grinded with a sequence of steps that ranged from 320 to 1000 grit and polished using 15  $\mu$ m diamond polishing paste in order to obtain a representative microindentation hardness numbers since no effects of sample grinding or polishing damage can be tolerated (Sakar-Deliormanli and Güden, 2006).

Hardness is a property of restorative materials that is generally considered important to applications involving friction and wear. A hard material will scratch, abrade and wear away opposing tooth structure. Therefore, it is desirable for a restoration to be able to resist abrasion and have a wear rate equal to that of enamel. In addition, the restorative material should not increase the wear rate of an opposing enamel surface. Materials with low hardness will probably not damage the natural antagonists (Seghi et al., 1991).

In general, conventional feldspathic dental porcelain is more abrasive of enamel than other restorative materials, such as gold or acrylic resin. An in vitro study by Mahalick et al., (1971) reported enamel-porcelain wear to be 2.4 times greater than wear of enamel-acrylic resin and 17 times that of enamel-gold. However, the rate of tooth substance wear has been found to be a function of roughness. Monasky & Taylor (1971), tested a variety of surface finishes of porcelain against tooth substance and concluded that the rate of tooth substance wear was a function of porcelain roughness. They recommended glazing or polishing porcelain to reduce enamel attrition. A ceramic restorative material that combines good strength without the disadvantage of increased enamel wear would be a significant addition to clinical dental practice (Willems et al., 1993).

The results of this study indicated that In-Ceram (1116.21 VHN) had the highest microhardness compared to Turkom-Cera (1002.12 VHN) and Vitadur-N (812.81 VHN) all-ceramic materials. Statistical analysis carried out using ANOVA and Tukey's HSD multiple range test, at 0.05 significance level, revealed significant differences between the three all-ceramic materials tested.

Guazzato et al., (2002) reported a microhardness value of 1173 VHN (11.5 GPa) for In-Ceram. The same author has reported in two studies a microhardness value of 1122 VHN (11 GPa) for In-Ceram. The value of 1122 VHN (11 GPa) reported by Guazzato et al., (2004a) and Guazzato et al., (2004b) is in excellent agreement with the microhardness value reported for In-Ceram (1116.21 VHN) in the present study.

Seghi et al., (1995) and Al-Shehri (2002), reported Vickers microhardness for different all-ceramic materials. Their values of 842.3 VHN (8.26 GPa) and 805.5 VHN (7.9 GPa), respectively, for Vitadur-N are in agreement with the value of Vitadur-N (812.81 VHN) found in the present study. Morena, et al. (1986) reported microhardness value for Vitadur-N of 675 VHN (6.62 GPa), however a load of 500 g was used in their study, while load of 1000 g was used in the present study.

Laboratory studies have attempted 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 relate 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. Despite the high strength reported with high alumina-based ceramics, they are susceptible to fatigue failure that can considerably reduce their strength over time. The other limitation of this study is that, the influence of fatigue in the oral cavity was not considered. Therefore, further studies are highly recommended to evaluate the fracture analysis and fatigue behavior of the new all-ceramic material Turkom-Cera.

# 3.5 Conclusions

In this study, the biaxial flexural strength and hardness of three all-ceramic core materials were tested in vitro. Within the limitations of this in vitro study, the findings disclosed the following:

- Turkom-Cera (506.8±87.01 MPa) had significantly higher flexural strength over In-Ceram (347.4±28.83 MPa) and Vitadur N (128.7±12.72 MPa) all-ceramic materials. Thus, the null hypothesis was rejected.
- In-Ceram (1116.21±72.25 VHN) had significantly higher microhardness compared to Turkom-Cera (1002.12±55.17 VHN) and Vitadur-N (812.81±41.88 VHN) all-ceramic materials. Thus, the null hypothesis was rejected.

**CHAPTER FOUR** 

# EFFECT OF LUTING CEMENTS AND SURFACE TREATMENTS ON THE BOND STRENGTH TO TURKOM-CERA<sup>TM</sup> ALL-CERAMIC MATERIAL

# 4.1 Introduction

Proper selection and application of luting agents for final cementation of all-ceramic restorations are key factors for their clinical success. Bonding of ceramic to dental tissue is based on the adhesion of luting cement to the ceramic substrate, together with the adhesion of luting cement to enamel and/or dentine. Long-term stable ceramic-resin bonds rely on chemical bonds and micromechanical interlocking at the resin- ceramic interface (Blatz et al., 2004).

Studies have documented the rationale for using conventional luting cements with the Procera AllCeram system (Odén and Andersson, 1998; Prestipino et al., 1998). In addition, the manufacturer of high-strength ceramics (egs: Procera AllCeram and Turkom-Cera) recommended using conventional luting cements for luting their restorations.

Clinical trials on full-coverage high-strength ceramic restorations reported acceptable success rates with conventional luting agents (Oden et al., 1998; Odman and Andersson, 2001). However, in the event of compromised retention or marginal seal, even high-strength ceramic crowns might benefit from adhesive bonding with a composite resin luting agent. Several in vitro and in vivo studies on this topic recommended adhesive cementation of ceramic and even high-strength ceramic restorations (Burke, 1995; Malament and Socransky, 1999b; Burke et al., 2002; Blatz et al., 2003a, Hill, 2007; Pegoraro et al., 2007).

Previous studies have revealed that most clinical failures would initiate from the cementation or internal surfaces. Failure rates due to high-strength ceramic fractures have been reported to range between 2.3 % and 8 % (Andersson and Oden, 1993; Özcan et al., 2001; Özcan and Vallittu, 2003). Therefore, the integrity of the luting cement to

ceramic surfaces plays a major role in the longevity of the restoration; the failures originating from cementation surfaces identified the need for a reliable conditioning method to strengthen this critical area.

In order to enhance the bond strength of luting cement to the ceramic surface, different surface treatments on ceramic surface have been recommended such as sandblasting, etching with different acids and grinding with diamond burs (Kern and Thompson, 1995; Friederich and Kern, 2002; Janda et al., 2003; Valandro et al., 2006). All of these procedures are intended to improve the bond strength by producing micromechanical retention and thus modifying the porcelain surface texture (Stangel et al., 1987; Filho et al., 2004). In addition to this mechanically retentive surface, the use of silane-coupling agent provides a chemical interaction, which is attributed to its bifunctional characteristic. A high proportion of porcelain allows reaction of the silane agent both to the crystal portion of the treated porcelain and to the organic portion of the luting agent (Nagayassu et al., 2006; Kim et al., 2006; Matinlinna and Vallittu, 2007a).

The effect of different luting cements and surface treatments on the bond strength to Turkom-Cera<sup>TM</sup> all-ceramic material have not been studied. Therefore, the objectives of this study were:

- 1. To determine the shear bond strength of Turkom-Cera luted with different cements.
- 2. To compare the effect of various surface treatments on the shear bond strength of Turkom-Cera when luted with resin-based cement.
- 3. To investigate the association between shear bond strength and modes of failure.

#### Null hypotheses

- There is no difference in the shear bond strength of different luting cements to Turkom-Cera.
- There is no effect of various surface treatments on the shear bond strength of Turkom-Cera when luted with resin-based cement.
- 3. There is no association between shear bond strength and mode of failure.

# 4.2 Materials and methods

### 4.2.1 Materials used

Four types of luting agents were used; zinc phosphate cement (Elite, GC Corporation, Tokyo, Japan), glass ionomer cement (Fuji I, GC Corporation, Tokyo, Japan), resin modified glass ionomer cement (Fuji Plus, GC Corporation, Tokyo, Japan) and resin luting cement (Panavia-F, Kuraray Medical Inc., Okayama, Japan) with its silane coupling agent (Table 4.1). Seventy Turkom-Cera (Turkom-Ceramic (M) Sdn Bhd, Puchong, Malaysia) discs 10 mm in diameter and 3 mm thick were prepared and used in this study.

Luting Cement	Brand	Manufacturer	Lot No.
Zinc phosphate cement	Elite	GC Corporation, Tokyo, Japan	Liquid: 0407071 Powder: 0407061
Glass ionomer cement	Fuji I	GC Corporation, Tokyo, Japan	Liquid: 0511041 Powder: 0511041
Resin modified glass ionomer cement	Fuji Plus	GC Corporation, Tokyo, Japan	Powder: 0704121 Liquid: 0704111
Resin luting cement	Panavia F	Kuraray Medical Inc., Okayama, Japan	A paste: 00245D B paste: 00140B
Silane coupling agent	Clearfil Silane Kit	Kuraray Medical Inc., Okayama, Japan	Primer: 00589A Activator: 00184A

 Table 4.1:
 Luting materials used

# 4.2.2 Methods

### **4.2.2.1** Specimen preparation before surface treatment and bonding

Perspex split mould with five circular openings of 10 mm diameter and 3 mm thickness was used for the preparation of the Turkom-Cera disc specimens (Figure 4.1).

The same procedures used in section 3.2.2.1.1 were used for the preparation of Turkom-Cera disc specimens. A total of seventy Turkom-Cera ceramic discs with 10 mm diameter and 3 mm thickness were prepared.



Figure 4.1: Perspex mould with five holes of 10 mm diameter and 3 mm thickness.

To ensure accurate shear bond strength testing of the ceramic-cement interface, each specimen was embedded in a die stone using plastic mould 30 mm in diameter and 30 mm high (Figure 4.2A & B). The specimens were fixed into the plastic moulds using double sided tape and embedded with die stone (Densite, Shufo, Japan) (Figure 4.3). The bonding surface of the specimens was at the same level of the embedding medium to form one flat surface.

After hardening for 24 hours at room temperature, the bonding surface of the specimens were sanded with a series of silicon carbide (SiC) abrasive papers in sequence (No. 400, 600, 800 and 1000 grit, Buehler) using a water-irrigated lapping machine (Metaserv<sup>®</sup> 2000, Buehler, UK) until the ceramic disc was perfectly flushed with the mounting mould and a flat surface was attained (Figure 4.4).





Figure 4.2A & B: Turkom-Cera disc fixed to the plastic mould before embedding with the die stone.



Figure 4.3: Turkom-Cera disc embedded in the die stone.



**Figure 4.4:** Lapping the specimen with a Metaserv<sup>®</sup> 2000.

All specimens were rinsed under running water and dried before further treatment. The ground bonding surface was examined under microscope (Zoom Stereo EMZ-1, MEIJI Techno Co., Ltd., Saitama, Japan) at 30x magnification to ensure that no abrasive particles were left on the surface.

### 4.2.2.2 Surface treatments and sample distribution

Four luting cements (Elite, Fuji I, Fuji Plus & Panavia F) were used to evaluate the effect of different luting cements on the bond strength to Turkom-Cera. In addition, the following surface treatments have been applied:

- I. Polishing with silicone carbide paper up to 1000 grit (control).
- **II.** Polishing with silicone carbide paper up to 1000 grit + Sandblasting.
- **III.** Polishing with silicone carbide paper up to 1000 grit + Silane.
- **IV.** Polishing with silicone carbide paper up to 1000 grit + Sandblasting + Silane.

According to the luting cements and surface treatments used, seven different groups were evaluated.

Group 1: Sandblasting + Zinc Phosphate cementGroup 2: Sandblasting + Glass ionomer cementGroup 3: Sandblasting + Resin modified glass ionomer cement

Group 4: Sandblasting + Resin cement

Group 5: Silane + Resin cement

**Group 6:** Sandblasting + Silane + Resin cement

**Group 7:** Control + Resin cement

### 4.2.2.3 Bonding procedure

All samples were mounted and secured on the shear bond test apparatus recommended by ISO/TS 11405/2003 in order to bond a uniform amount of cement onto the Turkom-Cera bonding surface (Figure 4.5A & B).

The alignment apparatus consists of a holder for the specimen, a cylindrical split brass mould (Figure 4.6A & B) resulting in samples with a defined bond area of 3 mm diameter and 3 mm height, a silicone pad and an added load of 1 kg (Kern and Thompson, 1995; and Friederich and Kern, 2002).



Figure 4.5A & B: Front (A) and lateral (B) views of the shear bond test apparatus used.



Figure 4.6A & B: Open (A) and closed (B) views of the cylindrical split brass mould used.

All cements were mixed according to manufacturer's instructions at room temperature (24°C). A fresh standard mix (full spoon of powder to three drops of liquid) of zinc-phosphate cement (Elite) was prepared. Zinc phosphate cement was mixed slowly to a constant creamy consistency on a cooled, dry and heavy mixing glass slab over a wide area for 60 seconds.

A fresh standard mix (full spoon of powder to two drops of liquid) of glass ionomer cement (Fuji I) was prepared. The powder and liquid were dispensed onto the mixing pad provided by the manufacturer and mixed rapidly for 20 seconds.

A fresh standard mix (one level small scoop of powder to one drop of liquid) of resin modified glass ionomer cement (Fuji Plus) was prepared. The powder and liquid were dispensed onto the mixing pad provided by the manufacturer and mixed rapidly for 20 seconds.

Equal amount of Panavia F paste A and paste B was dispensed on the mixing pad provided by the manufacturer and mixed for 20 seconds. Care was taken to ensure that there was no water mist on the mixing pad or spatula before using them because the presence of water could shorten the working time of the mixed paste. The paste was used within 3 minutes after mixing.

Sandblasting was performed with 50- $\mu$ m aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles at an air pressure of 2.5 bars for 13 seconds from a distance of 10 mm (Sadan et al., 2003). The discs were then steam cleaned and air dried.

The silane coupling agent used is a mixture of Clearfil Porcelain Bond Activator and Clearfil SE Bond Primer. One drop each of Porcelain Bond Activator and Clearfil SE Bond Primer was dispensed into a well of the mixing dish and mixed together. The mixture was applied to the surface and left for 5 seconds. Then the volatile ingredients were evaporated with gentle air flow. The mixture was prepared immediately before application. The cements were placed, using a plastic instrument, into the 3 mm diameter hole in the brass split mould while it was slightly raised to ensure a uniform flow onto the bonding surface and to avoid trapping of air bubbles. The brass split mould was carefully adapted to the bonding surface by raising the mounted specimen using the screw at the bottom of the mounted specimen. The split mould together with the mounted specimen was then quickly secured on to the bonding apparatus and tightly screwed (Figure 4.7A & B). A sharp blade was used to remove the excess cement before setting from the top of the brass split mould. A layer of Oxyguard II (oxygen-blocking gel) was applied in the case of Panavia F.



**Figure 4.7A & B:** Brass mould adapted to the bonding jig (A), and cement placement (B).

Specimens were allowed to set under a constant load of 1 kg for 15 minutes using a polyvinylsiloxane (Express putty, 3M ESPE, St. Paul, MN, USA) putty mould that was placed over the brass split mould and held in place by the weight (Figure 4.8). The 1 kg load was removed and the samples were allowed to set at room temperature for an additional 30 minutes with the polyvinylsiloxane mould still in place.

The samples were carefully removed from the apparatus by unscrewing the horizontal screw while securing the sample with hand to allow the bond to remain undisturbed. The brass split mould was separated using a sharp blade and the excess cement was removed with a scalpel blade to standardize the bonding area. The final bonded specimen is shown in Figure 4.9. Then, the specimens were stored in distilled water at 37°C for 24 h before testing.



**Figure 4.8:** load application during bonding.



Figure 4.9: Bonded specimen.

### 4.2.2.4 Testing procedure

The bonded specimens were mounted in an ISO/TS 11405 shear test jig and tested using a universal testing machine (Instron® Corp., England) (Figure 4.10A & B). The shear test jig consists of a solid block for holding the specimen and a vertical shearing blade with a 0.5-mm blunt edge.



Figure 4.10A & B: Shear jig (A) attached to the Instron Universal Testing Machine (B).

The 0.5-mm knife-edge shearing blade was mounted on the crosshead of the instron testing machine and applied a shearing load to the adhesive interface at a crosshead speed of 1 mm/min. The knife-edge shearing blade was placed at a distance of approximately 0.5 mm above the bonded specimen at their adhesive interface (Figure 4.11).



Figure 4.11: Specimen during shear bond strength testing.

The maximum load at failure was recorded in Newton, and the shear bond strengths of the specimens were calculated and expressed in MPa by dividing the force (N) at which the bond failure occurred by the bonding area (mm<sup>2</sup>).

### 4.2.2.5 Assessment of mode of failure

The bonded surfaces were observed under a microscope (Zoom Stereo EMZ-1, MEIJI Techno Co., Ltd., Saitama, Japan) at 30X magnification to evaluate adhesive and cohesive failure modes. According to many researchers (Kamada et al., 1998; Friederich and Kern, 2002; Blatz et al., 2004; Piwowarczyk et al., 2004), failures were categorized as follows:

- a) Adhesive failure at the ceramic-cement interface.
- b) Cohesive failure within the cement or ceramic.
- c) Mixed failure: combination of adhesive and cohesive failures.

#### 4.2.2.6 Statistical analysis

The first objective was to determine the shear bond strength of Turkom-Cera luted with different luting cements (Elite/ Fuji I/ Fuji Plus/ Panavia F). The second objective was to compare the effect of various surface treatments (no further treatment/ sandblasting/ silane/ sandblasting+silane) on the shear bond strength of Turkom-Cera when luted with resin-based cement. A separate analysis for each objective will be performed.

Descriptive statistics of shear bond strength were performed. To compare shear bond strength between the four groups (luting cements / surface treatments), One Way ANOVA test should be conducted. However, this is dependent on the assumptions to be met. The assumption of normal distribution of shear bond strength should be tested by histogram and Shapiro-Wilk test. Following that, the Levene's test should be conducted to test for equal variances of shear bond strength between the four groups. Whenever significant differences found, a post hoc test using Tukey's HSD test will be performed to test which pair of groups differ from each other significantly.

Then, in a case where the assumptions were not met, an equivalent nonparametric Kruskal-Wallis test will be conducted. Subsequently, a post hoc test using Mann-Whitney tests with Bonferroni correction will be conducted to test which pair of groups differ from each other significantly.

Regarding the association between shear bond strength and modes of failure, descriptive statistics for modes of failure and shear bond strengths will be recorded and the result will be descriptively analyzed. The SPSS software package (SPSS, SPSS Inc., Chicago, IL) was used to perform the statistical analysis. Statistical significance will be set at  $\alpha$ = 0.05.

# 4.3 Results

### **4.3.1** Effect of luting cements on the shear bond strength

Specimens were divided into four groups according to luting cements used; Group 1: zinc phosphate cement (Elite), Group 2 glass ionomer cement (Fuji I), Group 3: resin modified glass ionomer cement (Fuji Plus) and Group 4: resin cement (Panavia F). Descriptive analysis was performed and the mean and median shear bond strength for all groups are presented in Table 4.2.

**Table 4.2:** The mean and median shear bond strength (MPa) for the four luting cements used

				95% Confidence Interval		
Cement	n	Mean (SD)	Median (IQR)	Lower Bound	<b>Upper Bound</b>	
Elite	10	0.92 (0.42)	0.95 (0.59)	0.62	1.22	
Fuji I	10	2.04 (0.78)	2.11 (1.21)	1.48	2.60	
Fuji Plus	10	4.37 (1.18)	4.22 (1.06)	3.52	5.22	
Panavia F	10	16.42 (3.38)	15.92 (4.20)	14.01	18.84	

Since the distribution of shear bond strength was not normally distributed as indicated by histogram and Shapiro-Wilk test (Appendix III), nonparametric Kruskal-Wallis Test was then done to compare the shear bond strength between Elite, Fuji I, Fuji Plus and Panavia F. Results were shown in Table 4.3. There was a significant difference in shear bond strength between the four groups (p<0.001).

**Table 4.3:**Comparison of shear bond strength (MPa) between Elite, Fuji I, Fuji Plus<br/>and Panavia F by Kruskal Wallis Test

Cement	n	Mean (SD)	Median (IQR)	Chi-Square ( <i>df</i> )	<i>P</i> value <sup>a</sup>	
Elite	10	0.92 (0.42)	0.95 (0.59)			
Fuji I	10	2.04 (0.78)	2.11 (1.21)	34.837	< 0.001	
Fuji Plus	10	4.37 (1.18)	4.22 (1.06)	(3)		
Panavia F	10	16.42 (3.38)	15.92 (4.20)			
<sup>a</sup> Kruskal Wallis Test was used.						
Significant leve	el was	set at 0.05.				

\* Details on statistical analysis see Appendix III

Further analysis using Mann-Whitney Post hoc test with Bonferroni correction as multiple pairwise comparisons (Appendix III) revealed that there were significant differences between shear bond strength of Elite and Fuji I (p=0.018), Elite and Fuji Plus (p<0.001), Elite and Panavia F (p<0.001), Fuji I and Fuji Plus (p<0.001), Fuji I and Panavia F (p<0.001) and also between Fuji Plus and Panavia F (p<0.001). Results are summarized in Table 4.4.

Pairewise comparison	Mean (SD)	Median (IQR)	P value <sup>a</sup>		
Elite	0.92 (0.42)	0.95 (0.59)	0.019*		
vs Fuji I	2.04 (0.78)	2.11 (1.21)	0.018		
Elite	0.92 (0.42)	0.95 (0.59)	<0.001*		
vs Fuji Plus	4.37 (1.18)	4.22 (1.06)	<0.001		
Elite	0.92 (0.42)	0.95 (0.59)	<0.001*		
vs Panavia F	16.42 (3.38)	15.92 (4.20)	<0.001**		
Fuji I	2.04 (0.78)	2.11 (1.21)	<0.001*		
vs Fuji Plus	4.37 (1.18)	4.22 (1.06)	<0.001**		
Fuji I	2.04 (0.78)	2.11 (1.21)	-0.001*		
vs Panavia F	16.42 (3.38)	15.92 (4.20)	<0.001*		
Fuji Plus	4.37 (1.18)	4.22 (1.06)	<0.001*		
vs Panavia F	16.42 (3.38)	15.92 (4.20)	<0.001**		
<sup>a</sup> Mann-Whitney Test was used.					
* Bonferroni correction was used	1.				

**Table 4.4:**Multiple pairwise comparisons of shear bond strength (MPa) of the four<br/>luting cements using Mann-Whitney Test with Bonferroni correction

### 4.3.1.1 Testing mode of failure

A cross-tabulation was performed between the four treatment groups (Elite, Fuji I, Fuji Plus and Panavia F) and modes of failure (Table 4.5). The Chi-square test (Appendix III) was used to test if there is any association between treatment groups and modes of failure. Due to unmet assumption of Chi-Square test and non-meaningful combination of different modes the result can only be descriptively analyzed.

	Mode of		
	Adhesive	Mixed	
Cement	n (%)	n (%)	Total
Elite	10 (100%)	0 (0%)	10
Fuji I	10 (100%)	0 (0%)	10
Fuji Plus	10 (100%)	0 (0%)	10
Panavia F	7 (70%)	3 (30%)	10

**Table 4.5:** Distribution of modes of failure in each treatment group (Elite, Fuji I, Fuji<br/>Plus and Panavia F)

As shown in Table 4.5, we can conclude that with Elite, Fuji I and Fuji Plus, the modes of failure were 100% adhesive mode. While for Panavia F, the modes of failure were only 70% adhesive mode.

Descriptive summary for modes of failure and shear bond strengths was performed (Table 4.6). The identified modes of failure were: adhesive and mixed.

**Table 4.6:** Descriptive summary for modes of failure and shear bond strengths (MPa)<br/>(effect of luting cements)

	Elite		Fuji I		Fuji Plus		Panavia F	
Failure mode	n	Mean SBS (SD)	n	Mean SBS (SD)	n	Mean SBS (SD)	n	Mean SBS (SD)
Adhesive	10	0.92 (0.42)	10	2.04 (0.78)	10	4.37 (1.18)	7	14.79 (2.17)
Mixed	0	0	0	0	0	0	3	20.25 (2.43)

(SBS: shear bond strength)

As shown in Table 4.6, the shear bond strength for the adhesive mode of failure was in ascending order; Elite (0.92 MPa), Fuiji I (4.04 MPa), Fuji Plus (4.37 MPa) and Panavia F (14.79 MP). In general, the shear bond strength for the mixed mode of failure (20.25 MPa) was higher compared to that of the adhesive mode (0.92 to 14.79 MPa).
### **4.3.2** Effect of surface treatments on the shear bond strength

Specimens were divided into four groups according to surface treatment used; Group 1: Polished with silicone carbide paper up to 1000 grit (Control), Group 2: Sandblasting, Group 3: Silane and Group 4: Sandblasting + Silane. The mean shear bond strength and standard deviation for all groups are given in Figure 4.12.



Figure 4.12: The mean shear bond strength (MPa) of the four treatment groups.

Since the assumption of normal distribution of shear bond strength of the four groups was met as indicated by histogram and Shapiro-Wilk test (Appendix III) and the variances were equal by Levene's test (p=0.110, Appendix III), One Way ANOVA was performed to compare shear bond strength between the four groups. The results were shown in Table 4.7. There was a significant difference in shear bond strength between the four groups (p<0.001).

 Table 4.7:
 Comparison of shear bond strength (MPa) between the four surface treatments by One Way ANOVA

				F Statistics	Р
Treatment	n	Mean	SD	(df)	value <sup>a</sup>
Control	10	10.83	1.45		
Sand blasting	10	16.42	3.38	18.64	< 0.001
Silane	10	16.18	2.54	(3,36)	
Sand blasting + Silane	30	19.13	2.43		
<sup>a</sup> One Way ANOVA was used.					
Significant level was set at 0.05.					

\* Details on statistical analysis see Appendix III

Multiple comparisons using Tukey HSD Post Hoc Test was used to determine the pair of means that differ significantly (Table 4.8). Based on Tukey HSD test, the control group (10.8±1.5 MPa) showed significantly lower mean shear bond strength than the other three groups (p<0.001). There was no significant difference between the mean shear bond strength of the other three groups, sandblasting (16.4±3.4 MPa), Silane (16.2±2.5 MPa) and sandblasting + silane (19.1±2.4 MPa) (p>0.050).

Pairewise comparison	Mean (SD)	Mean Difference	P value				
Control	10.83 (1.45)	-5.59	<0.001*				
vs Sandblasting	16.42 (3.38)	5.59					
Control	10.83 (1.45)	-5.35	0.001*				
vs Silane	16.18 (2.54)	5.35					
Control	10.83 (1.45)	-8.29	<0.001*				
vs Sandblasting+Silane	19.13 (2.43)	8.29					
Sandblasting	16.42 (3.38)	0.24	0.10				
vs Silane	16.18 (2.54)	-0.24					
Sandblasting	16.42 (3.38)	-2.7	0.10				
vs Sandblasting+Silane	19.13 (2.43)	2.7					
Silane	16.18 (2.54)	-2.94	0.06				
vs Sandblasting+Silane	19.13 (2.43)	2.94					
* 2 pairs of means are significantly different by Tukey's HSD Test							

**Table 4.8**:Multiple pairwise comparisons of shear bond strength (MPa) of the four<br/>surface treatments using Tukey's HSD Test

### 4.3.2.1 Testing mode of failure

A cross-tabulation between treatment groups and modes of failure was obtained (Table 4.9). The objective is to test if there is any association between treatment groups and modes of failure. The Chi-square test\* was used to test if there is any association between modes of failure and treatment groups (Appendix III). Due to unmet assumption of Chi-Square test and non-meaningful combination of different modes the result can be only descriptively analyzed.

	Mode of		
	Adhesive		
Treatment	n (%)	n (%)	Total
Control	10 (100%)	0 (0%)	10
Sand blasting	7 (70%)	3 (30%)	10
Silane	7 (70%)	3 (30%)	10
Sand blasting + Silane	5 (50%)	5 (50%)	10

**Table 4.9:** Distribution of modes of failure in each treatment group

From the above table, it is clear that the modes of failure were 100% adhesive in the control group. The sandblasting and Silane groups showed 70% adhesive mode for each. While in the Sandblasting + Silane group, the modes of failure were only 50% adhesive.

Descriptive summary for modes of failure and shear bond strengths was recorded (Table 4.10). The identified modes of failure were: adhesive and mixed. As shown in Table 4.10, the shear bond strength for the adhesive mode of failure was in ascending order; Control (10.83 MPa), Silane (14.67 MPa), Sandblasting (14.79 MPa) followed by Sandblasting + Silane (16.93 MP) group. However, the shear bond strength for the mixed mode of failure was in ascending order; Silane (19.72 MPa), Sandblasting (20.25 MPa) followed by Sandblasting + Silane (21.32 MP). Overall, the shear bond strength for the adhesive mode of failure (19.72 to 21.32 MPa) was higher compared to that of the adhesive mode (10.83 to 16. 93 MPa).

 Table 4.10:
 Descriptive summary for modes of failure and shear bond strengths (MPa) (effect of surface treatments)

							Sandblasting +		
		Control	Sa	Sandblasting		Silane		Silane	
Failure		Mean SBS	_	Mean SBS		Mean SBS		Mean SBS	
mode	n	( <b>SD</b> )	n	( <b>SD</b> )	n	( <b>SD</b> )	n	( <b>SD</b> )	
Adhasiya	1	10.83	7	14.79	7	14.67	5	16.93	
Adhesive	0	(1.45)	/	(2.17)	/	(0.84)	5	(0.52)	
Minod	0	0	2	20.25	2	19.72	5	21.32	
Mixed		3	(2.43)	3	(0.31)	3	(1.00)		

(SBS: shear bond strength)

\* Details on statistical analysis see Appendix III

## 4.4 Discussion

#### 4.4.1 Methodology

Adhesion between tooth structure and the restoration is one of the most important factors determining the success of a restoration (Toman et al., 2008). Laboratory tests of restorative materials are very important, since constant improvement of new adhesive systems makes long-term clinical trials difficult. Once a product is proven to be unsuitable for use, it might no longer be on the market (Braem et al., 1994).

In the present study, shear bond test was used to evaluate the bond strength of luting cements to Turkom-Cera. In addition, the same test was used to evaluate the effect of different surface treatments on the bond strength of resin cement to Turkom-Cera. The test method is fast and easy to perform. The sample preparation is also easier than for tensile measurement and there are only minor influences from variations in loading direction (Watanabe and Nakabayashi, 1994). However, it can be questioned whether a tension test might be more appropriate for testing the bond strength of luting cements to ceramics (Della Bona and van Noort, 1995). But, as the intention was to evaluate relative differences between luting cements and pretreatment techniques and how they could result in improved bonding, which may be of clinical interest, the method used was thought to be appropriate (Derand et al., 2006).

It has been shown in several studies that thermocycling and long-time storage in water decrease bond strength between resin luting materials and ceramic surfaces, as well as tooth materials (Kern and Thompson, 1995; Kato et al., 1996; Lu et al., 2001; Blatz et al., 2003d; Hummel and Kern, 2004).

On the other hand, several studies have found that neither water storage nor thermal cycling significantly altered the bond strength of resin cement to ceramic specimens (Kamada et al., 1998; Hooshmand et al., 2002; Filho et al., 2003). In this study, the principal objective was to study the main improvements in bond strength using different luting cements and surface treatments. Thus, as a first step, only dental materials and no ageing processes were used to study the bonding strength. Furthermore, ceramic constructions are subjected to high loads immediately after placement, so it is important to study the bond strength after surface pretreatment and without simulated ageing processes (Derand et al., 2006).

Removal of the limited amount of glassy matrix present in glass infiltrated alumina ceramic materials by etching did not result in a significant amount of microscopic retentive features (Madani et al., 2000). Hydrofluoric acid etching did not create a microretentive surface on the high alumina core ceramics, and it was almost ineffective for etching of the glass phase for micromechanical bonding (Özcan et al., 2001). Therefore, sandblasting is an alternative method for surface roughening.

Air particle abrasion with  $50-\mu m Al_2O_3$  at 2.5 bar was recommended as a preferred surface treatment method for glass-infiltrated aluminum-oxide ceramic and densely sintered aluminum-oxide ceramic (Awliya et al., 1998; Madani et al., 2000; Lu et al., 2001). The combination of air particle abrasion and the resin luting cement Panavia, which contains the adhesive phosphate monomer MDP, provided successful and predictable resin bonds to glass-infiltrated aluminum-oxide ceramic (Kern and Thompson, 1995; Madani et al., 2000; Lu et al., 2001; Nakamura et al., 2004), and densely sintered aluminum-oxide ceramic (Awliya et al., 1998; Blatz et al., 2003b). Therefore this study focused on this line of treatment.

During testing, the vertical shearing blade was approximated as closely as possible to the ceramic adhesive interface. If the blade were placed away from the adhesive interface, a bending movement would be created which could cause some deviations in the results. In this study, the distance from the vertical shearing blade to the adhesive interface was 0.5 mm in order to prevent displacement during loading (ISO/TS 11405:2003).

The width of the blade used for shear bond strength tests would also have some effect on the stress pattern produced during the test. It has been found that the smaller the contact interface, the greater the bond strength values. Oliveira et al., (2009) studied the effect of chisel width on shear bond strength of composite to dental enamel and found that the mean bond strength values increased more than 20 % when a 0.5 mm-width chisel was used in comparison with a 3.0 mm-width chisel. This suggests that there is a dissipation of tensions through the chisel/composite interface. According to ISO/TS 11405/2003, shearing blade with a width of 0.5 was recommended for shear bond strength testing. Therefore, a shearing blade with a 0.5 mm blunt edge was used in this study.

The bonded area for mechanical testing by different researchers had not been constant. It has been shown that smaller areas of testing produced higher shear bond strengths and less cohesive fractures in dentine. Pashley et al., (1995) reported that as cross-sectional area of a bonded specimen reduced, the number of cohesive failure of dentine decrease to zero at about 2 mm<sup>2</sup>. On the other hand, Fowler et al., (1992) stated that the shear and tensile bond strengths of adhesive resins had no significant relationship with the surface area of the bonded specimen. In this study, the bonded area was limited to 3 mm by

using a split mould with a central hole of 3 mm diameter in accordance with ISO (ISO/TS 11405, 2003).

The split mould used in this study helped in controlling the bonding area. Some researchers used adhesive tape to limit the bonding area. In either case it has been reported that the formation of a thin film was unavoidable. It has been demonstrated that these small extensions of material around the specimen can lead to concentration of stress at the periphery of the bonding cement (Van Noort et al., 1991). In this study, the excess cement was easily removed with a scalpel blade to standardize the bonding area.

The thickness of the specimen was controlled by using the brass split mould, which helped to ensure a consistent and uniform thickness (3 mm) of the specimens. In addition, the specimens were allowed to set under a constant load of 1 kg (Kern and Thompson, 1995; Friederich and Kern, 2002; Blatz et al., 2003b) for 15 minutes in accordance with ISO (ISO/TS 11405, 2003).

An in vitro study by Hara et al., (2001) studied the effect of crosshead speed on shear bond strength and concluded that the slower the crosshead speed, the lower the shear bond strength. At a slower speed, more time is available for crack to propagate and cause failure of bond at a lower threshold (Fairhust et al., 1993). ISO recommended a standard crosshead speed of  $0.75 \pm 0.30$  mm/min (ISO/TS 11405, 2003). Therefore, a crosshead speed of 0.5 mm/min was used in this study in accordance with the above recommendation.

#### **4.4.2** Effect of luting cements

This study was carried out to evaluate the shear bond strength of different luting cements (zinc phosphate cement Elite, glass ionomer cement Fuji I, resin modified glass ionomer cement Fuji Plus and resin luting cement Panavia F) to Turkom-Cera ceramic material.

The results of the current study indicated that the bond strength of resin luting cement Panavia F (16.42  $\pm$  3.4 MPa) to sandblasted Turkom-Cera discs was higher to that obtained by zinc phosphate cement Elite (0.92  $\pm$  0.4 MPa), glass ionomer cement Fuji I (2.04  $\pm$  0.8 MPa) and resin modified glass ionomer cement Fuji Plus (4.37  $\pm$  1.2 MPa). Statistical analysis using Dunnett's T3 post-hoc test at a pre-set significance level of 5 % showed statistically significant difference between the four luting cements tested. The mean shear bond strength of Panavia F was significantly higher than Elite, Fuji I and Fuji Plus (p<.05).

In interpreting the results of this study, one has to take into account the internal strength of the cement used. Ultimately, cement with a bond strength that competes with the strength of the cement or one of the substrates to be bonded to can be used. The tensile strength of zinc phosphate and glass ionomer cements is much lower than that of resin-modified glass ionomer cements, which have a lower strength compared to resin composite cements (Mitchell et al., 2000). This fact is reflected in the highest shear bond strength value of the resin cement tested. In general, the ranking of the bond strength results increased up from zinc phosphate cement to glass ionomer cement to resin-modified glass ionomer cement to resin luting cement. This trend may be related to the intrinsic strength of the cement. The higher the resin contents the higher the strength (Attin et al., 1996; Özcan et al., 2001).

The results of this study are in agreement with the results of other in vitro studies (Piwowarczyk et al., 2004; Begazo et al., 2004). Piwowarczyk et al., (2004) found that the shear bond strengths between sand basted high-strength aluminum oxide ceramic and resin cements were significantly higher than those of zinc phosphate, glass ionomer and resin-modified glass ionomer cements. Another in vitro study conducted by Begazo et al., (2004) found that the shear bond strength to aluminum oxide-reinforced glass ceramic material increases significantly from conventional glass ionomer cement, resin-modified glass ionomer cements to resin cement.

In vitro studies on bonding strengths of cements to dental ceramics differ within a wide range and assessment of their clinical significance is difficult. Since, the all-ceramic restoration is cemented to dentine, not only is the cement-ceramic interface important, but also the dentine-cement interface can be an important factor that determines the longevity of the restoration.

The shear bond strength of human dentine was found to be 13.4 MPa (Sengun et al., 2003). It has been also suggested that 10-13 MPa is the minimum strength needed for clinical bonding (Thurmond et al., 1994; Sengun et al., 2003; Begazo et al., 2004). On the other hand, the in vitro bond strengths to acid-etched human dentine of various commercial resin composite bonding cements, which have been in clinical use for a relatively long time, are reported to range from 1.1 MPa to 14.8 MPa (Piwowarczyk et al., 2007). The shear bond strength of dentine to Panavia F (Sengun et al., 2003) and Fuji Plus (Irie and Suzuki, 2001), were 7.7 MPa and 7.0 MPa, respectively. Due to variation in experimental set-up or preparatory procedures, the shear bond strengths reported in the literature are difficult for comparison. Nevertheless, the shear bond values reported are much lower than the shear bond strength values found for the ceramic-cement interface. A microtensile bond strength test of dentine and a Cerec 2

inlay cemented with Panavia F showed a similar result; de-bonding occurred more often at the cement-dentine interface than at the cement-inlay interface (Uno et al., 2000). This finding was also supported by another study (Strub and Beschnidt, 1998) which found that the resin composite had higher bond strength to the ceramic material than to the prepared dentine.

In short, based on the previous considerations the use of the resin cement Panavia F will give the most reliable bond to the ceramic material and fracture will most probably occur at the cement-dentine interface.

For conventional zinc phosphate cement, Øilo (1978) reported tensile bond strength to dentine of 0.6 MPa, whereas Richardsson et al., (1990) reported 0.9 MPa. Although these values seemed to be very low, and are considerably inferior to those suggested as the acceptable minimum strength for clinical bonding, zinc phosphate cements have been successfully used clinically for a very long time to lute cast dental restorations and currently recommended for luting high-strength ceramics (egs: Procera AllCeram and Turkom-Cera) (Prestipino et al., 1998; Oden et al., 1998; Odman and Andersson, 2001). To assess the clinical performance of bonding systems, in vitro studies should, therefore, be supplemented with clinical studies with long-term follow-up.

### 4.4.3 Effect of surface treatments

In order to have a reliable and satisfactory union between ceramic and resin cement, a combination of chemical and mechanical retention must occur. Porcelain surface treatments modify its texture, increasing the micromechanical retention of the resin cement. Chemical retention is achieved with the use of silane coupling agents that reacts with the vitreous compounds of the ceramic and with the composite organic matrix (Stangel et al., 1987; Filho et al., 2004).

Sandblasting, using aluminum oxide particles, is an alternative method for creation of micromechanical retention. This technique was utilized in the present study for being a commonly method employed to increase roughness of high-strength ceramic materials (Kern and Thompson, 1995; Awliya et al., 1998; Madani et al., 2000; Friederich and Kern, 2002; Özcan et al., 2008).

Surface roughening methods increase surface energy and, therefore, its wettability (Blatz et al., 2003a). Mechanical interlocking of the cements to roughened ceramic specimens will enhance bond strength values. The present study investigated the effect of different surface treatments on shear bond strengths of resin cement to the Turkom-Cera high-alumina core. The use of Panavia F directly on the polished ceramic surface showed statistically significantly lower bond strength than on the sandblasted ceramic surface.

The present study demonstrated that roughening of the Turkom-Cera surface increased the adhesion of resin cement. The data clearly showed that sandblasting the surface with alumina particles was effective surface treatment for producing high bond strength. The bond strength values differed significantly between the control group ( $10.8 \pm 15$  MPa) and the sandblasted group ( $16.4 \pm 3.4$  MPa). These results correspond to the findings of previous studies which have found a strong and durable bond between Panavia resin luting cement and air particle-abraded high-alumina ceramic (Madani et al., 2000; Friederich and Kern, 2002; Begazo et al., 2004; Hummel and Kern, 2004).

The mechanical retention provided by surface treatment is of paramount importance for proper adhesion. However, the association with a chemical procedure (silanization) is required for better results (Kato et al., 2001; Shimada et al., 2002; Blatz et al., 2003b; Begazo et al., 2004; Zohairy et al., 2004). Thus, a silane coupling agent was also used in the present study for the same reason.

An in vitro study by Matsumura and colleagues emphasized the importance of the compatibility between the silane coupling agent and the resin cement (Matsumura et al., 1997). Therefore, the current study used the silane coupling agent recommended by the manufacturer.

Silane coupling agents provide covalent chemical bonds between silica-ceramic surfaces and bonding agent/resin cements as well as a rewetting effect on the roughened ceramic surface. The exact role of rewetting effects, micromechanical interlocking, and chemical interaction of silane coupling and bonding agents on the resin bond to high-alumina ceramic surfaces is still unclear (Kern and Thompson, 19995; Blatz et al., 2003d).

In this study, the surface treatment by silane coupling agent improved the shear bond strength of Panavia F to polished ceramic surfaces. The group treated with silane coupling agent ( $16.2 \pm 2.5$  MPa) showed significantly greater shear bond strength than the non-silanated group (control) ( $10.8 \pm 1.5$  MPa). These results do not correspond with those of previous studies (Kern and Thompson, 1995; Friederich and Kern, 2002; Blatz et al., 2003d), which showed that resin cement performed better without silane coupling agent.

On the other hand, these results correspond to the results of other works done by Blatz et al., (2003b) & Nakamura et al., (2004), which found that the use of the recommended silane coupling agent with Panavia resulted in significantly higher bond strength values before and after long-term storage and thermocycling.

In this study, the specimens were silanated with a mixture of Clearfil Porcelain Bond Activator, which contained 3-methacryloxypropyl trimethoxysilane (γ-MPTS), and Clearfil SE Bond Primer, which contained acidic phosphate ester monomer 10methacryloyloxydecyl dihydrogen phosphate (MDP) that promotes the catalysis of silane reaction. In addition, the phosphate ester group of the adhesive monomer MDP bonds chemically to metal oxides such as aluminum oxides (Friederich and Kern, 2002; Blatz et al., 2003d; Yoshida et al., 2006). The exact bonding mechanisms and the role of these monomers when bonding to oxide-based ceramics are still unknown (Blatz et al., 2003d). However, the high bond strength may be attributed to ceramic oxide and ester bond and the mechanical properties of Panavia F cement (Sen et al., 2000).

It has been found that the presence of the glassy phase in ceramics facilitates better siloxane bonds (Valandro et al., 2006). The silanol groups then react further to form a siloxane (-Si-O-Si-O-) network with the silica on the surface. Turkom-Cera ceramic system tested in this study is based on glass infiltration technique. Most probably, the glass infiltration facilitated better silane bonding, and therefore, higher bond strength values were obtained for these ceramics.

The limitation of this study is that it could not be determined whether the bond was due to the specific surface configuration of Turkom-Cera; to the silane coupling agent, which is a content of the Clearfil Porcelain Bond Activator; or to the phosphate ester monomer MDP, which is also a content of the bonding agent Clearfil SE Bond Primer. Therefore further investigation is needed.

In the current study, a mean shear bond strength value up to  $19.1 \pm 2.4$  MPa was achieved with Panavia F resin cement on the sandblasted and silanated Turkom-Cera specimens. For specimens silanated with clearfil silane ( $16.2 \pm 2.5$  MPa) or sandblasted with 50-µm Al<sub>2</sub>O<sub>3</sub> particles ( $16.4 \pm 3.4$  MPa), the result was lower, but not significantly different. This finding confirmed that Panavia F cement in combination with silane or sandblasting is suitable for bonding Turkom-Cera material, and stressed the importance of the selection of appropriate surface treatments for optimal bonding. The bonding mechanism of Panavia F cement to sandblasted Turkom-Cera material treated with silane is unclear, even though Clearfil silane may show improved bonding from the condensation between trimethoxysilyl groups in the silane coupler and hydroxyl groups in the ceramic material (Madani et al., 2000).

#### 4.4.4 Mode of failure

The current study also addressed the issue of failure modes. With regard to the luting cements used with sandblasted Turkom-Cera ceramic, failure modes for zinc phosphate, glass ionomer and resin modified glass ionomer cements were completely adhesive between the cement–bonding substrate interface for all specimens.

After different surface treatments to Turkom-Cera specimens, fracture analysis regarding adhesive, cohesive or complex failures has been done. For the control group, the resin cement Panavia F showed completely adhesive failure for all specimens. Complex adhesive and cohesive failures were also seen in this study. When Turkom-Cera was treated with clearfil silane or abraded with 50-µm Al<sub>2</sub>O<sub>3</sub> particles, Panavia F has shown complex adhesive and cohesive failures in 30 % of specimens in each group. This was increased to 50 % when Panavia F cement was used with sandblasted and silanated Turkom-Cera specimens. Although complex failure was observed for the three treatment groups, higher bond strength was obtained by using Panavia F with sandblasted and silanated Turkom-Cera specimens.

Kern & Thompson, (1995) reported cohesive porcelain fractures using phosphate monomer containing composite resin (Panavia Ex). However, in this study pure ceramic and pure cement cohesive failures were not observed. Particularly interesting was the fact that all complex failures were seen in the specimens that exhibited high bond strengths of more than 18 MPa. It seems that, the bond strength values may be accountable for the modes of failure at the bonded interface (Chung and Hwang, 1997). This study has given rise to the tentative conclusion that higher bond strength values increase complex (adhesive and cohesive) failure modes.

### 4.5 Conclusions

- Within the limitations of this in vitro study, it was found that the mean shear bond strength between sandblasted Turkom-Cera ceramic and Panavia F was significantly higher than those of zinc phosphate, glass ionomer and resinmodified glass ionomer cements. Thus, the null hypothesis was rejected.
- 2. Within the results of this study, it was found that when using Panavia F resin cement and Clearfil silane; sandblasted Turkom-Cera specimens produced the highest mean shear bond strength values. Almost similar shear bond strength values were obtained for Turkom-Cera specimens when sandblasted with 50-μm Al<sub>2</sub>O<sub>3</sub> or silanated with clearfil silane. Therefore, these three surface treatments appeared to be the methods of choice for the cementation of Turkom-Cera restorations. Thus, the null hypothesis was rejected.
- 3. In this study, all complex failure modes were seen in the specimens that exhibited high bond strengths of more than 18 MPa. Therefore, it can be concluded that higher bond strength values increase complex (adhesive and cohesive) failure modes. Thus, the null hypothesis was rejected.

### **CHAPTER FIVE**

EVALUATION OF THE OCCLUSAL FRACTURE RESISTANCE OF TURKOM-CERA COMPARED TO TWO OTHER ALUMINA-BASED CERAMIC SYSTEMS (PART I): EFFECT OF DIFFERENT LUTING CEMENTS ON THE FRACTURE RESISTANCE OF TURKOM-CERA

## 5.1 Introduction

The patients' desire for all-ceramic restorations has increased because of their inherent esthetics, excellent biocompatibility and improved physical properties (Snyder and Hogg, 2005; Christensen, 1999; Blatz, 2002). However, most dental ceramics are brittle and tensile stresses caused by external loading can lead to the propagation of cracks starting at flaws and other defects (Seghi et al., 1995). This is more critical for posterior areas in the mouth, where the forces are much higher than for the anterior region and can reach 522 N in the average individual (Bakke et al., 1990; Pallis et al., 2004).

Various types of all-ceramic systems have therefore been developed in an attempt to improve these mechanical properties. These systems may be categorized according to their fabrication (pressable, slip casting, milling, or sintering) and material composition (feldspar: high leucite and low leucite; glass ceramic: lithium disilicate and mica; core reinforced: alumina, magnesia, and zirconia) (Qualtrough and Piddock, 2002; Yilmaz et al., 2007).

Several studies on fracture resistance of all-ceramic restorations have reported high fracture resistance comparable to that of metal-ceramic restorations (Diaz-Arnold et al., 1999; Komine et al., 2004; Pallis et al., 2004; Potiket et al., 2004, Yoshinari and Derand, 1994; Strub and Beschnidt, 1998). However, many factors such as the fabrication technique, the final surface finish of the restoration and the luting method could affect fracture resistance (Chen et al., 1999; Kelly, 1999; Attia and Kern, 2004).

It has been suggested that resin bonding of ceramic restorations to the supporting tooth structure increases retention, marginal adaptation and fracture resistance of the restoration (El-Mowafy, 2001; Sorensen et al., 1991; Burke, 1996; Yoshinari and Derand, 1994; Pagniano et al., 2005). Therefore, adhesive cementation may be

beneficial for high-strength ceramic full coverage restorations, especially in situations of compromised retention or high occlusal load.

Recent developments in the technology and research of new dental materials have resulted in the increase in number of materials available for esthetic restorations. Evaluations of the physical properties of the materials are necessary before these materials can be recommended for standard treatment (Potiket et al., 2004).

A new all-ceramic alumina core material, Turkom-Cera, was introduced in an attempt to provide a high-quality, high strength, cost effective coping that will result in improved clinical success. Independent studies of basic comparative data are necessary to characterize this new material in relation to mechanical properties. Laboratory studies are useful tools to identify preferred cementation methods and luting materials before they are used clinically (Awliya et al., 1998; Blixt et al., 2000). Therefore, the objectives of this study were to:

- Determine the occlusal fracture resistance of Turkom-Cera copings compared to In-Ceram and Procera AllCeram copings.
- Compare the effect of different luting cements on the occlusal fracture resistance of Turkom-Cera copings.
- 3. Assess mode of fracture of the copings.

#### Null hypotheses

- There is no difference in the occlusal fracture resistance of Turkom-Cera, In-Ceram and Procera AllCeram copings.
- There is no effect of different luting cements on the occlusal fracture resistance of Turkom-Cera.
- 3. There is no association between occlusal fracture resistance and mode of fracture.

# 5.2 Materials and methods

### 5.2.1 Materials used

Three types of all-ceramic systems namely, Turkom-Cera (Turkom-Ceramic (M) Sdn. Bhd., Kuala Lumpur, Malaysia), In-Ceram (Vita Zahnfabrik, Bad Sackingen, Germany) and Procera AllCeram (Nobel Biocare, Göteborg, Sweden), and three types of luting cements namely, zinc phosphate cement (Elite, GC Corporation, Tokyo, Japan), glass ionomer cement (Fuji I, GC Corporation, Tokyo, Japan), and resin luting cement (Panavia-F, Kuraray Medical Inc., Okayama, Japan) with its silane coupling agent (Clearfil Silane Kit, Kuraray), were used in this study (Table 5.1 and Figs. 5.1 - 5.2).

Table 5.1: Luting materials used in this study

Material	Manufacturer	Туре	Batch number
Elite	GC Corporation,	Zinc phosphate	Liquid: 0407071
cement	Tokyo, Japan	cement	Powder: 0407061
Fuji I	GC Corporation,	Glass ionomer	Liquid: 0511041
cement	Tokyo, Japan	cement	Powder: 0511041
Panavia F	Kuraray Medical Inc.,	Resin luting	A paste: 00245D
cement	Okayama, Japan	cement	B paste: 00140B
Clearfil	Kuraray Medical Inc.,	Silane coupling	SE bond primer: 00589A
Silane Kit	Okayama, Japan	agent	Porcelain bond activator: 00184A





**Figure 5.1:** Zinc phosphate cement Elite (left) and glass ionomer cement Fuji Plus (right) used.



**Figure 5.2:** Panavia F resin luting cement (left) and Clearfil silane coupling agent (right) used in this study.

## 5.2.2 Methods

A sound and crack-free human maxillary first premolar was selected to receive a complete-coverage all-ceramic crown preparation (Figure 5.3). The root surfaces were notched for anchorage and the roots were embedded in a stone block within 2 mm below the cemento-enamel junction. A dental surveyor (Figure 5.4a & b) was used to position the long axis of the tooth. An all-ceramic crown preparation was fabricated on the mounted maxillary first premolar.

### **5.2.2.1** Preparation of the tooth

A dental surveyor was modified and used to prepare the axial walls of the tooth to ensure a consistent degree of taper (Figure 5.4a & b). The handpiece was adapted to the horizontal arm of the surveyor in such a way that the long axis of the tapered round-ended carbide bur (850, 016, Komet, Germany) was parallel to that of the tooth. A tapered bur will impart an inclination of 2-3 degrees to any surface it cuts if the shank of the instrument is held parallel to the intended path of insertion of the preparation (Shillingburg et al., 1997). Therefore, the axial preparation was made with a wall angle of 2 - 3 degrees (4 - 6 degrees angle of convergence). A chamfer margin was prepared

by means of the round-ended tapered rotary instrument. All margins were placed above the cemento-enamel junction.



Figure 5.3: The embedded tooth.



Figure 5.4a & b: The dental surveyor used for positioning and preparation of the tooth.

After the completion of axial preparation, the occlusal surface of the tooth was cut flat 6.00 mm above the top of the stone block (Figure 5.4), with a carbide wheel (Komet No. 909, 204, 040, Komet, Germany). All sharp angles or internal line angles were rounded to prevent stress concentration in the copings. These series of reductions resulted in a tapered preparation with 4 - 6 degrees angle of convergence, a 1.0 mm circular chamfer margin and total preparation height of 4.00 mm (Figure 5.5a,b,c & d).



Figure 5.5 (a,b,c and d): Different views of the stone die.

### 5.2.2.2 Construction of metal dies

Six impressions of the prepared tooth were taken using a sectional tray and polyether impression material (Impregum Penta, 3M ESPE, Germany). The six impressions of the prepared tooth were poured in self-curing acrylic resin (Pattern Resin, GC, Japan) to produce six patterns of the prepared tooth. The six acrylic patterns were embedded in a base of wax (College Wax, Metrodent Limited, England) in order to be fitted in the jig (Figure 5.6) which was constructed to facilitate testing with the instron testing machine. Then the six acrylic patterns, with their wax bases, were sprued, invested and cast with a non-precious alloy (Wiron 99, BEGO, Germany) and finished to be the master dies (Figure 5.7). Cobalt-chromium alloy was used because of its markedly superior physical properties to porcelain, to ensure that the die will not break (Tuntiprawon and Wilson, 1995).



Figure 5.6: The acrylic patterns with their wax bases and the mounting jig.



Figure 5.7: The six metal dies used.

## 5.2.2.3 Fabrication of all-ceramic copings

### 5.2.2.3.1 Fabrication of Turkom-Cera copings

The fabrication of the Turkom-Cera all-ceramic copings does not require an industrial process due to the low temperatures and absence of shrinkage during fabrication process. It does not require more than the Turkom-Cera fused alumina kit, standard laboratory furnace, propane gas flame and a standard laboratory micromotor.

Following the Turkom-Cera technique for fabrication of all-ceramic coping discussed in chapter two, five impressions were taken for each of the six master dies using a sectional tray and polyether impression material (Impregum Penta, 3M, Germany) (Figure 5.8a). The thirty impressions were poured with die stone on the vibrator (Densite, Shufo, Japan) (Figure 5.8b).



Figure 5.8a & b: Taking impression of the metal die (a) and the stone dies (b).

The stone dies were precisely prepared so that the shapes and margins were clearly indicated. Using this technique, the stone die was covered by a red plastic foil 0.1 mm thick. The covered stone die was dipped in the Turkom-Cera Alumina Gel following the manufacturer's instructions.

After drying of the alumina gel, the coping with the red plastic foil was removed from the stone die and fired in the furnace (Programat p300, Ivoclar Vivadent) for 5 minutes at 1150 °C. The sintered Turkom-Cera coping was crystal hardened, using Turkom-Cera crystal powder, in a second firing process in the same furnace for 30 minutes at 1150 °C. The excess crystals were removed with a diamond bur. A total of thirty Turkom-Cera copings with a thickness of 0.6 mm were fabricated.

#### 5.2.2.3.2 Fabrication of In-Ceram copings

Two of the six master dies were randomly selected and five impressions were made for each master die using a sectional tray and polyether impression material Impregum. The ten impressions were poured on the vibrator with die stone.

In the dental laboratory, each stone die was duplicated by taking an impression using a plastic ring and an addition polymerization silicone material (Dent Silicone Plus, Shofu Inc., Kyoto, Japan). These impressions were poured with In-Ceram special plaster to make refractory models. The In-Ceram alumina slip was prepared by mixing In-Ceram alumina powder with In-Ceram mixing fluid and the additive supplied by the manufacturer. The slip was formed by sequential addition of alumina powder into the glass beaker containing In-Ceram mixing fluid and additive and vibration of the alumina and liquid mixture using the In-Ceram Vitasonic unit (Figure 5.9).



Figure 5.9: Preparation of the In-Ceram slip using the In-Ceram Vitasonic unit.

In-Ceram alumina slip was applied to the models following the manufacturer's instructions (Figure 5.10a). After applying a stabilizer, the copings were fired on the plaster dies in the furnace (Inceramat, Vita Zahnfabrik, Bad Säckingen, Germany) for 6 h at 120 °C and then followed by 4 h at 1120 °C (Figure 5.10b & c).



**Figure 5.10a,b & c:** Application of the In-Ceram alumina slip (a) and firing using Vita Inceramat furnace (b and c).

The In-Ceram copings were then glass infiltrated, using In-Ceram glass powder, in a second firing process in the same furnace for 30 minutes at 200 °C and then followed by 4 h at 1100 °C (Figure 5.11a & b). Excess glass was removed with a diamond bur. A total of ten copings with a thickness of 0.6 mm were fabricated.



**Figure 5.11a & b:** Application of the In-Ceram glass powder (a) and firing using Vita Inceramat furnace (b).

#### 5.2.2.3.3 Fabrication of Procera AllCeram copings

Two of the six master dies were randomly selected and one impression was made for each master die using a sectional tray and polyether impression material Impregum.

The two impressions were poured with die stone and processed to fabricate the master dies which were then trimmed, mounted and scanned utilizing the computerized Procera-Scanning Machine (Nobel Biocare) linked to a computer and modem (Figure 5.12a & b). The laboratory technician has set the finish line on the computer screen and determined the desired coping thickness (0.6 mm) and emergence profile to ensure the excellent marginal adaptation, fit and quality of the final restoration (Figure 5.13a,b,c & d). This detailed information was then forwarded to Nobel Biocare in Sweden, where five densely sintered aluminium oxide copings were manufactured with the same dimensions and thickness of 0.6 mm for each master die (total of ten Procera copings were fabricated from the two master dies).



**Figure 5.12a & b:** The Procera AllCeram Scanner connected to a computer and modem (a) and the die during scanning process (b).



Figure 5.13 a,b,c & d: Determination of the finish line and coping design.

During the industrial coping manufacturing process, the Procera AllCeram system fabricated a slightly enlarged dies and sintered the alumina onto these dies to compensate for shrinkage. Upon completion, the copings were evaluated to ensure quality. Once inspected and approved, they were sent back to the dental laboratory (Figure 5.14).



Figure 5.14: Schematic drawings of Procera AllCeram fabrication.

### 5.2.2.4 Crown cementation procedures

After the impressions were completed, the dies were first cleaned with acetone, steam cleaned and air dried prior to cementation of the first all-ceramic coping. After cementation and testing of the first coping, the cement on the dies was removed ultrasonically. Then the dies were steam cleaned and air dried prior to cementation of the following coping.

All copings were internally sandblasted with 50  $\mu$ m aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) particles at an air pressure of 2.5 bars for 13 seconds from a distance of 10 mm. The copings were then steam cleaned and air dried (Sadan et al., 2003). Sandblasting with 50  $\mu$ m aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) particles was found to be superior to other surface treatments as in the study by Awliya et al., (1998), and was also recommended by Madani et al., (2000). A full spoon of Elite (zinc phosphate cement) powder to three drops of liquid were mixed slowly at room temperature (24°C) to a constant creamy consistency on a cooled, dry and thick mixing glass slab over a wide area for 60 seconds. A full spoon of Fuji I (glass ionomer luting cement) powder to two drops of liquid were dispensed onto the mixing pad and mixed rapidly at room temperature (24°C) for 20 seconds. Then a coating of cement was applied to the internal surface of each coping.

With Panavia F (dual-cured composite resin cement) the ED primer was applied to the entire surface of the metal die and allowed to set for 60 seconds before air drying with gentle air flow. The fit surfaces of all copings were silanated with a mixture of Clearfil Porcelain Bond Activator and Clearfil SE Bond Primer. The mixture was applied to the internal surface of the coping and left for 5 seconds before air drying with gentle air flow. Sufficient amount of the Panavia F (one complete turn from each cartridge A &B) was dispensed, mixed for 20 seconds and applied to the internal surface of each coping.

Manual finger pressure was used to initially seat each crown on its die, and for the zinc phosphate and glass ionomer cements, each crown was held in place while any excess cement removed before the luting agent set completely. Whilst for resin luting cement (Panavia F), any excess paste remaining at the margins was removed with a disposable brush and a layer of Oxyguard II (Kuraray) was applied for three minutes around the margins of each specimen. The specimens were then placed in a custom-made vertical loading apparatus (Makramani Load), for 10 minutes under a 5 kg load (Figure 5.15). Following cementation, all specimens were placed in a sealed container of distilled water and left in an incubator at a constant temperature of 37°C for 24 hours.



Figure 5.15: Application of the load during cementation using the Makramani Load.

## 5.2.2.5 Testing procedure

#### 5.2.2.5.1 Effect of ceramic material on the fracture resistance

Three types of all-ceramic systems namely, Turkom-Cera, In-Ceram and Procera AllCeram were used for this test. Ten copings from each of the above three mentioned ceramics were cemented using Panavia F (dual-cured composite resin cement) onto their corresponding dies according to the manufacturer's instructions. Two master dies were used for each group and each of them was used to lute five copings.

### 5.2.2.5.2 Effect of luting materials on the fracture resistance of Turkom-Cera

Thirty Turkom-Cera copings and three different types of luting media, zinc-phosphate cement (Elite), glass-ionomer cement (Fuji I) and dual-cured composite resin cement (Panavia F) with its silane coupling agent (Clearfil Silane Kit, Kuraray), were used in this test. The specimens were divided into three groups according to the luting cements used. Ten copings were cemented with each type of the above mentioned luting agents. Two master dies were used for each group and each of them was used to lute five copings. All copings were cemented onto their corresponding dies according to the manufacturer's instructions.

The master die with cemented coping was removed from the storage container and mounted in specially designed jig and subjected to testing on the Instron Testing Machine. A 1.6 mm stainless steel bar mounted on the crosshead of the Instron Testing Machine was used and applied a compressive load at the center of the occlusal surface, along the long axis of the cemented copings, at a crosshead speed of 1mm/min until fracture was observed (Figure 5.16). The maximum force to produce fracture was recorded in Newtons. The fractured crowns were removed and the master die was ultrasonically cleaned before a new coping was cemented. The force at failure was noted and the failed coping examined in order to determine the mode of fracture.



Figure 5.16: Loading of a crown with a 1.6 mm stainless steel bar.

### 5.2.2.6 Assessment of mode of fracture

The mode of fracture was classified using categories originally described by Burke & Watts (1994) (Table 5.2 and Figure 5.17).

Mode of fracture	Description				
Ι	Minimal fracture or crack in coping.				
II	Less than half of coping lost.				
III	Coping fracture through midline (half of coping displaced or lost).				
IV	More than half of coping lost.				
V	Severe fracture of coping and/or die.				

(After Burke and Watts, 1994)



**Figure 5.17:** Description of modes of fracture. (After Burke and Watts, 1994)

#### 5.2.2.7 Statistical analysis

The first objective was to determine the load at fracture of Turkom-Cera copings compared to In-Ceram and Procera AllCeram copings. The second objective was to compare the effect of different luting cements (Elite, Fuji I and Panavia F) on the load at fracture of Turkom-Cera copings. A separate analysis for each objective will be performed.

Descriptive statistics were recorded for load at fracture of each group. To compare load at fracture between the three groups (ceramic materials / luting cements), One Way ANOVA test should be conducted. However, this is dependent on the assumptions to be met. The assumption of normal distribution of load at fracture should be tested by histogram and Shapiro-Wilk test. Following that, the Levene's test should be conducted to test for equal variances of load at fracture between the three groups. Whenever significant differences were found, a post hoc test using Scheffe's test will be performed to test which pair of groups differ from each other significantly.

Whenever assumptions were not met, an equivalent nonparametric Kruskal-Wallis test will be conducted. Subsequently, a post hoc test using Mann-Whitney tests with Bonferroni correction will be conducted to test which pair of groups differ from each other significantly.

Regarding the association between load at fracture and modes of fracture, descriptive statistics for modes of fracture and load at fracture will be recorded and the result will be descriptively analyzed. Statistical analysis will be carried out using a computer program (SPSS, SPSS Inc., Chicago, IL). Statistical significance will be set at  $\alpha$ = 0.05.

# 5.3 Results

### **5.3.1** Effect of ceramic material on the fracture resistance

The objective is to test whether the mean load at fracture of Procera AllCeram, Turkom-Cera and In-Ceram copings cemented with resin luting cement (Panavia F) differs from each other. Descriptive analysis was performed and the mean load at fracture and standard deviation for all groups are recorded in Table 5.3.

 Table 5.3:
 The mean load at fracture (N) of Procera AllCeram, Turkom-Cera and In-Ceram copings

		Moon		95% Confid	95% Confidence Interval		
Ceramic	n	(N)	SD	Lower Bound	Upper Bound		
Procera AllCeram	10	1953.5	210.68	1802.79	2104.21		
Turkom-Cera	10	2183.6	164.09	2066.22	2300.98		
In-Ceram	10	2041.7	200.43	1898.32	2185.08		

The histogram and Shapiro-Wilk test were used and showed that there was a normal distribution of load at fracture of the three groups (Appendix IV). Since the assumption of normal distribution was met, the equality of variances (homogeneity) was tested using the Levene's test (Appendix IV) and showed that there was no significant deviation from homogeneity (p=0.601). Thus, the parametric One Way ANOVA procedure was used to achieve objective. The results were recorded in Table 5.4. There was a significant difference in load at fracture between the three groups (p=0.040).

**Table 5.4:** Comparison of load at fracture (N) between Procera AllCeram, Turkom-<br/>Cera and In-Ceram by One Way ANOVA

Ceramic	n	Mean (N)	SD	F Statistics (df)	P value <sup>a</sup>		
Procera AllCeram	10	1953.5	210.68				
Turkom-Cera	10	2183.6	164.09	3.626	0.040		
In-Ceram	10	2041.7	200.43	(2,27)			
<sup>a</sup> One Way ANOVA was used.							
Significant level was set at 0.05.							

\* Details on statstcal analysis see Appendix IV
Further analysis using Scheffe's Post Hoc Test was done to determine the pair of means that differ significantly (Appendix IV). Based on Scheffe's Post Hoc Test (Table 5.5), the mean load at fracture of Turkom-Cera (2183.6 ± 164.09 N) was significantly higher than Procera AllCeram (1953.5 ± 210.68 N) (p=.042). Furthermore, there was no significant difference in load at fracture between In-Ceram (2041.7 ± 200.43 N) and Procera AllCeram (p=0.598) and also between Turkom-Cera and In-Ceram (p=0.275).

Pairewise comparison	Mean (SD)	Mean Difference	P value			
Procera AllCeram vs Turkom-Cera	1953.5 (210.68) 2183.6 (164.09)	-230.10 230.10	0.042*			
Procera AllCeram vs In-Ceram	1953.5 (210.68) 2041.7 (200.43)	-88.20 88.20	0.598			
Turkom-Cera vs In-Ceram	2183.6 (164.09)141.902041.7 (200.43)-141.90		0.275			
* 2 pairs of means are significantly different by Scheffe's Test						

Table 5.5: Multiple pairwise comparisons of load at fracture (N) using Scheffe's Test

#### **5.3.1.1** Testing mode of fracture

A cross-tabulation between treatment groups (Procera, Turkom-Cera and In-Ceram) and modes of fracture was obtained (Table 5.6). The Chi-square test was used to test if there is any association between treatment groups and modes of fracture (Appendix IV). Due to unmet assumption of Chi-Square test and non-meaningful combination of different modes the result can be only descriptively analyzed.

Examination of the mode of fracture of specimens revealed that (80 %) of Turkom-Cera and (80 %) Procera AllCeram copings exhibited minimal fracture (Figure 5.18). Whereas, only 60 % of In-Ceram copings exhibited minimal fracture.

		Mode of Fracture						
	Less than MinimalMore than half of coping lostSevere fracture of die and/or coping							
Ceramic	n (%)	n (%)	n (%)	n (%)	Total			
Procera	8 (80%)	0 (0%)	1 (10%)	1 (10%)	10			
Turkom-ceram	8 (80%)	1 (10%)	0 (0%)	1 (10%)	10			
In-ceram	6 (60%)	3 (30%)	0 (0%)	1 (10%)	10			

**Table 5.6:** Distribution of modes of fracture in each treatment group (Procera, Turkom-Cera and In-Ceram)

Descriptive summary for modes of fracture and mean load at fracture was recorded (Table 5.7). The identified modes of fracture were: minimal fracture (crack line), less than half of coping lost, more than half of coping lost and severe fracture of die and/or coping.

	Procera		Turkom-Ceram		In-Ceram	
Mode of Fracture	n	Mean LAF (SD)	n	Mean LAF (SD)	n	Mean LAF (SD)
Minimal fracture	8	1911.88 (215.40)	8	2191.63 (182.85)	6	1982.67 (111.67)
Less than half of coping lost	0	0	1	2205.00	3	2009.33 (189.06)
More than half of coping lost	1	2068.00	0	0	0	0
Severe fracture of die and/or coping	1	2172.00	1	2098.00	1	2493.00

**Table 5.7:** Descriptive summary for modes of fracture and mean load at fracture (N) (effect of ceramic materials)

(LAF: load at fracture)

As shown in Table 5.7, the minimal fracture mode occurred at a higher load with Turkom-Cera group (2191.63 N) followed by In-Ceram (1982.67 N) and Procera (1911.88 N) groups. Furthermore, the minimal fracture mode occurred at lower load than sever fracture mode with Procera and In-Ceram groups. However, the minimal fracture mode occurred at higher load than sever fracture mode with Turkom-Cera group.

\* Details on statstcal analysis see Appendix IV

## **5.3.2** Effect of luting materials on the fracture resistance

The objective is to test if the mean load at fracture of Turkom-Cera copings differs among the three treatment groups: zinc phosphate cement (Elite), glass ionomer cement (Fuji I) and resin luting cement (Panavia F). Descriptive analysis was performed and the mean and median load at fracture for all groups are recorded in Table 5.8.

		Mean	Median	95% Confidence Interval		
Cement	n	( <b>SD</b> )	(IQR)	Lower Bound	<b>Upper Bound</b>	
Flito	10	1537.39	1495.6	1405.05	1660 72	
Ente	10	(185.01)	(333.25)	1403.03	1009.75	
Engi I	10	1294.40	1273.5	1204.02	1294 77	
Fuji I	10	(126.33)	(242.3)	1204.05	1304.77	
Donovio F	10	2183.60	2185.5	2066 22	2300.08	
Panavia F	10	(164.09)	(207.0)	2000.22	2500.98	

Table 5.8: The mean and median load at fracture (N) of the three luting cements used

Since the assumption of normal distribution was not met as indicated by histogram and Shapiro-Wilk test (Appendix IV), comparison using nonparametric Kruskal-Wallis Test was done to achieve objective (Table 5.9). According to the Kruskal-Wallis Test there was a significant difference between the three groups (p<.001).

Cement	n	Mean (SD)	Median (IQR)	Chi-Square ( <i>df</i> )	<i>P</i> value <sup>a</sup>		
Elite	10	1537.39 (185.01)	1495.6 (333.25)	22 228	<0.001		
Fuji I	10	1294.40 (126.33)	1273.5 (242.3)	(2)	<0.001		
Panavia F	10	2183.60 (164.09)	2185.5 (207.0)				
<sup>a</sup> Kruskal Wallis Test was used.							
Significant level was set at 0.05.							

**Table 5.9:** Comparison of load at fracture (N) of Turkom-Cera copings between Elite,Fuji I and Panavia F cements by Kruskal-Wallis Test

A Post hoc test using Mann-Whitney tests with Bonferroni correction as multiple pairwise comparisons (Appendix II) revealed that there were significant differences between load at fracture of Elite and Fuji I (p=0.030), Elite and Panavia F (p<0.001) and also between Fuji I and Panavia F (p<0.001). Results are summarized in Table 5.10.

Pairewise comparison	Mean (SD)	Median (IQR)	P value <sup>a</sup>
Elite	1537.39 (185.01)	1495.6 (333.25)	0.020*
vs Fuji I	1294.40 (126.33)	1273.5 (242.3)	0.030*
Elite	1537.39 (185.01)	1495.6 (333.25)	< 0.001*
vs Panavia F	2183.60 (164.09)	2185.5 (207.0)	< 0.001*
Fuji I	1294.40 (126.33)	1273.5 (242.3)	<0.001*
vs Panavia F	2183.60 (164.09)	2185.5 (207.0)	<0.001*
<sup>a</sup> Mann-Whitney Test was used.			
* Bonferroni correction was used.			

**Table 5.10:**Multiple pairwise comparisons of load at fracture (N) using Mann-<br/>Whitney Test with Bonferroni correction

### 5.3.2.1 Testing mode of fracture

A cross-tabulation between treatment groups and modes of fracture was obtained (Table 5.11). The Chi-square test was used to test if there is any association between treatment groups and modes of fracture (Appendix IV). Due to unmet assumption of Chi-Square test and non-meaningful combination of different modes the result can be only descriptively analyzed.

Examination of the mode of fracture of specimens revealed that 90 % of the Turkom-Cera copings cemented with zinc phosphate cement and 80 % cemented with resin luting cements exhibited minimal fracture (Figure 5.18). Whereas, only 60 % of Turkom-Cera copings cemented with glass ionomer cement exhibited minimal fracture.

	Mode of Fracture					
Cement	Minimal Fracture n (%)	Less than half of coping lost n (%)	Severe fracture of die and/or coping n (%)	Total		
Elite	9 (90%)	0 (0%)	1 (10%)	10		
Fuji I	6 (60%)	3 (30%)	1 (10%)	10		
Panavia F	8 (80%)	1 (10%)	1 (10%)	10		

**Table 5.11:** Distribution of modes of fracture in each treatment group (Elite, Fuji I and Panavia F)

Descriptive summary for modes of fracture and mean load at fracture was recorded (Table 5.12). The identified modes of fracture were: minimal fracture (crack line), less than half of coping lost and severe fracture of die and/or coping.

	Elite		Fuji I		Panavia F	
Mode of Fracture	n	Mean LAF (SD)	n	Mean LAF (SD)	n	Mean LAF (SD)
Minimal fracture	9	1552.80 (189.30)	6	1297.83 (138.00)	8	2191.63 (182.85)
Less than half of coping lost	0	0	3	1327.00 (124.76)	1	2205.00
Severe fracture of die and/or coping	1	1398.70	1	1176.00	1	2098.00

**Table 5.12:** Descriptive summary for modes of fracture and mean load at fracture (N)

(LAF: load at fracture)

As shown in Table 5.12, the minimal fracture mode occurred at a higher load with Panavia F (2191.63 N) followed by Elite group (1552.80 N) and Fuji I group (1297.83 N). Generally, the minimal fracture mode occurred at a higher load than sever fracture mode in all groups.

# 5.4 Discussion

#### 5.4.1 Methodology

This study was aimed to evaluate the strength of three types of all-ceramic systems and the influence of the luting cements on the strength of all-ceramic material Turkom-Cera fused alumina. The results provide an indication of the effect of luting cements on the strength of experimental copings, and how materials may behave relative to each other.

The methods used in this study were similar to previous studies (Sobrinho et al., 1998a; Harrington et al., 2003; Webber et al., 2003; AL-Makramani et al., 2008). For experimental convenience, the occlusal surfaces of the preparations and ceramic restorations were flat to assist in the ease of fabrication and testing and did not replicate the cuspal inclines found clinically. However, preparation of a relatively flat occlusal surface to facilitate the scanning process for Procera crowns has been recommended (Procera clinical manual, 1998).

According to Andersson et al., (1998) a moderate chamfer tooth preparation with rounded, smooth contours and no sharp line angles were necessary to create optimal precision of fit of Procera AllCeram crowns. The general preparation design of the tooth should correspond to the computer scanner probe. Since the tip of the scanner probe is rounded, the design of the preparation shoulder should be chamfered or bevelled (Prestipino et al., 1998; Neiva et al., 1998). Therefore, chamfer finish line was used in this study.

The recommended values of axial wall taper of tooth preparations vary widely. Rosenstiel et al., (1995) recommends a convergence angle of 6 degrees. Whilst Shillingburg et al., (1997) recommended convergence angles of 10, 14, 19, and 22 degrees for anterior teeth, premolars, maxillary molars, and mandibular molars, respectively. However, a convergence angle of 10-12 degrees has been reported and accepted in the literature. In this study a convergence angle of 4-6 degrees was used due to the limitation of the apparatus used for tooth preparation. However, in Chapter 6 a convergence angle of  $12^{\circ}$  (6° axial taper) was used for teeth preparation since a paralleling apparatus and a special jig were modified and used to achieve the desired degree.

In order to avoid the influences of preparation design, loading direction and loading stylus radius, an identical abutment analogue and loading apparatus were used for all test specimens. Metal dies were designed to represent a tooth prepared for a full-ceramic crown, thereby ensuring a standard size and shape for construction. In addition to that, the load was directed vertically in the centre of the occlusal surface down the long axis of each cemented crown (Dickinson et al., 1989; Friedlander et al., 1990; Miller et al., 1992; Ku et al., 2002).

Sandblasting and application of silane were used for the treatment of the fitting surface of the copings prior to cementation. Sandblasting method has been reported as being effective in preparing high alumina-content core ceramics for cementation because it roughens the internal surface of the high alumina core and increases the area available for bonding. It was also expected that the application of silane would have a minimal effect on the bond between the resin and ceramic due to the high alumina content of the cores. Therefore, its action was most likely to be as a wetting agent (Kern and Thompson, 1995; Awliya et al., 1998; Neiva et al., 1998; Madani et al., 2000; Webber et al., 2003).

An even thickness of the internal core is particularly important because small variations in the thickness can have a considerable effect on the overall fracture resistance of the restoration (Riley, 1977). The Turkom-Cera copings were prepared by one technician only, who has attended many training courses arranged by the Turkom-Cera Company. In-Ceram and Procera copings were prepared by a different technician. The two technicians were supervised and instructed to keep the coping thickness standard (0.6 mm). After finishing, all copings were visually inspected under 2.5x magnifications, measured for circumferential thickness on the buccal, occlusal, lingual, mesial and distal surfaces using a vernier caliper, and then matched to their specific die.

All copings were luted to their corresponding dies under standardized conditions. A constant load of 5 kg was applied vertically and considered adequate to ensure good seating without being sufficient to result in any form of damage to the cemented coping (Harrington et al., 2003; Webber et al., 2003).

In this study, a 0.5 cm Optosil silicone block was positioned between the 5 kg mass and copings during load application to avoid damage to the crowns by stress peaks. The vertical direction of the applied load was provided by a custom-made vertical loading apparatus (Makramani Load) specially designed and prepared for this study. The standardized cementation measures were used to yield equivalent cement film thickness. Both seating discrepancies and cement film thickness were supposed to be variables of influence (Tuntiprawon and Wilson, 1995; Groten and Probster, 1997).

### 5.4.2 Effect of ceramic materials

This study was aimed to evaluate the fracture resistance of Turkom-Cera fused alumina copings compared to In-Ceram and Procera AllCeram copings using metal supporting structure. The metal dies used in this study, although not replicating the elastic modulus of teeth, were homogenous in composition and provided even, void-free support for the ceramic restorations.

Sobrinho et al., (1998a,b); Cha et al., (2001); Webber et al., (2003) & many other studies used metal dies for testing the fatigue properties of all-ceramic crown systems because the metal dies did provide a reproducible support. Furthermore, the metal dies do eliminate the variability seen with natural tissues. In the present study, none of the six metal dies used were found to be broken or damaged. Some investigators used resin dies instead of metal dies as a supporting structure. Chai & co-workers (2000) compared the probability of fracture of four systems of all-ceramic crowns using composite resin master dies. From the results of fracture mode of the tested crowns, it was clear that up to 50% of the tested samples from each group suffered from fracture of the supporting die. Therefore, metal supporting dies were used in this study to ensure that the supporting die will not break before the coping.

The results of the present study indicated that Turkom-Cera copings luted with the resin luting cement (Panavia F) provided load at fracture (2183.6 N) that was higher to that obtained by Procera AllCeram (1953.5 N) and In-Ceram (2041.7 N) copings luted with the same cement. Statistical analysis using Scheffe's Post Hoc test showed significant difference between the mean load at fracture of Turkom-Cera and Procera AllCeram (p<0.05). The same test showed no significant difference between the mean load at fracture of Turkom-Cera and In-Ceram and also between the mean load at fracture of In-Ceram and Procera AllCeram (p>0.05). Burke (1992), found that biting forces of up to 800 N have been measured clinically in natural teeth and that experimental forces of this value may be considered to be of clinical relevance. In this study, crowns were supported by and bonded onto the metal dies, and the degree to which the metal-resin bond influenced the fracture resistance of the restorations is not known. The results of this study cannot be directly compared with either mean chewing forces or maximal biting forces because the copings were cemented to metal dies. Scherrer & de Rijk (1993), found that a die with a high modulus of elasticity can result in increased fracture loads of ceramic. However, as the substrate was consistent for all groups, this was not considered a confounding factor.

This study was in agreement with the findings of previous studies (Webber et al., 2003; Neiva et al., 1998; Harrington et al., 2003), which found no difference in fracture resistance of Procera AllCeram and In-Ceram crowns that were resin cemented. This may be attributed to resin cementation of die and ceramic, which act as a bonded system with load transfer through each interface.

Examination of the mode of fracture of specimens revealed that the majority of Turkom-Cera (80 %) and Procera AllCeram (80 %) copings exhibited minimal fracture (Fig. 5.18). Whereas, only 60 % of In-Ceram copings exhibited minimal fracture.



Figure 5.18: Most common mode of fracture for ceramic copings (minimal fracture).

This study evaluated the fracture resistance of all-ceramic materials supported by metal dies. The advantages of using such abutments are: maintaining the possibility of standardized preparation, ensuring that the die does not break or get damaged during testing and the identical physical quality of materials. Furthermore, natural teeth show a large variation depending on their age, individual structure and storage time after extraction, thus causing difficulties in achieving standard support (Strub and Beschnidt, 1998; Potiket et al., 2004). However, abutments made of metal do not reproduce the actual force distribution that may occur on crowns cemented to natural teeth. Chemomechanical interaction between the dentine and the luting agent also cannot be tested with this type of simulation (Webber et al., 2003; Sobrinho et al., 1998a; Cha et al., 2001). Therefore, another study has been conducted to evaluate the fracture resistance of Turkom-Cera copings supported to natural tooth structure (Chapter 6).

#### **5.4.3** Effect of luting materials

This in vitro study was conducted to evaluate the compressive strength of Turkom-Cera copings using different luting agents. Therefore, an analogue with the size and shape of a human tooth was used rather than a regular geometric configuration (Ku et al., 2002). Metal dies were designed to represent a tooth prepared for a full-ceramic crown, thereby ensuring a standard size and shape for construction.

The present study attempted to isolate the cement layer as the only variable. Attempts were made to standardize the other variables that may have an effect on the results of the fracture strength, as discussed earlier in this chapter.

The results of the present study indicated that Turkom-Cera copings luted with the resin luting cement (Panavia F) provided load at fracture (2183.6 N) that was significantly superior to that obtained when conventional luting agents were used, as important statistical difference was identified. The glass ionomer cement (Fuji I) group gave the lowest load at fracture (1294.40 N) of the three examined groups.

Different luting cements have been used with all-ceramic crown restorations. An apparent fracture strength increase of all-ceramic restorations that are luted using resin luting cement has been reported. The results of this study are in agreement with the results of previous studies (Jensen et al., 1989; Yoshinari and Derand, 1994; Burke, 1995; Groten and Probster, 1997).

An effect similar to results of this study was reported by another research finding. AL-Makramani et al., (2008a) examined the effect of different luting cements on the fracture resistance of Procera AllCeram copings. The copings cemented with Panavia F (1954 N) exhibited significantly higher load at fracture values than those cemented with zinc phosphate cement (1092 N) or glass ionomer cement (785 N).

In contrast, Casson et al., (2001) found that zinc phosphate cement group produced higher mean load at fracture (1216 N) than glass ionomer (754 N) or resin cement (989 N) groups. Another study by McCormick et al., (1993) reported that all-ceramic crowns luted with zinc phosphate, glass-ionomer and composite resin luting cements did not show any statistically significant difference among fracture strengths. However, in that study, a dentinal bonding agent was not used in conjunction with the resin composite luting material, a factor which may explain the apparent divergence between the results obtained by McCormick & those obtained by other studies.

In the present study, the zinc phosphate cement (Elite) group produced a surprisingly high mean load at fracture (1537.4 N) than glass ionomer cement (Fuji I) group (1294.4 N). Several authors have implicated the effect of modulus of elasticity of materials on fracture resistance (Scherrer and de Rijk, 1993; Lee and Wilson, 2000; Casson et al., 2001). Given that high modulus materials are necessary for high stress areas, Casson et al., (2001) has suggested that the elastic modulus of cements could lessen the effect of internal flaws in the ceramic. The modulus of elasticity of zinc phosphate cement is generally regarded as being higher than that of glass ionomer cement (Li and White, 1999).

An in-vitro study investigated the effect of luting media on the fracture resistance of allceramic crown (Casson et al., 2001). The glass ionomer group gave the lowest fracture resistance of any of the luted groups. This goes in agreement with the present study in which glass ionomer cement produced the lowest load at fracture with Turkom-Cera copings.

Examination of the mode of fracture of specimens revealed that the majority of Turkom-Cera copings cemented with either zinc phosphate cement (90 %) or resin luting cement (80 %) exhibited minimal fracture of copings (Figure 5.18). Whereas only 60 % of Turkom-Cera copings cemented with glass ionomer cement exhibited minimal fracture.

This study evaluated the fracture strength of Turkom-Cera all-ceramic material supported to metal dies. Another study has been conducted to evaluate the fracture resistance of Turkom-Cera copings supported to natural tooth structure (Chapter 6).

# 5.5 Conclusions

The results achieved from this study provided a description of load-bearing capacity of Turkom-Cera, In-Ceram and Procera AllCeram copings and the effect of different luting cements on the load at fracture of Turkom-Cera copings. Within the limitations of this study, it can be concluded that:

- Turkom-Cera copings luted with the resin luting cement (Panavia F) provided load at fracture (2183.6 N) that was significantly higher to that obtained by Procera AllCeram (1953.5 N) copings luted with the same cement. Thus, the null hypothesis was rejected.
- There was no significant difference between the mean load at fracture of Turkom-Cera and In-Ceram (2041.7 N) and also between the mean load at fracture of In-Ceram and Procera AllCeram.
- 3) The luting agents used in this study significantly affected the recorded load at fracture of Turkom-Cera copings; Turkom-Cera copings cemented with resin luting cement Panavia F (2183.6 N) were significantly stronger than Turkom-Cera copings cemented with either zinc phosphate Elite (1537.4 N) or glass ionomer Fuji I (1294.4 N) cements. Thus, the null hypothesis was rejected.
- Turkom-Cera copings cemented with zinc phosphate cement (Elite) were significantly stronger than Turkom-Cera copings cemented with glass ionomer cement (Fuji I).

### CHAPTER SIX

# EVALUATION OF THE OCCLUSAL FRACTURE RESISTANCE OF TURKOM-CERA COMPARED TO TWO OTHER ALUMINA-BASED CERAMIC SYSTEMS (PART II): AN IN VITRO STUDY

# 6.1 Introduction

Early types of metal-free ceramics did not enjoy success in dentistry, especially in the posterior region (Shimada et al., 2002). The high crystalline content ceramic systems have been developed in an attempt to improve the strength of metal-free restorations as well as deliver more esthetic results than conventional metal-fused-to-ceramic restorations (Ozcan et al., 2001; Valandro et al., 2006). Glass infiltrated alumina ceramic (eg. In-Ceram Alumina, Vita Zahnfabrik, Bad Sackingen, Germany), densely sintered aluminum oxide ceramic (eg, Procera AllCeram, Nobel Biocare AB, Gothenburg, Sweden) and zirconium oxide ceramic (eg, Lava 3M ESPE, St. Paul, MN, USA) are popular oxide-based high-strength ceramic materials that offer favorable esthetic characteristics, mechanical properties and biocompatibility (Blatz et al., 2004; Della Bona et al., 2007).

It is obvious from the different studies in relation to the occlusal fracture resistance of all-ceramic systems that the values reported are highly variable. This is because the testing of the occlusal fracture resistance of crowns is not a standard procedure like a bending test for a geometrically well-defined bar. As reported by some researchers (Webber et al., 2003; AL-Makramani et al., 2008a; Di Iorio et al., 2008), the results of the fracture load of all-ceramic crowns may be influenced by different factors. These include the microstructure of the ceramic material, preparation design, shape and thickness of the restoration, size and distribution of surface flaws, the magnitude, direction and location of the applied load, luting methods, the elastic modulus of the restoration components and storage conditions before loading to fracture.

Regarding the effect of finish line on the occlusal fracture resistance of all-ceramic crowns, it was shown that preparation with a 1.2 mm shoulder finish line and sharp axiogingival line angle produced the strongest Dicor crowns, while crowns prepared with a chamfer finish line produced the weakest restoration when cemented to metal dies (Doyle et al., 1990a; Doyle et al., 1990b). A similar in-vitro study on Procera AllCeram crowns found that the shoulder finish line showed significantly higher fracture resistance than chamfer finish line (Di Iorio et al., 2008). In contrast, some authors reported that the fracture resistance of Dicor crowns luted with a resin luting cement was unaffected by the type of finish line used (Bernal et al., 1993; Malament and Socransky, 1999a).

The present study investigated the effect of three variables on the occlusal fracture resistance of all-ceramic crowns. Attempts were made to standardize the other variables that may have an influence on the results of the fracture load. The studies concerning the effect of ceramic material, margin design and artificial ageing on the occlusal fracture resistance of Turkom-Cera restorations are limited. Therefore, the objectives of this study were to:-

- Determine the occlusal fracture resistance of Turkom-Cera copings compared to In-Ceram and Procera All-Ceram copings cemented to extracted teeth.
- Examine the effect of finish line design on the occlusal fracture resistance of Turkom-Cera copings.
- 3. Study the effect of artificial ageing (water storage and thermocycling) on the occlusal fracture resistance of Turkom-Cera copings.

#### Null hypotheses

- 1. There is no difference in the occlusal fracture resistance of Turkom-Cera, In-Ceram and Procera AllCeram copings when cemented to natural teeth.
- There is no effect of finish line design on the occlusal fracture resistance of Turkom-Cera copings.
- 3. There is no effect of artificial ageing (water storage and thermocycling) on the occlusal fracture resistance of Turkom-Cera copings.

# 6.2 Materials and methods

### 6.2.1 Materials used

Fifty sound and crack-free maxillary premolar teeth, extracted for orthodontic reasons (the patient's ages ranged from 15-20 years), were used for this study. In addition, three types of all-ceramic systems were used for coping production namely, Turkom-Cera<sup>™</sup> (Turkom-Ceramic (M), Puchong, Malaysia), In-Ceram (Vita Zahnfabrik, Bad Sackingen, Germany) and Procera AllCeram (Nobel Biocare, Goteborg, Sweden), and one type of resin luting cement (Panavia-F, Kuraray Medical Inc., Okayama, Japan) with its silane coupling agent (Clearfil Silane Kit, Kuraray), were used in this study.

# 6.2.2 Methods

#### 6.2.2.1 Specimen collection and storage

Based on criteria, the selected teeth were free of cracks and fractures, had no evidence of caries or restorations and had no previous endodontic treatment. The average buccolingul, mesio-distal crowns width and teeth length were 9.1 mm, 7.3 mm and 22.3 mm, respectively (Appendix V). The teeth were obtained directly after extraction and stored in 0.5% Chloramine-T trihydrate solution for one week (ISO/TS 11405/2003). Both calculus deposits and residual periodontal tissues were removed by Ultrasonic Scaler (Peizon® Master 400, EMS, Nyon, Switzerland). All teeth were examined under stereo microscope (Olympus SZ61, Olympus Corp., Tokyo, Japan) at 30x to detect cracks before including them in the study. Throughout this study, the teeth were kept hydrated in distilled water as this storage solution does not seem to alter dentine permeability (Goodis et al., 1993). The storage solution was changed every one week and the teeth were stored at 4 degrees Celsius (ISO/TS 11405/2003).

### 6.2.2.2 Preparation of teeth

The teeth were embedded in epoxy resin 2.0 mm below the cemento-enamel junction using a plastic mould with 30 mm diameter and 30 mm height (Figure 6.1). Two layers of nail polish were applied to the external surface of the entire roots. A dental surveyor was used to position the long axis of the teeth vertically.



Figure 6.1: The tooth embedded in the epoxy resin.

The preparation of the teeth was carried out using high-speed handpiece attached to a paralleling apparatus (Figure 6.2), which allowed standardized preparations. The apparatus consists of a specimen fixture as well as vertical and horizontal arms. The specimen fixture holds the specimen and designed in a way that the fixture can rotate the specimen against a diamond bur (Figure 6.3a & b). The vertical arm of the apparatus which holds the handpiece permits vertical as well as rotational movement around the tooth. The high-speed handpiece was attached to the vertical arm of the paralleling apparatus using a custom made jig (Figure 6.4). This jig secures the handpiece to the vertical arm in such a manner that the attached bur can be fixed at a set angle to that of the tooth during preparation.



Figure 6.2: The paralleling apparatus used.



Figure 6.3a & b: Two views of the specimen fixture.



Figure 6.4: The jig used to fix the handpiece to the vertical arm of the apparatus.

Figure 6.5 shows a schematic illustration of the paralleling apparatus and tooth preparation assembly.



Figure 6.5: Schematic illustration of the paralleling apparatus.

The axial taper angle used in the present study was  $6^{\circ}$ . According to Shillingburg et al., (1997) a tapered bur will impart an inclination of 2-3 degrees to any surface it cuts if the shank of the instrument is held parallel to the intended path of insertion of the preparation. A specially designed jig which consists of a semi-circular transparent Perspex block with a protractor fixed to its side was used to set the degree of taper (Figure **6.6**). A hole was drilled along the outer side of the perspex block corresponding to a taper angle of  $3^{\circ}$ .



Figure 6.6: The jig used to set the degree of taper.

Therefore, to achieve a  $6^{\circ}$  axial taper preparation, the handpiece was secured to the apparatus so that the attached tapered diamond bur was oriented at a  $3^{\circ}$  angle to the vertical axis of the tooth (Figure 6.7). This, in additional to the  $3^{\circ}$  taper of the tapered bur, resulted in a total axial taper angle of  $6^{\circ}$  corresponding to a convergence angle of  $12^{\circ}$ .



Figure 6.7: The diamond bur oriented at 3° to the vertical axis.

The axial reduction was performed by rotating the specimen in the fixture against a coarse rotating tapered diamond burs (5856.314.018, chamfer; and 8847KR.314.018, shoulder, Komet GmbH, Lemgo, Germany) as shown in Figure 6.8. After that, the preparation surfaces were finished with a fine grit diamond burs (No. 5856.314.018, chamfer; and 8847KR.314.018, shoulder).



Figure 6.8: The tooth during axial preparation.

After the completion of axial preparation, the occlusal surface of the teeth was cut flat. A pencil was used to mark the prepared tooth 4 mm above the margin and the occlusal surface was flattened with a diamond wheel (Komet No. 909.204.055) to the marked line (Figure 6.9a), which resulted in a preparation with 4.0 mm height (Figure 6.9b).

The preparation was smoothed and all sharp angles or internal line angles were rounded with a fine abrasive disk (Sof-Lex Discs, 3 M Corp., St. Paul, Minn.) connected to a micromotor handpiece. All preparations were made under copious water irrigation by the same investigator.



Figure 6.9a & b: The tooth before (a) and after (b) oclussal reduction.

These series of reductions resulted in a standardized teeth preparation with 6° axial taper, a 1.2 mm circumferential chamfer/shoulder margin, placed 0.5 mm occlusal to the cemento-enamel junction, and total preparation height of 4.0 mm. The finished preparation is illustrated in Figure 6.10a &b.



Figure 6.10a & b: Occlusal (a) and buccal views (b) of the finished preparation.

In summary, 30 premolar teeth were prepared with chamfer margin and divided randomly into 3 groups (n=10) for each of the three ceramic systems used (In-Ceram, Procera and Turkom-Cera). In addition, 20 premolars were prepared with chamfer margin (n=10) and round shoulder margin (n=10) and used to study the effect of artificial ageing (water storage and thermocycling) and finish line design on the fracture resistance of Turkom-Cera all-ceramic copings.

### 6.2.2.3 Impression and die preparation

The teeth were dried with an air/water syringe and impression was taken for each tooth using a bottle cap with pipette which act as an impression tray and silicone impression material (Aquasil Monophase Ultra; Dentsply Caulk, Dentsply International Inc., Milford, Germany) (Figure 6.11a & b). The impression material was injected into the cap and placed on the tooth while maintaining finger pressure until setting.



**Figure 6.11a & b:** The impression material, tooth model and plastic cap (a); and impression of the prepared tooth (b).

Then, the impressions were boxed (Figure 6.12a) using boxing wax (Boxing In Wax, Metrodent Ltd., Huddersfield, Enland). The impressions were then vaporized with a wetting agent and poured in die stone (Densite, Shufo, Kyoto, Japan) (Figure 6.12b). The stone was mixed with distilled water in a 20cc liquid to 100 grams of stone ratio as recommended by the manufacturer. After a 4 hours setting time, the dies were trimmed and numbered according to their respective teeth (Figure 6.13).



Figure 6.12a & b: Boxing of the impression (a) and pouring with die stone (b).



Figure 6.13: The die numbered according to its respective tooth.

#### 6.2.2.4 Fabrication of all-ceramic copings

The fabrication technique for Turkom-Cera, In-Ceram and Procera AllCeram copings has been discussed in Chapter Five (5.2.2.3). 20 randomly selected stone dies with chamfer margin and the 10 stone dies with shoulder margin were sent to Turkom-Cera dental laboratory and 30 Turkom-Cera copings were fabricated with a thickness of 0.6 mm. The remaining 20 stone dies with chamfer margin were randomly divided into two groups of 10 specimens for In-Ceram (n=10) and Procera (n=10) and sent to other dental laboratory. 10 In-Ceram and 10 Procera copings were fabricated with a thickness of 0.6 mm.

The macroscopic fit of all copings on the corresponding stone dies, and finally on the specimens were visually assessed. Copings that were found to rock or did not seat on the finish line were rejected and refabricated to ensure 10 copings per group.

#### 6.2.2.5 Cementation

The specimens were identified by their numbers. The teeth were pumiced with a prophylaxis cup mounted on a slow speed handpiece and rinsed with an air/water syringe prior to cementation. The copings were then internally sandblasted with 50  $\mu$ m aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) particles at an air pressure of 2.5 bars for 13s from a distance of 10 mm. After that, the copings were steam cleaned and air dried.

All copings were cemented to their respective teeth using Panavia F resin luting cement. The ED primer was applied to the entire surface of the tooth and allowed to set for 60s before air drying with gentle air flow. The fit surfaces of all copings were silanated with a mixture of Clearfil Porcelain Bond Activator and Clearfil SE Bond Primer. The mixture was applied to the internal surface of the coping and left for 5s before air drying with gentle air flow. Sufficient amount of the Panavia F (one complete turn from each cartridge A &B) were dispensed, mixed for 20s and applied to the internal surface of each coping.

Finger pressure was applied to initially seat each coping on its respective tooth, and each coping was held in place while any excess paste remaining at the margins was removed with a disposable brush. A layer of Oxyguard II (Kuraray) was applied for three minutes around the margins of each specimen. The specimens were then placed in a custom-made vertical loading apparatus (Makramani Load) (AL-Makramani et al., 2008a), for 10 minutes under a 5 kg load. Following cementation, the 30 specimens with chamfer margin and the 10 specimens with shoulder margin were placed in a sealed container of distilled water and left in an incubator at a constant temperature of 37°C for 24 hours.

#### 6.2.2.6 Water storage and thermocycling

Ten Turkom-Cera specimens with chamfer margin were stored in distilled water at 37°C for 30 days and subjected to thermal cycling according to the ISO recommendations (ISO/TS 11405/2003).

The specimens were submitted to 500 thermo-cycles in distilled water between 5°C and 55°C (Figure 6.14). The exposure to each bath was 30s and the transfer time was 10s.



Figure 6.14: Specimens during thermocycling.

### 6.2.2.7 Testing Procedure

The tooth with cemented coping was removed from the storage container, secured in a mounting jig and subjected to testing in a universal testing machine (Shimadzu, Shimadzu Corp., Tokyo, Japan) (Figure 6.15). A 3 mm stainless steel bar, mounted on the crosshead of the Shimadzu testing machine was used and applied a compressive load at the centre of the occlusal surface, along the long axis of the cemented copings, at a crosshead speed of 1mm/min until failure occurred (Figure 6.16). A piece of tin foil 0.7 mm thick was placed between the loading piston and the specimen to distribute the force over a larger area and to avoid loading stress peaks on the coping surface. The maximum force to produce fracture was recorded in Newtons. The failed copings were examined in order to determine the mode of fracture. The mode of fracture was classified using categories as described by Burke & Watts (1994) (Table 5.2 and Figure



Figure 6.15: The universal testing machine used.



Figure 6.16: The specimen during testing.

### 6.2.2.8 Statistical analysis

The first objective was to determine the occlusal fracture resistance of Turkom-Cera copings compared to In-Ceram and Procera AllCeram copings. Descriptive statistics will be recorded for load at fracture of the three groups. The normally distributed data of load at fracture (Histogram) will be analyzed by One Way ANOVA to achieve the objective, provided that equal variances will be assumed (Levene's test). Then, Tukey's HSD test will be carried out for post-hoc comparisons.

Whenever assumptions of normal distribution and equal variances of load at fracture between the three groups will not be met, an equivalent nonparametric Kruskal-Wallis test will be conducted. Subsequently, a post hoc test using Mann-Whitney tests with Bonferroni correction will be performed to test which pair of groups differ from each other significantly.

Regarding the second objective, effect of finish line design and artificial ageing on load at fracture of Turkom-Cera copings, descriptive statistics will be recorded for load at fracture of each group. Then, independent samples t-test should be used to determine the significant differences in load at fracture between the two groups of Turkom-Cera copings with chamfer and shoulder finish lines and also between the two groups of Turkom-Cera copings with and without artificial ageing. However, this is dependent on the assumptions of normal distribution (histogram and Shapiro-Wilk test) and equal variances (Levene's test) of load at fracture between each two groups to be met.

Whenever assumptions of normal distribution and equal variances of load at fracture between each two groups will not be met, an equivalent nonparametric Mann-Whitney tests will be performed to test which pair of groups differ from each other significantly. In addition, descriptive statistics for modes of fracture and load at fracture will be recorded and the result will be descriptively analyzed. Statistical analysis will be carried out using a computer program (SPSS Inc., Chicago, IL). Statistical significance will be set at  $\alpha$ = 0.05.

### 6.3 Results

### 6.3.1 Effect of ceramic material on the fracture resistance

The objective is to test if the mean load at fracture of Procera AllCeram, Turkom-Cera, and In-Ceram all-ceramic copings differ from each other. Descriptive analysis was performed and the mean load at fracture and standard deviation for the three groups are recorded in Table 6.1.

				95% Confidence Interval		
				Lower Upper		
Ceramic	n	Mean	SD	Bound Bound		
Procera AllCeram	10	975.0	112.7	894.3	1055.6	
Turkom-Cera	10	1341.9	216.5	1187.0	1496.8	
In-Ceram	10	1151.6	180.1	1022.7	1280.4	

 Table 6.1:
 The mean load at fracture (N) and standard deviation for Procera

 AllCeram, Turkom-Cera, and In-Ceram copings

Since the sample size is 30 and fulfills the central limit theorem assumption, only histogram (Appendix V) was used and showed normal distribution of the load at fracture of the three groups. Since the assumption of normal distribution was met, the equality of variances (homogeneity) was tested using the Levene's test (Appendix V) and showed that there was no significant deviation from homogeneity (p=0.163). Therefore, the parametric One Way ANOVA procedure was used to achieve objective. The results were recorded in Table 6.2. There was a significant difference in load at fracture between the three groups (p<0.001).

Ceramic	n	Mean (N)	SD	F Statistics (df)	<i>P</i> value <sup>a</sup>		
Procera AllCeram	10	975.0	112.7				
Turkom-Cera	10	1341.9	216.5	10.98	< 0.001		
In-Ceram	10	1151.6	180.1	(2,27)			
<sup>a</sup> One Way ANOVA was used.							
Significant level was set at 0.05.							

 Table 6.2:
 Comparison of load at fracture between Procera AllCeram, Turkom-Cera and In-Ceram copings by One Way ANOVA

Further analysis using Tukey HSD Post Hoc Test was done to determine the pair of means that differ significantly (Appendix V). Based on Tukey HSD Post Hoc Test (Table 6.3), the mean load at fracture of Turkom-Cera (1341.9  $\pm$  216.5 N) was significantly more than Procera AllCeram (975.0  $\pm$  112.7 N) (p<0.001). There were no significant differences between the mean load at fracture of In-Ceram (1151.6  $\pm$  180.1 N) and Procera AllCeram (p=0.080) and also between the mean load at fracture of Turkom-Cera and In-Ceram (p=0.056).

Pairewise comparison	Mean (SD)	Mean Difference	P value	
Procera AllCeram	975.0 (112.7)	-366.93	0.0011	
vs Turkom-Cera	1341.9 (216.5)	366.93	<0.001*	
Procera AllCeram	975.0 (112.7)	-176.60	0.000	
vs In-Ceram	1151.6 (180.1)	176.60	0.080	
Turkom-Cera	1341.9 (216.5)	190.33		
vs In-Ceram	1151.6 (180.1)	-190.33	0.056	
* 2 pairs of means are significated	ntly different by Tukey HS	SD Test	•	

Table 6.3: Multiple pairwise comparisons of fracture load (N) using Tukey HSD Test

### 6.3.1.1 Mode of fracture

A cross-tabulation between treatment groups and modes of fracture was obtained (Table 6.4). The Chi-square test was used to test if there is any association between treatment groups (Procera, Turkom-Cera and In-Ceram) and modes of fracture (Appendix V). Due to unmet assumption of Chi-Square test and non-meaningful combination of different modes the result can be only descriptively analyzed.

Examination of the mode of fracture of specimens revealed that (70 %) of Turkom-Cera and (80 %) Procera AllCeram copings exhibited minimal fracture (Fig. 6.26). Whereas, only 40 % of In-Ceram copings exhibited minimal fracture.

	Mode of Fracture					
		Less than	More than	Severe		
Ceramic	Minimal	half of	half of	fracture of die		
	Fracture   coping lost   coping lost   and/or cop		and/or coping	Total		
	n (%)	n (%)	n (%)	n (%)		
Procera All-ceram	8 (80%)	1 (10%)	1 (10%)	0 (0%)	10	
Turkom-ceram	7 (70%)	2 (20%)	1 (10%)	0 (0%)	10	
In-ceram	4 (40%)	1 (10%)	2 (20%)	3 (30%)	10	

**Table 6.4:**Distribution of modes of fracture in each treatment group (Procera,<br/>Turkom-Cera and In-Ceram)

Descriptive summary for modes of fracture and mean load at fracture was recorded (Table 6.5). The identified modes of fracture were: minimal fracture, less than half of coping lost, more than half of coping lost and severe fracture of die and/or coping.

	Procera		Turkom-Ceram		In-Ceram	
Mode of Fracture	n	Mean LAF (SD)	n	Mean LAF (SD)	n	Mean LAF (SD)
Minimal fracture	8	999.72 (102.67)	7	1344.09 (194.26)	4	1158.32 (245.54)
Less than half of coping lost	1	965.36	2	1233.47 (362.48)	1	1077.61
More than half of coping lost	1	786.51	1	1543.40	2	1159.21 (139.41)
Severe fracture of die and/or coping	0	0	0	0	3	1162.12 (206.71)

 Table 6.5:
 Descriptive summary for modes of fracture and mean load at fracture (N)

(LAF: load at fracture)

As shown in Table 6.5, the sever fracture mode was not seen within Procera and Turkom-Cera groups. The minimal fracture mode occurred at a higher load with Turkom-Cera group (1344.09 N) followed by In-Ceram (1158.32 N) and Procera AllCeram (999.72 N) groups.

Furthermore, the minimal fracture mode occurred at a higher load than other modes of fracture in the Procera group. Whereas, in the Turkom-Cera group the minimal fracture mode occurred at a higher load than the less than half of coping lost mode, and at a lower load than the more than half of coping lost mode. Regarding In-Ceram group, the minimal fracture mode occurred at almost similar load to the other modes of fracture.

### 6.3.2 Effect of finish line on the fracture resistance of Turkom-Cera

Specimens were divided into two groups according to finish line used; Group 1 (Turkom-Cera with chamfer finish line) and Group 2 (Turkom-Cera with shoulder finish line). The mean and median load at fracture of the two groups are shown in Table 6.6.
		Mean	Median	95% Confidence Interva		
		( <b>SD</b> )	(IQR)	Lower Upper		
Finish line	n	( <b>n=10</b> )	( <b>n=10</b> )	Bound	Bound	
Turker Care (Chemfor)	10	1341.9	1407.38	1107.0	1406.9	
Turkom-Cera (Chamler)		(216.5)	(343.99)	1107.0	1490.0	
Turkom Core (Shouldor)	10	1545.2	1549.35	1/11 0	1679 7	
Turkom-Cera (Shoulder)		(186.6)	(318.38)	1411.0	10/8./	

**Table 6.6**: The mean and median load at fracture (N) of Turkom-Cera (Chamfer) and<br/>Turkom-Cera (Shoulder) groups

For testing normality, the histogram and Shapiro-Wilk test (Appendix V) were used and showed no normal distribution of the mean load at fracture of the two groups. Since the assumption of normal distribution was not met, comparison of the load at fracture between the two groups was performed using the nonparametric Mann-Whitney U Test (Table 6.7). There was no significant difference in the load at fracture between the two groups (p=0.059). Therefore, there was no influence of the finish line on the load at fracture of Turkom-Cera all-ceramic copings.

**Table 6.7:**Comparison of load at fracture (N) between Turkom-Cera (Chamfer) and<br/>Turkom-Cera (Shoulder) groups by Mann-Whitney Test

Variable	Turkor (Char (n=	n-Cera mfer) :10)	Turkor (Shou (n=	P		
	Mean Median (SD) (IQR)		Mean (SD)	Median (IOR)	value	
Load at fracture (N)	1341.9 (216.5)	1407.38 (343.99)	1545.2 (186.6)	1549.35 (318.38)	0.059*	
* 2 pairs of medians are not significantly different by Mann-Whitney Test.						

# 6.3.3 Effect of water storage and thermocycling on the load at fracture of Turkom-Cera

Specimens were divided into two groups; Group 1 (Turkom-Cera without ageing) and Group 2 (Turkom-Cera with ageing). The mean and median load at fracture of the two groups are shown in Table 6.8.

				95% Confidence Interval	
Finish line	n	Mean (SD)	Median (IQR)	Lower Bound	Upper Bound
Turkom-Cera (no aging)	10	1341.9 (216.5)	1407.38 (343.99)	1187.0	1496.8
Turkom-Cera (with aging)	10	1174.3 (140.6)	1145.03 (234.02)	1073.8	1274.9

Table 6.8:The mean and median load at fracture (N) of Turkom-Cera (no aging) and<br/>Turkom-Cera (with aging) groups

For testing normality, the histogram and Shapiro-Wilk test (Appendix V) were used and showed no normal distribution of the mean load at fracture of the two groups. Since the assumption of normal distribution was not met, comparison of the load at fracture between the two groups was performed using the nonparametric Mann-Whitney U Test (Table 6.9). There was no significant difference in the load at fracture between the two groups (p=0.059). Therefore, there was no influence of the artificial ageing used in this study (water storage and thermocycling) on the load at fracture of Turkom-Cera all-ceramic copings.

Variable	T (no a	C ging)	T (with a	P			
	Mean	Median	Mean	Median	value		
	(SD) (IQR)		( <b>SD</b> )	(IQR)			
Load at fracture (N)	1341.9 N	1407.38	1174.3 N	1145.03	0.059*		
Load at fracture (N)	(216.5)	(343.99)	(140.6)	(234.02)			
* 2 pairs of medians are not significantly different by Mann-Whitney Test.							

**Table 6.9:**Comparison of load at fracture (N) between Turkom-Cera (no aging) and<br/>Turkom-Cera (with aging) groups using the Mann-Whitney Test

# 6.4 Discussion

# 6.4.1 Methodology

An ideal experimental model of an in-vivo situation to determine the occlusal fracture resistance of all-ceramic crowns is difficult to achieve. The occlusal fracture resistance of a clinical ceramic crown is influenced by several factors, such as method of luting, loading condition and the elastic modulus of the supporting die (Scherrer and de Rijk, 1993, Webber et al., 2003; AL-Makramani et al., 2008a). However, the so called "crunch-the-crown" mechanical test had been widely used to examine the occlusal fracture resistance of sound and restored teeth (Al-Wahadni et al., 2009).

Mechanical tests on ceramic materials are difficult to carry out because of the presence of several limitations related to specimen preparation (Di Iorio et al., 2008). The present study attempted to isolate the ceramic material, artificial ageing and the finish line as the only variables. Natural teeth with comparable size and length were selected for this study to eliminate a possible effect of variations. In addition, a paralleling apparatus was used to prepare the teeth which allowed standardized preparations for all teeth.

In vivo, the teeth are supported by a visco-elastic periodontal ligament, which was not duplicated in the mounting of the specimens in the current study. The ability of the artificial ligament to reproduce the complex visco-elastic properties exhibited by ligament in vivo is limited (King and Setchell, 1990; Gu and Kern, 2006). In the clinical situation, the periodontal ligament may help to dissipate some of the applied load but at high loads simulation of a periodontal ligament in vitro is not useful as previous work has indicated that the root compresses the simulated ligament and impacts against the rigid mounting system and this might not reflect the clinical reality (Fokkinga et al., 2006; Gu and Kern, 2006; Good et al., 2008).

In the current study, two layers of nail varnish were therefore used for coating the root surfaces prior to embedding them in epoxy resin, which helped to avoid external reinforcement of the root by resin (Gu and Kern, 2006).

In the present study, the ceramic copings were cemented to natural teeth to replicate fracture load results more related to clinical situations than using ceramic discs (Wakabayashi and Anusavice, 2000; Ku et al., 2002) or crowns cemented to resin or metal die replicas (Neiva et al., 1998; AL-Makramani et al., 2009). Die replicas made of steel or resin fail to reproduce the actual force distribution at the inner surface of the crown or to reliably produce the characteristics of bonding between crowns and prepared teeth (Kelly, 1999; AL-Makramani et al., 2008a). However, die replicas provide a standardized preparation and identical physical qualities of materials used in comparison with natural teeth (Potiket et al., 2004; AL-Makramani et al., 2008b). Natural teeth show a large variation depending on their age, individual structure, and storage time after extraction, thus, causing difficulties in standardization (Strub and Beschnidt, 1998; Potiket et al., 2004).

Since the effect of the veneering porcelain on the load at fracture of high-strength allceramic restorations is still debatable, the copings were not veneered with porcelain (Webber et al., 2003; Beuer et al., 2008b). In fact, fracturing of multilayer crowns starts at their weakest part. In the case when a stronger and stiffer core substructure is veneered with weaker porcelain, the failure usually occurs in the weak veneering porcelain or at the bond between the core and veneer (Aboushelib et al., 2006; Zahran et al., 2008). In addition, the veneering procedures could actually introduce factors (such as: flaws, cracks, voids, or internal stresses) that influence the results of mechanical tests (Vult von Stevern et al., 2006; Di Iorio et al., 2008). As stated by Miranda et al., (2001) flaws play a crucial role in the fracture resistance of brittle materials. Therefore, all-ceramic copings without porcelain veneering were loaded until fracture in this study.

The most commonly used artificial ageing technique is long-term water storage. Another widely used ageing technique is thermocycling (De Munck et al., 2005). The ISO/TS 11405 standard (2003) indicates that a thermocycling regimen comprised of 500 cycles in water between 5°C and 55°C is an appropriate artificial ageing test. In order to evaluate the effect of water storage and thermocycling on the fracture resistance of Turkom-Cera copings, 10 Turkom-Cera specimens were stored in distilled water for 30 days and subjected to 500 cycles in water between 5°C and 55°C before testing their fracture resistance.

The present study was conducted to evaluate the occlusal fracture resistance of copings fabricated using three ceramic systems and bonded to prepared teeth using resin luting cement. Such an in vitro study does not require natural teeth as a control group for comparison of results (Al-Wahadni et al., 2009; Komine et al., 2004; Potiket et al., 2004) since the stress distribution in restored teeth is different than in unrestored teeth (Arola et al., 2001). Furthermore, studies have found a large variability in the occlusal fracture resistance of extracted unprepared natural teeth (Attia et al., 2004; Attia et al., 2006).

# 6.4.2 Discussion of results

In the present study, the load at fracture of Turkom-Cera, In-Ceram and Procera AllCeram all-ceramic copings cemented to extracted teeth using resin luting cement was evaluated. The data showed that the mean load at fracture for Turkom-Cera, In-Ceram and Procera AllCeram were: 1341.9 N, 1151.6 N and 975.0 N, respectively. Statistical

analysis showed that the differences were significant between Turkom-Cera and Procera AllCeram (p<0.001). However, no significant differences were detected between Turkom-Cera and In-Ceram (p=0.056) and also between In-Ceram and Procera AllCeram (p=0.080).

The results of this study for Turkom-Cera and In-Ceram copings were in agreement with those obtained in a previous study (AL-Makramani et al., 2009), which found that Turkom-Cera had a significantly higher load at fracture than Procera AllCeram. Furthermore, the results of this study for In-Ceram and Procera AllCeram copings were in agreement with those obtained in previous studies (Webber et al., 2003; Neiva et al., 1998; Harrington et al., 2003; AL-Makramani et al., 2009), which found no significant differences in load at fracture between Procera AllCeram and In-Ceram copings that were resin cemented.

Comparison with the load values reported in Chapter 5, which evaluated the fracture resistance of Turkom-Cera, In-Ceram and Procera AllCeram copings cemented on a metal master die using resin luting cement, shows that the load necessary to fracture the Turkom-Cera, In-Ceram and Procera AllCeram copings in the current study was less than that reported in Chapter 5. In this study, extracted natural teeth were used as abutments. However, metal dies are very rigid and have a higher modulus of elasticity than dentine so that metal dies deform less which results in a lower shear stress at the inner crown surface (Scherrer & de Rijk, 1993). Therefore, the fracture load of all-ceramic restorations may be greater if crowns are supported by dies with a high modulus of elasticity (Scherrer & de Rijk, 1993). This factor should also be considered when interpreting the results of the studies utilizing different die materials.

The results of the present study show that there is no influence of the finish line design on the load at fracture of Turkom-Cera all-ceramic copings. Statistical analysis revealed no significant difference between shoulder (1545.2 N) and chamfer (1341.9 N) margins used in this study (p=0.059). In this study only Turkom-Cera copings were evaluated. Due to this limitation, the load at fracture values obtained in this study should be compared with caution with results obtained in the studies where copings were veneered with feldspathic porcelain.

Results of the present study concurred with other studies on glass-ceramic crowns (Dicor) which did not demonstrate any differences in the loading capacity in relation to the type of finish lines used (Bernal et al., 1993; Malament and Socransky, 1999a).

Di Iorio et al., (2008) found that the load at fracture for Procera (alumina-based) crowns with shoulder preparation was significantly higher than the chamfer preparation. In the current study, the load at fracture of Turkom-Cera copings with shoulder margin (1545.2 N) was higher than chamfer margin (1341.9 N), however, statistical analysis revealed no significant difference between them (p=0.059). A possible reason for this may be that the occlusal forces were also borne by the circumferential shoulder margin, and there was less stress concentration on the axial walls compared to chamfer margin (Beuer et al., 2008b).

Conversely, a study on ceramic optimized polymer (Ceromer) crowns demonstrated that the fracture resistance of the chamfer finish line specimens was greater than that of the shoulder finish line (Cho et al., 2004). In the oral environment, the influence of water and changing temperature can promote crack propagation and decrease the fracture resistance of all-ceramic restorations (Kelly, 1995). In-vitro investigations showed that storage in water for extended periods and/ or changing temperature will alter the failure load data (Mante et al., 1993; Kern et al., 1994;). In the current study, the mean value of load at fracture in the Turkom-Cera group subjected to water storage and thermocycling was (1174.3 N) compared to (1341.9 N) in the Turkom-Cera group without water storage and thermocycling. Statistical analysis in relation to the amount of load at fracture within the two groups of Turkom-Cera in this study did not show statistically significant difference (p=0.059). This is in agreement with another study which found that thermocycling did not significantly reduce the fracture resistance of IPS Empress 2 crowns (Fiket et al., (2005). However, this is contrast with the result of a recent study which found that thermocycling adversely affects the ability of the IPS e.max Press crowns to resist the applied load (Abou-Madina and Abdelaziz, 2009). This reduction in the fracture resistance of cemented IPS e.max crowns could be the result of deterioration of the luting cement underneath (Blatz et al., 2004; Kern and Wegner, 1998).

The classification description of mode of failure by Burke & Watts (1994) was useful in distinguishing between minimal loss of crown material and catastrophic damage (mode II–IV). Examination of the mode of failure of specimens in the current study revealed that Procera AllCeram, Turkom-Cera and In-Ceram copings exhibited (80%), (70%) and (20%) of minimal fracture, respectively. The copings made from Turkom-Cera with shoulder margin and Turkom-Cera with artificial ageing exhibited 50% and 60% of minimal fracture, respectively.

Clinically, dental restorations are subjected to cyclic forces ranging from 60 N to 250 N during normal function and up to 500 N to 800 N for short periods (Zahran et al., 2008; Waltimo and Könönen, 1993; Waltimo and Könönen, 1995). Waltimo & Könönen (1993), reported that the maximum biting force in the molar region was 847 N for men and 597 N for women. The maximum biting force in the premolar region has been reported to be between 181 and 608 N (Widmalm and Ericsson, 1982). However, the normal masticatory forces in human beings have been reported to range from 37% to 40% of the maximum biting force (Lundgren and Laurell, 1986; Gibbs et al., 1981).

Although the results of the present study cannot be directly compared with the in vivo situation, the mean loads at fracture for all groups (ranging from 975.0 to 1545.2 N) exceed the clinically anticipated loads in the molar and premolar regions. However, clinical trials are necessary to validate the results.

Clinically, crown failure usually occurs under a complex type of stresses. However, all specimens in the current study were tested using vertical loads which appear to be appropriate for posterior teeth (Probster, 1992). Therefore, clinical implications of the current study must be limited to that application.

In this study, the specimens were loaded until failure in a single cycle, even though restorations may fail clinically through slow crack growth caused by cyclic fatigue loading (Baran et al., 2001). Subjecting the specimens to cycling fatigue loading could be considered in further investigation to give more information about the longevity and performance of Turkom-Cera crowns in condition relatively resemble the clinical situation.

# 6.5 Conclusions

Under the conditions of this study, it was found that:

- Turkom-Cera copings cemented to extracted teeth using the resin luting cement (Panavia F) provided load at fracture (1341.9 N) that was significantly higher to that obtained by Procera AllCeram (975.0 N) copings tested under the same conditions. However, there was no significant difference between the mean load at fracture of Turkom-Cera and In-Ceram (1151.6 N) and also between In-Ceram and Procera AllCeram copings tested under the same conditions. Thus, the null hypothesis was rejected.
- There is no influence of the finish line on the load at fracture of Turkom-Cera all-ceramic copings. Thus, the null hypothesis was accepted.
- 3) Comparison of the determined values of load at fracture for Turkom-Cera groups of samples before and after artificial ageing (water storage and thermocycling) showed that the artificial ageing had no significant effect on the load at fracture of Turkom-Cera copings. Thus, the null hypothesis was accepted.

#### **CHAPTER SEVEN**

# MARGINAL INTEGRITY OF TURKOM-CERA COMPARED TO TWO OTHER ALL-CERAMIC MATERIALS: EFFECT OF FINISH LINE

# 7.1 Introduction

Marginal fit is an important factor for the success and longevity of an indirect restoration because an inadequate adaptation of the restoration can result in damage to the tooth and its supporting periodontium (Suárez et al., 2003). Clinically, it is important that crown margins fit the prepared tooth precisely to minimize plaque accumulation and therefore reduce risk of gingivitis, periodontitis, secondary caries and pulpitis (Bergenholtz et al., 1982; Bader et al., 1991; Felton et al., 1991b; Mjör and Toffenetti, 2000). These defects are common reasons for the failure of restorations (Karlsson, 1986; Goodacre et al., 2003; Sailer et al., 2007).

The results of different studies on marginal discrepancy of all-ceramic crowns showed a high variation within one crown system. In-Ceram crowns varied from a mean marginal discrepancy of 28  $\mu$ m from one study to 161  $\mu$ m in (Pera et al., 1994; Sulaiman et al., 1997). Yeo et al., (2003) reported mean marginal openings of 112  $\mu$ m for In-Ceram crowns. Quintas et al., (2004) tested Procera AllCeram crowns and reported a mean marginal discrepancy of 25  $\mu$ m. However, May et al., (1998) reported mean marginal openings of less than 63  $\mu$ m (56  $\mu$ m to 63  $\mu$ m). Another study found marginal discrepancies of 83  $\mu$ m for Procera crowns (Sulaiman et al., 1997).

The evaluation of the marginal discrepancy of crowns depends on a number of factors; measurements of cemented or uncemented crowns, storage time and artificial ageing after cementation, type of abutment used for measurements, type of measuring instruments and location/quantity of measurements (Beschnidt et al., 1999; Groten et al., 2000; Good et al., 2009).

Many studies have evaluated the marginal discrepancies of single restorations fabricated using various systems and materials. In some studies, four measurements per specimen have been performed (Hung et al., 1990; Holmes et al., 1992; Leong et al., 1994; Sulaiman et al., 1997; Oruc and Tulunoglu, 2000; Hilgert et al., (2004); Goldin et al., 2005; Ayad, 2008). However, four measurements are not representative of the marginal gap in one specimen. According to Groten et al., (2000), fifty measurements are required to obtain clinically relevant information about gap size regardless of whether the measurement sites were selected in a systematic or random manner.

Regarding margin design, Shearer et al., (1996) found no significant difference between chamfer and shoulder margins in the fit of In-Ceram crowns. Whilst Suárez et al., (2003) found the same for Procera AllCeram crowns. Syu et al., (1993) reported no significant differences for marginal gaps among metal ceramic crowns with shoulder, shoulder-bevel, and chamfer finish lines. However, Hilgert et al., (2004) found that shoulder margin design for all-ceramic restorations presented better values of marginal gap than chamfer margin.

According to Limkangwalmongkol et al., (2009) there is no definite standard that exists regarding what constitutes clinically acceptable margin. All data should be analyzed under the consideration of the study design. McLean & von Fraunhofer, (1971) concluded that a marginal opening of 120  $\mu$ m represents the maximum clinically acceptable gap size. The studies on the marginal adaptation of Turkom-Cera are lacking. Therefore, the objectives of this study were:

- 1. To examine the marginal adaptation of Turkom-Cera, In-Ceram and Procera AllCeram copings.
- To determine the influence of the finish line on the marginal adaptation of Turkom-Cera copings.

#### Null hypotheses

- 1. There is no difference in the marginal adaptation of Turkom-Cera, In-Ceram and Procera AllCeram copings.
- 2. There is no influence of the finish line on the marginal adaptation of Turkom-Cera copings.

# 7.2 Materials and methods

#### 7.2.1 Materials used

Three types of all-ceramic systems were used for the coping production namely, Turkom-Cera<sup>™</sup> (Turkom-Ceramic (M), Puchong, Malaysia), In-Ceram (Vita Zahnfabrik, Bad Sackingen, Germany) and Procera AllCeram (Nobel Biocare, Göteborg, Sweden).

#### 7.2.2 Methods

#### 7.2.2.1 Specimen preparation

Forty sound and crack-free maxillary premolar teeth were used for this study. Thirty teeth were prepared with chamfer margin and 10 teeth with shoulder margin. The 30 prepared teeth with chamfer margin were randomly divided into three groups of 10 specimens each according to the type of all-ceramic system used. The 10 prepared teeth with shoulder margin were used with Turkom-Cera to study the influence of finish line on the marginal adaptation of Turkom-Cera all-ceramic system.

In the current study, the measurements of marginal adaptation were accomplished without cementing the copings. Therefore, the same specimens, before cementation, used for evaluation of the fracture resistance in Chapter 6 were used in this study. The methods of teeth collection, preparation and fabrication of all-ceramic copings have been discussed in Chapter 6. As mentioned before, the prepared teeth were embedded in epoxy resin 2.0 mm below the cemento-enamel junction. Prior to measurement, the macroscopic fit of all copings on the corresponding stone dies, and finally on the specimens were visually assessed. Copings that rocked or did not seat on the finish line were rejected and remade to ensure 10 copings per group.

#### 7.2.2.2 Marginal gap measurement

The copings were seated on the teeth without cementation by a custom made holding jig (Figure 7.1). The jig was developed to position the coping and the tooth model precisely, enabling the force to be applied parallel to the long axis of the tooth and to maintain the force applied so as to prevent the coping from dislodgement during measurement.



Figure 7.1: Front view of the holding jig used.

The holding jig was designed to accommodate a set containing a tooth model and a ceramic coping during each measurement, fixed in the frame by means of two 5-mm-wide screws positioned on both sides of the jig (Figure 7.2). Another 5-mm-wide screw, connected to a digital torque control motor (Tecnika; ATR, Dentsply, Pistoia, Italy),

was passed through the upper part of the jig until it touched the coping (Figure 7.3). To avoid damage to the copings by the upper screw, an additional silicone impression material was injected to a plastic tube, which was later fixed to the tip of the upper screw (Figure 7.1).



Figure 7.2: Specimen fixed in the holding jig.



Figure 7.3: The upper screw connected to the digital torque control motor.

A computer system consisting of a stereomicroscope (Olympus SZ61, Olympus Corp., Tokyo, Japan), a USB Color Digital Scientific Grade CCD camera (Xcam-alpha, Imaging Sources, Germany), a personal computer and a special software (Cell^B, Olympus Soft Imaging Solutions, Germany) was used to record the measurements (Figure 7.4).



Figure 7.4: The computer system used to record the measurements.

A 10-Ncm torque was placed on the upper screw using the digital torque control motor (Figure 7.3), and the assembly was taken to the table of the stereomicroscope in such a way that the long axis of the tooth was always parallel to the horizontal table of the microscope (Figure 7.5). This is to ensure a stable position for capturing the digital images by the camera without movement at each of the 4 sides (mesial, distal, buccal and palatal).



Figure 7.5: The jig-coping-tooth assembly on the table of the stereomicroscope.

The marginal gap of each specimen was reproduced at original magnification x30 (Stereomicroscope, Olympus SZ61, Olympus Corp., Tokyo, Japan) on a high-resolution ( $1280 \times 800$  pixel) computer monitor (Figure 7.6) and the images were captured with the digital camera (Xcam-Alpha) at each of the 4 sides, so that a video image of the marginal gap could be examined using the software (Cell^B). A stage micrometer (1/100 mm), provided by the manufacturer, was used for software calibration at each measurement session.

Then, the video images of the marginal gap were blindly examined using image analysis software (Cell^B). The marginal gap was determined as the vertical opening between the outermost edge of the coping margin and the prepared tooth margin. The marginal gap of each coping was measured three times at 50 points along the margin (15 mesial, 15 distal, 10 buccal and 10 palatal) that were randomly selected (Groten et al. 2000) in approximately equal distances using virtual screen ruler (Figure 7.7), for a total of 150 measurements per coping. The marginal fit of a coping was defined as a mean value of these 150 measurements. The mean value of marginal fit was calculated for each specimen, and this value was used to determine the mean marginal fit for each group.

# 7.2.2.3 Measurement of reliability

Prior to initiating the measurements, ten samples were selected randomly and subjected to reliability test. Marginal integrity was measured on three separate days for samples in the reliability test. Reproducibility of marginal integrity was analyzed using SPSS (17.0 for Windows) to compute the intraclass correlation coefficients value. The intraclass correlation coefficients value was 0.993 which indicate a high degree of reliability between measurements.



Figure 7.6: Marginal discrepancy image of one specimen (x30) on computer monitor.



Figure 7.7: Marginal discrepancy evaluation using Cell^B image processing software.

#### 7.2.2.4 Statistical analysis

The first objective was to determine the marginal integrity of Turkom-Cera copings compared to In-Ceram and Procera AllCeram copings. Descriptive statistics will be recorded for marginal integrity of the three groups. The normally distributed data (Histogram and Shapiro-Wilk) will be analyzed by One Way ANOVA to achieve the objective, provided that equal variances will be assumed (Levene's test). Then, Tukey's HSD test will be carried out for post-hoc comparisons.

Whenever assumptions of normal distribution and equal variances of marginal integrity between the three groups will not be met, an equivalent nonparametric Kruskal-Wallis test will be conducted. Subsequently, a post hoc test using Mann-Whitney tests with Bonferroni correction will be performed to test which pair of groups differ from each other significantly. Regarding the effect of finish line design on marginal integrity of Turkom-Cera copings, descriptive statistics will be recorded for marginal integrity of each group. Then, independent samples t-test should be used to determine the significant differences in marginal integrity between the two groups of Turkom-Cera copings with chamfer and shoulder finish lines. However, this is dependent on the assumptions of normal distribution (histogram and Shapiro-Wilk test) and equal variances (Levene's test) of marginal integrity between the two groups to be met. Whenever assumptions of normal distribution and equal variances of marginal integrity between the two groups to be met. Whenever assumptions of normal distribution and equal variances of marginal integrity between the two groups will not be met, an equivalent nonparametric Mann-Whitney tests will be performed to determine the significant difference between the two groups. Statistical analysis will be carried out using a computer program (SPSS Inc., Chicago, IL). Statistical significance will be set at  $\alpha$ = 0.05.

# 7.3 Results

#### **7.3.1** Effect of ceramic material on the marginal integrity

The objective is to test if the mean marginal integrity of Turkom-Cera, In-Ceram and Procera AllCeram copings differ from each other. Descriptive analysis was done and the mean marginal gap and standard deviation for the three groups are shown in Figure 7.8.



**Figure 7.8:** Marginal integrity (µm) of the three tested ceramics (Mean + SD). \* *Details on statistical analysis see appendix VI* 

The histogram and Shapiro-Wilk test were used and showed that there was approximately normal distribution of marginal integrity of the three groups (Appendix VI).

Since the assumption of normal distribution was met, the equality of variances (homogeneity) was tested using the Levene's test (Appendix VI) and showed that there was no significant deviation from homogeneity (p=0.834). Thus, the parametric One Way ANOVA procedure was used to achieve objective. The results were recorded in Table 7.1. There was a significant difference in marginal integrity between the three groups (p<0.001).

**Table 7.1:** Compare marginal integrity (μm) between Turkom-Cera, In-Ceram and<br/>Procera AllCeram copings by One Way ANOVA

Ceramic	Ν	Mean	SD	F Statistics (df)	P value <sup>a</sup>		
Turkom-Cera	10	49.19	12.18		< 0.001		
In-Ceram	10	71.51	13.74	22.99			
Procera	10	34.38	10.89	(2,27)			
<sup>a</sup> One Way ANOVA was used.							
Significant level was set at 0.05.							

Further analysis using Tukey's HSD Post Hoc Test was used to determine the pair of means that differ significantly (Appendix VI). The test showed that the mean marginal gap of Turkom-Cera, In-Ceram and Procera AllCeram copings differ from each other significantly (p<0.05) (Table 7.2). In-Ceram gave the highest mean marginal gap (71.51 ± 13.74 µm), whereas, Procera AllCeram gave the lowest mean marginal gap (34.38 ± 10.89 µm) and Turkom-Cera was in between (49.19 ± 12.18).

Pairewise comparison	Mean (SD)	Mean Difference	<i>P</i> value			
Turkom-Cera	49.19 (12.18)	-22.32	0.001*			
vs In-Ceram	71.51 (13.74)	22.32				
Turkom-Cera	49.19 (12.18)	14.81	0.032*			
vs Procera	34.38 (10.89)	-14.81				
In-Ceram	71.51 (13.74)	37.13	<0.001*			
vs Procera	34.38 (10.89)	-37.13				
* 2 pairs of means are significantly different by Tukey HSD Test						

**Table 7.2:** Multiple pairwise comparisons of marginal integrity (μm) of Turkom-Cera,In-Ceram and Procera AllCeram copings using Tukey's HSD Test

#### 7.3.2 Effect of finish line on the marginal integrity of Turkom-Cera

Specimens were divided into two groups according to finish line used; Group 1 (Turkom-Cera with chamfer finish line) and Group 2 (Turkom-Cera with shoulder finish line). The mean marginal integrity and standard deviation for the two groups are represented in Figure 7.9.



**Figure 7.9:** Marginal integrity  $(\mu m)$  of the Turkom-Cera with two finish lines.

Histogram and Shapiro-Wilk test (Appendix VI) were used and showed approximately normal distribution of marginal integrity of the two groups. In addition, the equality of variances was assumed as indicated by Levene's test (p=0.560). Thus, independent samples t-test was used to determine the significant differences in marginal integrity

between the two groups (Table 7.3). There was no significant difference in marginal integrity between the two groups (p=0.323). Therefore, there was no influence of the finish line on the marginal integrity of Turkom-Cera all-ceramic copings.

**Table 7.3:** Comparison of marginal integrity (µm) of the two groups using Independent t-test

Variable	Turkom-Cera (Chamfer)	Turkom-Cera (Shoulder)	Mean differ.	t statistic	Р	Levene's Test	
	(n=10) Mean (SD)	(n=10) Mean (SD)	(95% CI)	( <b>df</b> )	value	F	Sig.
Marginal			5.22	5.14			
integrity	49.2 (12.2)	44.0 (10.8)	(-5.58, 16.03)	(18)	0.323	0.36	0.560

# 7.4 Discussion

#### 7.4.1 Methodology

The development of ceramic systems with improved strength and esthetics has widened the use of all-ceramic restorations for anterior and posterior teeth. In addition to fracture resistance and esthetics, marginal accuracy is of clinical importance and can influence the success and longevity of all-ceramic restorations (Hilgert et al., 2003; Wolfart et al., 2003; Pilathadka et al., 2008; Limkangwalmongkol et al., 2009).

In this study, extracted natural teeth were used as abutments. Natural teeth present a great variation due to their age, structure of hydroxyapatite and dimensions and history of the teeth, making it difficult to standardize the abutments (Beschnidt and Strub, 1999; Hilgert et al., 2004). Several investigators have used metal or acrylic resin dies for measurements of the marginal accuracy (Pera et al., 1994; Sulaiman et al., 1997; Nakamura et al., 2000; Tinschert et al., 2001; Hilgert et al., 2004, Lee et al., 2008). The advantage of this method is that there is less variables and providing possibility of getting standardized preparation for all abutments. However, abutment made of steel or \* Details on statistical analysis see appendix VI

resin gives neither real information about the microstructure of the hard tissue of the teeth after preparation, nor about the chemo-mechanical adaptation of the luting material to the dentine (Beschnidt and Strub, 1999).

Regarding cementation of the copings, some investigators make use of it (Sorensen, 1990; Suárez et al., 2003; Albert and El-Mowafy, 2004), because they think that the most important misfit is the one that occurs in vivo, when the crowns are cemented to the abutments. In this study, as well as in other studies (Rinke et al., 1995; Groten et al., 1997; Sulaiman et al., 1997; Nakamura et al., 2000; Tinschert et al., 2001; Yeo et al., 2003), this was not accomplished. A study by Tinschert et al., (2001) stated that when cementing the crowns, the precision of primary adaptation is lost, and the influence of the cement type, viscosity and luting techniques become preponderant. Some authors believed that the cementation should not be used to compare systems for construction of crowns as they may influence the precision of primary adaptation of these restorations (Hilgert et al., 2003; Hilgert et al., 2004).

Since the coping was seated on the tooth without cementation, a special holding jig was modified and used to hold the coping. In addition, the jig helped to position the coping and the tooth model precisely, enabling the force applied by the digital torque control motor to be directed parallel to the long axis of the tooth and to maintain a constant force during measurement. Although a torque of 20 to 30 Ncm is frequently employed in gap evaluations (Lee et al., 2008), a study by Quintas et al., (2004) found that most ceramic copings fractured above 10 Ncm. Therefore, in this study, a value of 10 Ncm was applied as a maximum torque to avoid the breakage of copings during marginal discrepancy evaluation. This was because no cement material was used between the coping and supporting tooth, besides the fact that the thin ceramic copings (0.6 mm)

restricted the amount of torque applied to the upper screw of the holding jig. Furthermore, an additional silicone impression material inside a plastic tube was fixed to the tip of the upper screw to avoid damage to the copings.

Marginal fit of the copings was measured in this study without veneering in accordance with many researchers (Hilgert et al., 2003; Suárez et al., 2003; Quintas et al., 2004; Bindl and Mörmann, 2005; Beuer et al., 2008c). Previous studies (Pera et al., 1994; Shearer et al., 1996; Groten et al., 1997; Sulaiman et al., 1997, Bindl and Mörmann, 2003) have proved that it is mainly the retainer which determines the overall fit of a veneered restoration and various phases of porcelain firing did not significantly influence marginal adaptation of all-ceramic crowns, thus validating the use of ceramic copings in the present study.

There are many different locations between a tooth and a restoration where measurements can be made. In this study, the gap between the external edge of the coping and the prepared tooth margin was defined as the standard for marginal accuracy (Beschnidt and Strub, 1999; Song and Cho, 2000; Cho and Kang, (2002); Yeo et al., 2003; Okutan et al., 2006; Lee et al., 2008; Good et al., 2009). In the present study, distance between the coping margin and the tooth was recorded at 50 points that were randomly selected in approximately equal distances. According to Groten et al., (2000) fifty measurements are required for clinically relevant information about gap size regardless of whether the measurement sites are selected in a systematic or random manner.

Different aspects of tooth preparation design have been cited in the literature. However, considerable focus has still been directed towards the most appropriate finish line to use as new innovative ceramic systems are being introduced (Ayad, 2008). In this study, the marginal adaptation of Turkom-Cera copings was evaluated in function of the finish line of the preparation. Two types of finish lines were used, the chamfer and round shoulder, because they are the most recommended in the literature (Shearer et al., 1996; Beschnidt and Strub, 1999; Hilgert et al., 2003; Suárez et al., 2003; Yeo et al., 2003; Hilgert et al., 2004) for the preparation of all-ceramic crowns due to their horizontal configuration. With regard to a standardized preparation, the circumferential chamfer/shoulder width was 1.2 mm. All margins were placed apically to the cement-enamel junction.

#### 7.4.2 Effect of ceramic materials

According to the results of this study, the mean marginal discrepancy for Turkom-Cera  $(49.2 \pm 12.2 \ \mu\text{m})$ , In-Ceram  $(71.5 \pm 13.7 \ \mu\text{m})$  and Procera all-ceramic copings  $(34.4\pm10.9 \ \mu\text{m})$  differ from each other significantly (*p*<0.05). The Procera (34.38 ± 10.89 \ \mu\text{m}) copings had the lowest marginal discrepancy, whereas, In-Ceram (71.5 ± 13.7 \ \mu\text{m}) copings had the highest marginal discrepancy.

In this study, the copings that were found to rock or did not seat on the finish line were rejected and remade. Two In-Ceram and two Turkom-Cera copings were remade. However, none of the Procera copings were rejected. While the number of steps involved in the fabrication of all-ceramic crowns was not a direct sign of the quality of the marginal integrity, one may suggest that the more the steps involved and the more sensitive the techniques, the more possible that technical errors will occur. The differences in marginal integrity between the three all-ceramic systems tested may be related to the sensitivity of the technique and the number of steps in fabricating these

copings. The reproducible technique (CAD/CAM) and fewer laboratory steps of the Procera system would seemed to have contributed to these results, because none of the Procera copings were remade.

The mean marginal discrepancy recorded in the present study of 71.5  $\mu$ m for In-Ceram copings was in agreement with the finding of Quintas et al., (2004) who found a mean marginal discrepancy for In-Ceram of 57  $\mu$ m. The mean marginal discrepancy of 71.51  $\mu$ m for In-Ceram copings in this study was better than previously reported. Sulaiman et al., (1997) have tested In-Ceram crowns and found a mean marginal discrepancy of 161  $\mu$ m. Yeo et al., (2003) and Grey et al., (1993) evaluated the marginal discrepancy of conventional In-Ceram, which were 112  $\mu$ m and 123  $\mu$ m, respectively. Conversely, Pera et al., (1994) reported the marginal discrepancy of 28  $\mu$ m for In-Ceram crowns.

The current findings on the marginal discrepancy of Procera AllCeram copings in this study (34.4  $\mu$ m) were in agreement with those reported by Suárez et al., (2003) & Quintas et al., (2004) who found a mean marginal discrepancy of 38  $\mu$ m and 25  $\mu$ m, respectively. However, the mean marginal gap value (34.4  $\mu$ m) for Procera AllCeram recorded in the present study was smaller than previously reported values. An in vitro study of the marginal fit of Procera AllCeram crowns reported mean marginal openings of 63  $\mu$ m (May et al., 1998). Other researchers found marginal discrepancies for Procera AllCeram AllCeram crowns that range from 80 to 120  $\mu$ m (Sulaiman et al., 1997; Boening et al., 2000).

#### 7.4.3 Effect of finish line

This study has also evaluated the marginal adaptation of Turkom-Cera copings in relation to the finish lines of the preparations. The result of the present study showed that the finish line did not influence marginal adaptation of Turkom-Cera copings.

Although shoulder margin (44.0  $\mu$ m) has shown less marginal gap than chamfer margin (49.2  $\mu$ m) in this study, statistical analysis revealed no significant differences between them (p>.05). This is in agreement with previous studies. Shearer et al., (1996) showed no significant difference between chamfer and shoulder margins in the fit of In-Ceram crowns. Suárez et al., (2003) found no significant difference in marginal gap between chamfer and shoulder finish line designs of Procera AllCeram crowns. Quintas et al., (2004) found no significant differences in marginal gap of Procera, In-Ceram and Empress 2 all-ceramic copings with chamfer and shoulder finish lines. Syu et al., (1993) reported no significant differences for marginal gaps among metal-ceramic crowns with shoulder, shoulder-bevel, and chamfer finish lines.

In contrast, Lin et al., (1998) reported that the finish line has influenced the marginal adaptation of Procera AllCeram copings. A study by Rinke et al., (1995) showed that using a shoulder preparation with conventional and copy-milled In-Ceram crowns, produced significantly smaller marginal gaps compared to a chamfer preparation. A similar result was reported in another study. Hilgert et al., (2004) demonstrated that improved marginal fit was obtained with In-Ceram crowns fabricated using shoulder finish line compared with chamfer margin. While Pera et al., (1994) demonstrated that improved marginal fit was obtained with In-Ceram crowns fabricated on chamfer tooth preparations compared with shoulder margin.

Mitchell et al., (2001) reported that the edge of the shoulder finish line was easier to identify during crown fabrication than the chamfer finish line, and thus, the shoulder finish line ensured improved marginal fit. Hilgert et al., 2004 assumed that the better adaptation with shoulder finish line is attributed to a greater operational easiness by a larger volume in the margins. Besides, the sharper borders, proportionate for the chamfer finish line, can be more easily damaged during finishing or sandblasting.

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An explanation of the lack of agreement may be variation in the methods used by various investigators studying marginal accuracy. Good et al., (2009) suggested that the cause could be the use of different measuring instruments. Sample size, number of measurements per specimen and the method of technical crown manufacturing and the skills of the technician may also have influenced the variation (Beschnidt and Strub, 1999; Groten et al., 2000; Yeo et al., 2003). This study showed clinically acceptable marginal discrepancy of all groups tested.

It has been seen in the literature that the opinions on the clinical relevance of the size of marginal discrepancies are controversial. As a clinical goal, it has been suggested that marginal gaps of cemented restorations should range from 25 to 40  $\mu$ m. However, marginal openings within this range are seldom achieved clinically (Beuer et al., 2008c).

The debate over the maximum acceptable gap size includes a wide range of values, from about 50 to 120  $\mu$ m. Some authors agree that marginal discrepancies ranging from 50  $\mu$ m to 100  $\mu$ m seemed to be clinically acceptable with regard to longevity of the restorations (Pera et al., 1994; Hung et al., 1990; Weaver et al., 1991; May et al., 1998).

On the contrary, McLean & von Fraunhofer, (1971) have examined more than 1000 crowns after five years of clinical service and concluded that marginal opening of less than 120  $\mu$ m was clinically acceptable. In any way, all the values (mean between 34.38 and 71.51  $\mu$ m) found in this study are inside of this range, giving us larger safety for the clinical use of these restorations. However, the measurements recorded with a crown seated but not cemented demonstrate the minimal misfit of each crown, which is likely to increase after cementation because of the hydraulic backpressure of cement (Hoard et al., 1978; Moore et al., 1985; Mitchell et al., 2001; Okutan et al., 2006).

As a limitation of this study, marginal opening was measured, but the internal fit of the crowns was impossible to measure in this experimental design. Although certain studies focused on marginal fit, other studies (May et al., 1998; Boening et al., 2000; Nakamura et a., 2003) also evaluated the internal fit of the crowns. Measuring the internal fit of artificial crowns requires cementation and sectioning the specimens. In the case of sectioning, the number of measurements per specimen is limited.

Another limitation of this study was the lack of artificial ageing of the crowns, such as thermal cycling, which might affect the results. Further studies are required for developing new experimental designs to measure both the marginal and internal fit.

#### 7.5 Conclusions

Within the limitations of this study, the following conclusions were drawn:

- 1. The mean marginal discrepancy of the all-ceramic copings was, in descending order: In-Ceram (71.5 μm), Turkom-Cera (49.2 μm) and Procera (34.4 μm).
- The three different all-ceramic copings demonstrated significant differences among each other with respect to the marginal discrepancy. Thus, the null hypothesis was rejected
- 3. The mean marginal discrepancies of Turkom-Cera, In-Ceram and Procera AllCeram copings were within the range of clinical acceptance.
- 4. There were no significant differences in the mean marginal discrepancy of Turkom-Cera copings among the chamfer and shoulder groups (p>.05). Thus, the null hypothesis was accepted. In addition, both values were within clinically acceptable limits. Therefore, both finish lines can be used for the preparation of Turkom-Cera copings.

CHAPTER EIGHT

# CLINICAL EVALUATION OF TURKOM-CERA ALL-CERAMIC CROWNS

# 8.1 Introduction

All-ceramic restorations have desirable characteristic properties such as biocompatibility, aesthetics and low thermal conductivity (Odén et al., 1998). Early types of metal-free ceramics, used in the conventional porcelain jacket crowns, did not enjoy success in dentistry especially in the posterior region (Shimada et al., 2002). To overcome this problem, an alumina-reinforced porcelain core material was developed by McLean for the fabrication of such crowns (McLean and Hughes, 1965). A veneer porcelain placed on a core containing approximately 50 % fused alumina crystals, compared to the conventional feldspathic porcelain level of about 19 %, resulted in a dental ceramic with flexural strength from 100 to 130 MPa (Giordano et al., 1995).

The popularity of high-strength ceramic systems is increasing, and the range of their clinical indications is expanding constantly. Glass infiltrated alumina ceramic (eg. In-Ceram Alumina, Vita Zahnfabrik, Bad Sackingen, Germany), densely sintered aluminium oxide ceramic (eg, Procera AllCeram, Nobel Biocare AB, Gothenburg, Sweden), and zirconium oxide ceramic (Procera AllZirkon, Nobel Biocare, Göteburg, Sweden; Cercon, Dentsply Ceramco, Burlington, NJ, USA; Lava, 3M ESPE, St. Paul, MN, USA) are popular oxide-based high-strength ceramic materials which were developed in an attempt to improve the strength of metal-free restorations as well as deliver more aesthetic results than conventional porcelain-fused-to-metal restorations (Özcan et al., 2001; Blatz et al., 2004; Valandro et al., 2006; Della Bona et al., 2007).

Advances in dental ceramics include the introduction of a high-strength all-ceramic core material (Turkom-Cera<sup>™</sup>, Turkom-Ceramic (M) Sdn. Bhd., Puchong, Malaysia), particularly with aluminium oxide. A stone die is covered by a red plastic foil 0.1 mm thick and dipped in the Turkom-Cera Alumina Gel (99.98 %) following the manufacturer's instructions.

After drying of the alumina gel, the coping with the red plastic foil is removed from the stone die and sintered for 5 minutes at 1150 °C. The sintered coping is crystal hardened in a second firing process using Turkom-Cera crystal powder for 30 minutes at 1150 °C. Like all other infiltration ceramics, this core is then veneered with porcelain adjusted to have the correct coefficient of thermal expansion (See Appendix I).

The improved mechanical properties of all-ceramic materials such as toughness and strength have increased their clinical use in multi-unit fixed partial dentures (Taskonak and Sertgoz, 2006). However, the superior toughness and strength of ceramic materials are not the only factors that determine long-term survival.

Certain intraoral factors like stress corrosion and subcritical crack growth due to cyclic forces during repetitive occlusal contact; constant exposure to a moist and bacteria-rich environment; ingestion of hot or cold liquids and acids; and heavy or inadequate tooth brushing have significant effect on long-term survival of dental ceramics (Rekow and Thompson, 2001; Zhang and Lawn, 2005; Toksavul and Toman, 2007). In addition, it has been stated the specimens used for in vitro testing of dental ceramics sometimes differ significantly in both size and structure from the restorations they represent (Kelly, 1995). Therefore, in vivo evaluation has been the basis for establishing criteria for acceptable crowns (Haselton et al., 2000). The aim of this study was to evaluate the clinical performance and patient satisfaction of Turkom-Cera all-ceramic crowns over a variable observation period.

# 8.2 Materials and methods

This is a preliminary prospective study to evaluate the durability of Turkom-Cera<sup>TM</sup> all ceramic crowns through a two-year clinical trial and to complement the mechanical tests done on this material.

#### 8.2.1 Subjects

Between February 2007 and September 2009, at the Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia, 20 Turkom-Cera all-ceramic crowns (9 Premolars and 11 Molars) were placed in 16 patients (13 women and 3 men, ages 22-63 years), who requested for single crown restorations. These crowns were done only after careful examination of candidate and were found to be indicated and suitable for them.

The restored teeth had been affected by several problems: primary caries, defective amalgam or composite restorations, failed old crowns, endodontically treated teeth, and fractured teeth. Of the 20 Turkom-Cera crowns, 16 (80%) crowns were placed on vital teeth, and the remaining 4 (20%) crowns were placed on endodontically treated teeth. Vital teeth were restored with amalgam or composite restorative materials. Of those 4 endodontically treated teeth, 1 was reconstructed with a prefabricated screw-shaped post and amalgam core as a result of severe coronal destruction. The other 3 endodontically treated teeth were intact and did not require a post and core restoration and restored with composite material. Natural teeth were present in the opposing arch of 15 (93.75%) of the patients, while 1 (6.25%) of the patients had opposing ceramic material. Detailed information on the patients, location and distribution of the crowns related to evaluation time is presented in Table 8.1.

			No. of crowns*		Evaluation time
Patient	Age (Years)	Gender	Premolars	Molars	(months)
1	42	М	24,25		12, 12
2	27	F		46	12
3	29	F		16	15
4	63	F		36	20
5	47	F	45	37	18, 18
6	25	F	24		18
7	55	М		37	18
8	47	F	14	36	21, 21
9	37	F		46	24
10	59	F		27	24
11	49	F	14		28
12	26	М		36	27
13	53	F	14, 15		27, 27
14	22	F		36	28
15	25	F		36	29
16	52	F		47	31

**Table 8.1:** Location and observation time of the 20 crowns placed in 16 patients

\*Fédération Dentaire Internationale (FDI) tooth-numbering system

Prior to start, the study was reviewed and approved by the ethics committee of the Faculty of Dentistry, University of Malaya, Ethics approval No: UM.D/PD/211/07 (Appendix VII). The patients were carefully selected for this study using the following criteria: 1) over 18 years old with good general and dental health and with no active tooth decay (caries) present and no bleeding on probing (to indicate the absence of inflammation), 2) do not have any existing temporomandibular disorder (e.g. clicking, popping, pain on opening) or parafunctional habits (e.g. bruxism, clenching), 3) absence of removable or fixed orthodontic appliance 4) good oral hygiene and compliance, and 5) an interest in aesthetics.

The patients were informed verbally about the research methodology, risks and benefits as well as their rights to quit participating in this research at any time without any consequence on their future visits at Faculty of Dentistry, University of Malaya. Furthermore, the patients have been given the patient information sheet containing all information about the research (Appendix VII).
A written informed consent was requested and obtained from each patient indicating that the all-ceramic material used was new and no long-term clinical data were available at the time of insertion (Appendix VII). All patients were asked to participate in the study at the time of examination, and asked about their general satisfaction with the restorations at the end. All patients agreed to a recall period of 2-3 years with at least one recall visit every six months.

### 8.2.2 Treatment

Photographs and pre-apical radiographs were taken for the abutment teeth. Alginate impression were made for the jaws and cast in stone in order to fabricate the special tray and temporary crowns. Table 8.2 shows an overview of the clinical protocol of the study.

**Table 8.2:** Overview of the clinical protocol

1	Patient selection, information, consent form, photograph, radiograph and primary impression
2	Tooth preparation, final impression, provisional crown
3	Try-in of restoration, corrections
4	Adhesive cementation, finishing of restoration
5	Clinical reevaluation after cementation
6	Clinical reevaluation at time of recall

### 8.2.2.1 Preparation of teeth, impression making and pouring

A circumferential shoulder finish line with rounded internal line angles was used for the preparation of abutment teeth (Figure 8.1).



Figure 8.1: Buccal view of prepared lower right first molar.

The primary preparation was performed with medium and coarse diamond burs (837KR.314.012, 847KR.314.016, Komet Dental, Gebr. Brasseler, Lemgo, Germany). After tooth preparation, a finer diamond bur (856EF012; Komet) was used for definitive tooth contouring and finishing of the margin. The smoothness of the preparation and ability to transfer the details to the refractory die is essential for the precision and fit of the crown. In most instances, the width of the shoulder was approximately 1.2 to 1.5 mm. The teeth were prepared with occlusal reduction of approximately 2.0 mm. In the 20 crowns done, the preparation margins were located at the gingival margin or slightly (0.5 mm) subragingival. This is to facilitate impression making and evaluation of the marginal adaptation of the crown, while helping to maintain periodontal health.

Where needed and to ensure high definition of the margins in impressions, gingival displacement was obtained using a retraction cord (No. 00 Ultrapack®, Ultradent, South Jordan, Utah, USA). Complete-arch impressions of the prepared teeth were taken using polyether impression material (Impregum Penta, 3M ESPE, Seefeld, Germany) in custom-made self-curing acrylic resin (Ostron 100, GC Corp., Tokyo, Japan) trays.

The impressions of the prepared teeth were poured with die stone (Densite, Shufo, Japan), sectioned and pinned using the Pindex system (Coltene/Whaledent Inc., NY, USA) to allow accurate repositioning of the sectioned pieces and preparation of the Turkom-Cera core (Figure 8.2).

Complete-arch alginate impressions (Aroma Fine DF III, GC Corp., Tokyo, Japan) were made of the opposing dentitions and immediately poured with dental stone. Interocclusal registrations were recorded (Alminax, Whip-Mix Corp., Louisville, Ky) and the master casts were mounted on a semiadjustable articulator (Kavo, Leutkirch, Germany) (Figure 8.3).



Figure 8.2: The cast after sectioning.



Figure 8.3: The master casts mounted on the articulator.

Provisional crowns (Trim, Bosworth Co., Skokie, USA) were prepared to maintain gingival health and tooth position, and then cemented with temporary cement (Temp Bond NE, Kerr GmbH, Karlsruhe, Germany) (Figure 8.4).



Figure 8.4: The provisional crown on lower right first molar.

#### 8.2.2.2 Fabrication of Turkom-Cera copings and veneering

The detailed step by step fabrication procedures for Turkom-Cera coping are in Appendix I. In summary, the sectioned stone die is covered by a red plastic foil 0.1 mm thick and dipped in the Turkom-Cera Alumina Gel (99.98 %) following the manufacturer's instructions. After drying of the alumina gel, the coping with the red plastic foil is removed from the stone die and sintered for 5 minutes at 1150 °C. After that, the sintered coping is crystal hardened in a second firing process using Turkom-Cera crystal powder for 30 minutes at 1150 °C.

The 20 Turkom-Cera copings were prepared in the dental laboratory by the same certified dental technician following the manufacturer's instructions with a thickness of 0.6 mm. Like all other infiltration ceramics, the 20 Turkom-Cera copings were then veneered using feldspathic veneering ceramic material (Vintage AL, Shofu Inc., Kyoto, Japan) by the same certified dental technician following the manufacturer's instructions.

#### 8.2.2.3 Try-in and cementation

The temporary crowns were carefully removed, and all temporary cement fragments thoroughly cleaned from the abutments using cotton pellets and pumice slurry. The fit of the crown was evaluated intraorally to ensure complete seating on the abutment. Interproximal contacts were checked using waxed dental floss and 12µm thick articulating film (Arti-Fol Metalic, Dr. Jean Bauch KG, Köln, Germany), and if needed, minor adjustment was performed using a finishing diamond burs (Komet). Crowns that were found to rock or did not seat fully on the finish line were rejected and remade to avoid any adjustments of the fitting surface.

According to Davies (2004), the ideal articulating paper should not be more than 40 microns in thickness. Therefore, the occlusion and articulation of the crowns were evaluated carefully using 40 $\mu$ m thick articulating paper (Bausch Occlusionspapier, Dr. Jean Bauch KG, Köln, Germany), and the adjusted crowns were glazed before cementation. In addition to internal fit, proximal contacts, marginal adaptation and occlusal relationship, the individual crowns were also carefully evaluated in terms of shade match, surface texture and contour. After the trial insertion, the internal surfaces of the crowns were conditioned by air abrasion with 50  $\mu$ m aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) particles at an air pressure of 2.5 bars.

The copings were then steam cleaned and dried. The crowns were luted with a resin luting cement (RelyX U100, 3M ESPE, Seefeld, Germany). RelyX U100 is a selfadhesive resin luting cement. Etching and the use of primer and/or bonding as a pretreatment of the abutment are not necessary. At the cementation appointment, the gingival margins surrounding the abutment teeth were healthy, with no signs of color change or bleeding. Moisture control was performed by means of cotton rolls and highvelocity evacuation during cementation.

The cement was dispensed on the mixing pad, mixed with a plastic spatula for 20s and applied to the internal surface of the crown. Then, the crown was seated on the abutment tooth and the patient was instructed to bite gently on a cotton roll. Initial light-curing was performed for 2 seconds. Like other RelyX cements, the excess was easily removed with a dental probe and waxed dental floss. The luting cement was then polymerized from the occlusal and each margin using visible light with an intensity of 480 mW/cm<sup>2</sup> (Coltolux3, Coltene/Whaledent Inc., Mahwah, NJ, USA) for 20 seconds. After the luting cement had set, the occlusal contacts in centric and eccentric relations

were reevaluated. None of the 20 Turkom-Cera crowns needed to be adjusted after cementation. The margins were gently finished using tungsten carbide finishing burs (E.T carbide set, Komet) and Soflex discs (3M, St. Paul, MN). All clinical steps, from preparation to luting, were performed by the same investigator.

### 8.2.3 Evaluation criteria

The Turkom-Cera all-ceramic crowns were examined in accordance with the United States Public Health Service (USPHS) criteria (Table 8.3) (Bayne and Schmalz, 2005; Cvar and Ryge, 2005). Evaluation forms were developed for both clinical data and patient evaluation. Initial soft tissue health was confirmed by the absence of bleeding on probing throughout checkups. Two independent calibrated examiners evaluated the Turkom-Cera all-ceramic crowns for the following: (1) marginal integrity, (2) shade compatibility, (3) surface texture, (4) anatomic form, (5) secondary caries, (6) wear of crown and opposing dentition, and (7) cracks and fractures of the crowns. The modified USPHS criteria were used to assign a rating of Alpha, Bravo, or Charlie to each of the 7 categories of evaluation at baseline and subsequent recall appointments (Table 8.3).

Each crown was evaluated 7 days after cementation (baseline), and the patients were reexamined at intervals of 6 months for the following period. Clinical examinations included the use of a mirror, sharp explorer, radiographs and photographs. The restorations were evaluated over a period ranging from a minimum of 12 to a maximum of 31 months (mean, 21.5 months) after insertion. The distribution of the crowns related to the observation time is shown in Table 8.1. Alpha, Bravo and Charlie rankings were recorded and percent distributions were analyzed for each 6 months.

Category	Rating	Characteristic
Marginal integrity	Alpha (A)	Explorer does not catch when drawn across the surface of restoration toward the tooth, or, if the explorer does catch, there is no visible crevice along the periphery of the restoration.
	Bravo (B)	Explorer catches or there is visible evidence of a slight crevice, into which the explorer penetrates, indicating that the edge of the restoration does not adapt closely to the tooth structure.
	Charlie (C)	Explorer catches and penetrates a significant crevice defect that extends into the dentine.
Shade	Alpha (A)	Restoration appears to match the shade and translucency of the surrounding tooth tissues.
	Bravo (B)	Restoration does not match the shade and translucency of surrounding tooth tissues, but mismatch is within the normal range of the patient's tooth shades.
	Charlie (C)	Restoration does not match the shade and translucency of the surrounding tooth structure and mismatch is outside the normal range of the patient's tooth shades and translucency.
Surface texture	Alpha (A)	Smooth surface.
	Bravo (B)	Slightly rough or pitted, can be refinished.
	Charlie (C)	Rough, cannot be refinished.
Anatomic form	Alpha (A)	Restoration contour is in functional harmony with adjacent teeth and soft tissues.
	Bravo (B)	Restoration is slightly overcontoured/undercontoured.
	Charlie (C)	Restoration contour is not in functional harmony with adjacent teeth and soft tissues.
Secondary caries	Alpha (A)	No visual evidence of dark, deep discoloration adjacent to the restoration or tactile evidence of caries with the explorer.
	Bravo (B)	Visual evidence of dark, deep discoloration adjacent to the restoration, but no tactile evidence with the explorer that caries has penetrated into the dentine.
	Charlie (C)	Visual evidence of dark, deep discoloration adjacent to the restoration and tactile evidence that caries has penetrated into the dentine.
Wear	Alpha (A)	The restoration does not exhibit any signs of occlusal wear.
	Bravo (B)	The restoration exhibits slight signs of occlusal wear.
	Charlie (C)	The restoration exhibits considerable signs of occlusal wear.
Presence of cracks	Alpha (A)	No evidence of cracking, crazing and fracture within the restoration.
and fracture	Bravo (B)	Evidence of cracking or crazing within the restoration.
	Charlie (C)	Evidence of fracture.

Table 8.3:         Criteria for Modified USPHS rating used in this study*
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\* After (Lehner et al., 1997; Haselton et al., 2000; Otto, 2004; Taskonak, and Sertgoz, 2006)

All restorations were evaluated by the two examiners according to modified USPHS criteria. The two examiners worked as a team. The results were recorded separately on documentation sheets and compared to each other immediately after clinical examination. Whenever there was a difference in assessment results between the two examiners, joint examination was performed and a common rating was agreed upon (Cvar and Ryge, 2005). Difference in assessment results occurred mainly in relation with colour ratings; more specifically, there was some uncertainty as to whether the colour match to natural teeth should have been rated alpha or bravo. To be consistent, the same Dental Technician, who has been trained on Turkom-Cera system and technique, was responsible for the production of all crowns in the study.

In addition, subjects were asked about their experience with possible postoperative sensitivity and their general satisfaction with the restorations (Table 8.4).

 Table 8.4:
 Criteria for postoperative sensitivity and patient satisfaction\*

<b>Postoperativ</b>	<u>e sensitivity</u>
Alpha	Had no sensitivity at all to date
Bravo	Had slight sensitivity
Charlie	Unbearable sensitivity (patient asks for replacement)
Satisfaction of	of Patients with restoration
Alpha	Yes, I am satisfied; highly satisfied
Bravo	I am somewhat satisfied, would do it again
Charlie	I am not satisfied, would not do it again

\*(After Lehner et al., 1997)

### 8.2.4 Reliability test

To determine the interexaminer reliability, the restorations of the first 5 patients were assigned to both examiners for calibration. Each examiner has evaluated the patient independently, according to modified USPHS criteria, and recorded the results on documentation sheets. After that, the results of the two examiners were subjected to reliability analysis. The Kappa statistic was performed using SPSS (SPSS software V17.0 for Windows, SPSS, Chicago, IL, USA).

The general consensus in characterizing the different ranges of values for kappa with respect to the degree of agreement is that kappa values greater than 0.75 are considered to have a high degree of agreement beyond chance. Whereas, values below 0.40 have a poor degree of agreement beyond chance, and values between 0.40 and 0.75 represent a fair to good level of agreement beyond chance (Fleiss et al. 2003).

In this study, interexaminer reliability test yielded Kappa value of 0.785, which is greater than 0.75, giving evidence of good agreement between examiners (Fleiss et al. 2003).

### **8.2.5** Statistical analysis

Descriptive analysis will be performed to calculate the percentage of Alpha, Beta and Charlie ratings for each score of evaluation using SPSS software, version 17.0 (SPSS Inc. Headquarters, Chicago, Illinois, USA). Then, the data of the clinical evaluation will be descriptively analysed and compared at base line, 1 year and 2 years. According to Malament & Socransky (1999a), a restoration was considered to be a failure when ceramic fracture or partial debonding exposed the tooth structure and impaired esthetic quality or function, thus necessitating replacement of the crown.

# 8.3 Results

In total, 20 Turkom-Cera crowns (9 Premolars and 11 Molars) were evaluated from a minimum of 12 months to a maximum of 31 months in 16 patients. The detailed observation time for every location is presented in Table 8.1. At recall appointments all patients were presented for evaluation.

The frequency of evaluated restorations as a function of their observation time is presented in Figure 8.5. The mean time  $\pm$  SD in service for all restorations was 21.5  $\pm$  6.13 months (Table 8.5).



**Figure 8.5**: Distribution of the Turkom-Cera crowns done in relation to observation time in months.

Tooth	Total N	Mean Time	Range
Premolar	8	20.38	12-28
Molar	12	22.25	12-31
Overall	20	21.50	12-31

**Table 8.5**: The mean time (month) in service for all restorations

In Table 8.6, results of clinical evaluations using the modified USPHS criteria are compared at baseline, 1 year and 2 years. The ratings Alpha and Bravo were considered as successful, whereas Charlie rating was considered as failure. At the baseline and one year service times, 20 restorations distributed in 16 patients were examined. Whereas, only 9 restorations in 8 patients were evaluated after two years service time.

Measure	Baseline (n=20)		Year 1 (n=20)				Year 2 (n=9)				
	A	В	С	A	<u> </u>	B	С		A	В	С
Marginal Integrity	85	15	0	8	5	15	0	8	8.89	11.11	0
Shade	45	55	0	4	5 3	55	0	4	4.44	55.56	0
Surface Texture	100	0	0	8	5 1	5*	0	8	8.89	11.11*	0
Anatomic Form	90	10	0	9	0	10	0	8	8.89	11.11	0
Secondary Form	100	0	0	10	0	0	0	]	100	0	0
Wear	100	0	0	10	00	0	0	1	100	0	0
Cracks & fracture	100	0	0	8	5	15	0	1	100	0	0
Postoperative	100	0	0	10	00	0	0	-	100	0	0
Sensitivity											

**Table 8.6**:
 Scores of clinical evaluation (%) at baseline, year 1 and year 2

\*Alpha at Baseline.

A (Alpha); B (Bravo); C (Charlie).

At the baseline, all restorations received Alfa rating, except for the following that received Bravo ratings for: marginal integrity (15%); colour match (55%); and anatomic form (10%).

After 1 year, all 20 restorations were analyzed and all of them received Alfa rating, except for the following that received Bravo ratings for: marginal integrity (15%); colour match (55%); surface texture (15%); anatomic form (10%); and cracks & fracture (15%).

After 2 years, only 9 restorations were analyzed and all of them received Alfa rating, except for the following that received Bravo ratings for: marginal integrity (11.1%); colour match (55.6%); surface texture (11.1%); and anatomic form (11.1%).

In this study, changes occurred mainly in the score of surface texture. Comparing the score of surface texture at base line and first year, it was rated 100% Alpha at the baseline; however, it decreased to 85% Alpha at the end of the first year (Table 8.6). This is because the surface texture of 3 crowns was rated Bravo at first year due to chipping of the porcelain build-up.

Comparing the score of surface texture at base line and second year, it was rated 100% Alpha at the baseline; however, it decreased to 88.9% Alpha at the second year. This is because the surface texture of one crown was rated Bravo at second year due to chipping of the porcelain build-up.

However, no changes occurred between first year and second year score of surface texture parameter because at the second year only one crown rated Bravo, which was rated Bravo also at the first year. Although, the score for marginal integrity was rated 85% Alpha at the base line and remain the same at the first year, it was rated 88.9% Alpha at the second year (Table 8.6). However, the clinical evaluation showed no changes between the baseline, first year and second year score of marginal integrity since the 8 cases (88.9%) which were rated Alpha at second year were also rated Alpha at base line and at first year.

In this study, the score for shade compatibility was rated 45%, 45% and 44.44% Alpha at the base line, first year and second year, respectively (Table 8.6). However, the clinical evaluation showed no changes between the baseline, first year and second year score of shade compatibility since the 4 cases (44.44%) which were rated Alpha at second year were also rated Alpha at base line and at first year.

Although, the score for anatomic form was rated 90% Alpha at the base line and remain the same at the first year, it was rated 88.89% Alpha at the second year (Table 8.6). However, the clinical evaluation showed no changes between the baseline, first year and second year score of anatomic form since the 8 cases (88.89%) which were rated Alpha at second year were also rated Alpha at base line and at first year.

According to the results of clinical evaluation (Table 8.6), there were no changes between the baseline, first year and second year scores of secondary caries and wear of crown and opposing dentition parameters since all of them received Alpha rating at baseline, first year and second year.

The veneering porcelain chipped in 3 molar crowns in the marginal ridge, but did not compromise the integrity of the crowns since the contact areas were intact. In 2 crowns placed on mandibular right first molars, the veneering porcelain chipped 5 months and 9 months after cementation in a 27 and 37-year-old women, respectively. The third crown

was bonded to the preparation of mandibular left second molar, and the veneering porcelain chipped 11 months after cementation in a 55-year-old man.

During the whole observation period, 1 of the 20 Turkom-Cera crowns bonded to the preparation of maxillary right first molar of 29-year-old woman fractured after a service time of 14 months. A new all-ceramic crown was then reconstructed. Since the restoration required replacement, the crown was rated as a failure (Charlie).

Table 8.7 shows the frequency of postoperative sensitivity for the 16 restorations placed in vital teeth, all patients did not report any sensitivity during or after treatment. Table 8.8 shows the satisfaction of patients with their restoration. All patients in this study (100%) expressed satisfaction with their restorations.

**Table 8.7:** Postoperative sensitivity in 16 crowns placed on vital teeth

Restorations	Alpha	Bravo	Charlie
16	16	0	0
100%	100%		

**Table 8.8:** Results of patient satisfaction

Patients	Alpha	Bravo	Charlie
16 100%	16 100%	0	0

## 8.4 Discussion

This in vivo study evaluated the clinical performance of Turkom-Cera crowns cemented with self adhesive resin luting cement (Rely-X U100). The major problem with clinical follow-up studies is incomplete patient records. If all treated patients did not attend evaluations at ordered time sequences, the existence or absence of baseline records will determine whether a retrospective or prospective study can be performed (Segal, 2001; Gemalmaz and Ergin, 2002). Although these studies are complicated by the use of records obtained at different times, the population can be considered as a random sample if the mean and range of clinical evaluation times are provided (Sjögren et al., 1999; McLaren and White, 2000; Gemalmaz and Ergin, 2002). Table 8.1 shows the number of crowns in this study distributed over the evaluation period of 12 to 31 months.

In this study, the Turkom-Cera all-ceramic crowns were examined in accordance with the modified United States Public Health Service (USPHS) criteria, including 7 categories of evaluation. These criteria were originally developed by Cvar & Ryge in 1971 for clinical evaluation of dental restorative materials. The original categories of this evaluation system were color match, cavosurface marginal discoloration, anatomic form, marginal adaptation, and caries (Bayne and Schmalz, 2005; Cvar and Ryge, 2005). A slight modification has been made to these criteria by many authors (Lehner et al., 1997; Haselton et al., 2000; Otto, 2004; Taskonak, and Sertgoz, 2006 and others), adjusting them to their special needs. The original criteria were expanded to include other categories of interest including criteria for surface texture, postoperative sensitivity, proximal contact, occlusal contacts, fracture, and others (Bayne and Schmalz, 2005). When presented with a choice between an all-ceramic crown and a metal-ceramic crown, patients normally request material which has the greatest life expectancy. As a dentist, we will discuss the possible types of failure of an all-ceramic crown: recurrent dental caries, fracture of the ceramic material, loss of retention, poor aesthetics, defective margins and occlusal wear. However, these types of failures can occur with either an all-ceramic or a metal-ceramic crown. Besides, the failures may occur after a relatively short time or in a long clinical service time (Haselton et al. 2000).

There have been few studies on metal-ceramic crowns that describe the types of failure. Schwartz et al., (1970) reported that dental caries was the primary cause of failure (36.8%) for all restorations evaluated (cast gold crown, acrylic veneer crown, 3/4 crown). In a similar study conducted by Walton et al., (1986) dental caries also was the primary cause of failure (22%) of crowns observed (metal-ceramic, complete veneer metal, resin veneer metal, porcelain jacket, partial veneer). Another study by Coornaert et al., (1984) reported a failure rate of 2.38% which was mainly due to faulty design of coping and bruxism of the patients. However, failures due to caries, periodontal disease, or aesthetics were not indicated.

In the current study, one of the 20 Turkom-Cera crowns fractured. For this fractured crown, the patient indicated that the fracture of the crown occurred as a result of trying to break the shell of a hard nut. It appears that Turkom-Cera crowns are at slightly greater risk to loss by fracture than metal-ceramic crowns. However, the relatively low number of Turkom-Cera crowns (n=20) investigated in this study reduces the significance of this result. Furthermore, this additional risk may be relatively small in comparison to the risk of secondary caries or other hazards faced by all types of crowns.

Early types of full ceramic restorations showed a low to moderate failure rates for anterior full ceramic crowns. However, for posterior full ceramic crowns the failure rates increase dramatically. In a study that involved 679 aluminous ceramic crowns, McLean (1983) reported a 15.2% fracture rate for molars, 6.4% for premolars and 2.1% for incisors after 7 years. In a follow-up study of 101 glass ceramic crowns (Dicor), a failure rate of 16.3% was reported after 4 years, 22.8% for molars, 5.7% for premolars, and 0% for incisors/canines (Kelsey et al., 1995). Another study by Sjögren et al., (1999) reported a failure rate of 14% in a follow-up study of 98 Dicor crowns 74 months after luting, 30% for molars, 6% for premolars, and 12% for incisors/canines. This is showing the possible high risk of placing such restorations in the posterior area.

Clinical studies are necessary to assess both efficiency and success of new dental materials. The results of the current study cannot be directly compared with clinical trials of other all-ceramic systems since all of these trials were done under different conditions and utilize different research designs. However, the clinical evaluation results of other all-ceramic systems are reviewed.

Lehner et al., (1997) reported a survival rate of 95% in a follow-up study on 78 IPS Empress crowns after mean observation period of 19.7 months. This is comparable with results of two other clinical studies on IPS Empress crowns. Gemalmaz & Ergin, (2002) reported that 37 adhesively luted IPS Empress crowns exhibited a 94.6% survival rate after 24.56 months of clinical service. An in-vivo study by Toksavul & Toman, (2007) reported a survival rate of 95.24% for 79 IPS Empress 2 crowns after a mean follow-up period of 58 months. A clinical study by Scotti et al., (1995) reported a 98.4% survival rate for 63 In-Ceram Alumina crowns observed for a mean period of 37.6 months. One premolar crown had to be replaced because of fracture of the crown coping and veneering ceramic (Scotti et al., 1995).

In a clinical study of 80 In-Ceram Alumina crowns (58 anterior and 22 posterior) placed between 1994 and 1997, Haselton & colleagues (2000) reported that only one molar crown had fractured and the marginal ridge of one premolar crown had chipped. However, a clinical study conducted in a private practice reported that 223 In-Ceram Alumina crowns had a survival rate of 96% after three years, with higher survival rate for anterior crowns (98%) than posterior crowns (94%). Eight patients had single crown fractures, and three patients each had two crown fractures (McLaren and White, 2000). Another clinical study on the clinical performance of 24 In-Ceram Alumina crowns (two premolars and 22 molars) reported a survival rate of 92% after a mean clinical service time of 39 months. Two molar In-Ceram Alumina crowns fractured after respective service times of 14 and 17 months in the same patient (Bindl and Mörmann, 2002).

Odén et al., (1998) evaluated the clinical performance of 100 Procera AllCeram crowns for 5 years and reported 94% survival rate. Another prospective study evaluated 87 Procera AllCeram crowns within 5 to 10<sup>1</sup>/<sub>2</sub> years period reported a survival rate of 97.7% and 93.5%, respectively (Ödman and Andersson, 2001). The authors concluded that a good prognosis for Procera AllCeram crowns is also achievable in posterior teeth. This is in agreement with the results of other studies on Procera AllCeram crowns. Fradeani et al., (2005) showed a survival rate of 96.7% for 205 Procera crowns after 5 years, with 95.15% survival in the posterior area. Walter et al., (2006) reported a survival rate of 94.3% for 107 Procera crowns after 6 years of clinical service. Furthermore, a recent prospective clinical study of 103 Procera AllCeram crowns placed on posterior teeth reported a cumulative survival rate of 98.8% after five years. In addition to the one mechanical failure (fracture) reported, biologic complications were observed in 8 crowns, and root caries lesions were detected in 4 molar teeth (Zitzmann et al., 2007).

The failures reported in all studies were caused by core fractures, fractures of the veneering material (chipping), secondary caries, tooth or root fractures and loss of retention. However, it should be pointed out that secondary caries is a host response likely unrelated to the particular materials used in fixed prostheses (Della Bona and Kelly, 2008). Furthermore, when evaluating data from clinical studies, it must be taken into account that in most publications, only those restorations that had to be removed were considered failures (Wassermann et al., 2006).

In this study, only one molar crown has fractured after a service time of 14 months with a small fracture involving the veneering porcelain palatally and alumina coping at the palatal cervical margin only. The fractured part was small and had debonded from the crown and fallen off, and the crown had remained in place (Figure 8.6). This fracture mode appears favourable in comparison with the failures observed in previous allceramic systems, which normally show a cross-section breakage (Bindle et al., 2002; Fradeani et al., 2005; Walter et al., 2006).



Figure 8.6: The fractured crown on upper right first molar.

According to the results of this study, clinical changes occurred mainly in the score of surface texture. This is due to the chipping of veneering porcelain in 3 molar crowns in the marginal ridge during the first year. These three crowns were smoothed, finished, and not considered as failures because the frameworks were not fractured, the prepared teeth surfaces were not exposed and the crowns are still in function. The score of surface texture was rated 100% Alpha at base line and decreased to 85% Alpha at the end of the first year. In addition, the score of surface texture was rated 100% Alpha at the second year. However, no changes occurred between first year and second year score of surface texture parameter because at the second year only one crown rated Bravo, which was rated Bravo also at the first year.

A clinical change between the base line, first year and second year scores of marginal integrity, shade compatibility, anatomic form, secondary caries and wear of crown and opposing dentition parameters were not observed, and all have shown a satisfactory result.

In the current study, no crown was replaced because of secondary caries. This may be due to patient's oral hygiene and dietary habits, the dentist's patient selection and periodic recall sessions and the fit and performance of the crowns themselves. Additionally in this study, no failure was reported because of marginal deficiency. All crowns were rated satisfactory regarding the score of marginal integrity with 88.9% rated Alpha and 11.1% Bravo at the end of the study.

Even though Bravo ratings for marginal integrity and shade allowed for some compromise (Table 8.6), both Alpha and Bravo ratings are considered successful results. The Bravo ratings for marginal integrity and colour match in this study, although high, are based on previously published modified US Public Health Service criteria (Fradeani et al., 1997; Haselton et al., 2000).

The luting procedure is an important parameter that can influence the final resistance of all-ceramic restorations. According to several in vitro studies, it is recommended to use an adhesive cementation whenever possible, especially in posterior areas to enhance fracture resistance (Burke, 1995; Groten and Pröbster, 1997; Fradeani et al., 2005; AL-Makramani et al., 2008a; AL-Makramani et al., 2008b).

Several studies investigated bond strength of self adhesive resin cement (RelyX Unicem) to different types of ceramic: high-strength aluminium oxide, leucite-reinforced, lithium disilicate, machinable feldspathic and zirconia ceramics (Piwowarczyk et al., 2004; Kumbuloglu et al., 2005; Piwowarczyk et al., 2005; Reich et al., 2005). The reported bond strength values varied, depending on the ceramic treatment and the aging conditions. However, the achieved results are in agreement,

demonstrating that this cement obtained bond strength that is either higher or comparable to other investigated materials. It was also found that water- and thermostable bonds can be established between sandblasted high-strength aluminium oxide ceramic and the self adhesive resin cement (Piwowarczyk et al., 2004). Therefore, self adhesive resin cement RelyX U100 was used as a luting material instead of conventional phosphate cement in the present study.

# 8.5 Limitations

- Only Turkom-Cera crowns were evaluated to complement the physical and mechanical properties tested and were not meant to be compared to other types of all-ceramic crowns.
- 2. In this study, all clinical steps from preparation to luting of the crown were performed by the same clinician, so selection biases may have been introduced.
- 3. Even though, crowns were followed over a period of 31 months, they were not placed at the same time.
- 4. The shear bond strength of Panavia F resin cement to Turkom-Cera was investigated (Chapter 4) and showed very high value. However, another cement (RelyX U100 self adhesive resin cement) was used for luting Turkom-Cera crowns.
- 5. In this study the agreement between examiners was only 64 %. However, this percentage of agreement (64 %) could be considered low and it would be better to have at least 80 % agreement in future studies.
- 6. Other, more significant limitations of the study include the short follow-up time, the small sample and large number of variables for assessing the quality of crowns. Although some noteworthy conclusions can be drawn from the result of this study, they should be carefully interpreted due to the experimental design.

## 8.6 Conclusions

The clinical behaviour of Turkom-Cera crowns were evaluated in a prospective study over a two year period. In spite of the limitations of this study, the following conclusions were drawn:

- 1. Turkom-Cera crowns showed a low clinical failure rate. No breakage was observed on the premolars, but rather only one fracture was seen in 1 molar crown 14 months after insertion. The mode of fracture observed in this study appears favourable in comparison with the failure modes observed in previous all-ceramic systems, which normally show a cross-section breakage.
- 2. The initial clinical results of this aesthetic restorative material are encouraging. However, because of fatigue phenomena for all-ceramic materials, further studies with a longer observation period are necessary to provide a definitive prognosis regarding its long-term clinical behaviour.

CHAPTER NINE

SUMMARY AND RECOMMENDATIONS

## 9.1 Summary

All-ceramic crowns were routinely placed not only in the anterior aesthetic zone but also in the posterior where they were subjected to greater occlusal forces and stress from cyclic loading (Snyder and Hogg, 2005). Before starting time-consuming and costly clinical investigations, preclinical in vitro studies should be carried out to evaluate the clinically relevant properties for newly developed dental materials and products (Gu and Kern, 2003).

The strengths of brittle materials are usually measured in flexure (bending) because this test is generally easier to conduct than a pure tensile test. In uni-axial flexural strength tests, the principal stress on the lower surfaces of the specimens is tensile, and it is usually responsible for crack initiation in brittle materials. However, undesirable edge fracture (which can increase the variance of the failure stress value) can occur. Furthermore, these methods were designed for engineering materials that are usually associated with relatively large specimens.

The biaxial flexural strength test has been used frequently for the evaluation of fracture characteristics of brittle materials (Wagner and Chu, 1996; Albakry et al., 2004; Pagniano et al., 2005). The measurement of the strength of brittle materials under biaxial flexural strength conditions rather than uni-axial flexural strength is often considered more reliable because the maximum tensile stresses occur within the central loading area and spurious edge failures are eliminated. Besides, the bi-axial flexural strength test is recommended by ISO [6872, 1995 (E)] since the test standardizes specimen thickness, diameter, shape and roughness.

The biaxial flexural strength and hardness of the Turkom-Cera compared to two other all-ceramic systems (In-Ceram and Vitadur-N) were investigated in this study. Ten disc specimens for flexural strength and 5 disc specimens for hardness testing were made for each system. The flexural strength test was done in accordance with ISO 6872-1995 using the Instron universal testing machine. The hardness was determined using the Shimadzu Vickers microhardness indenter. According to the results of this study, Turkom-Cera has significantly higher flexural strength (506.8 MPa) than In-Ceram (347.4 MPa) and Vitadur-N (128.7 MPa) ceramic materials. However, In-Ceram core has significantly higher hardness (1116.2 VHN) than Turkom-Cera (1002.1 VHN) and Vitadur-N (812.8 VHN) ceramic materials

Adhesive composite resin cements are recommended for cementation of all-ceramic systems. However, it has been shown that full coverage densely sintered alumina crowns can achieve long-term clinical success with conventional luting agents (Oden et al., 1998; Odman and Andersson, 2001). Furthermore, the Turkom-Cera manufacturer has recommended that conventional luting cements such as zinc phosphate and glass ionomer cements be used for cementation of Turkom-Cera crowns.

A durable and predicable bond between resin luting cements and ceramic is usually created by two mechanisms: micromechanical attachment to porosities originated from hydrofluoric acid etching and/or gritblasting and chemical bonding by a silane-coupling agent (Filho et al., 2004). The common surface treatments are acid etching, airborne particle abrasion, silane-coupling agent, and combinations of these methods (Awliya et al., 1998; Özcana and Vallittu, 2003; Zhang et al., 2004; Nagayassu et al., 2006).

Turkom-Cera<sup>TM</sup> is a new all-ceramic material and no information is available on the strength of the bond between different cements and this new all-ceramic material. Therefore, a laboratory study with the purpose to find the optimal choice of luting cement and surface treatment for bonding Turkom-Cera restorations was conducted.

This study shows the results of the evaluation of shear bond strength of four different luting cements (Elite, Fuji I, Fuji Plus and Panavia F) to the Turkom-Cera material. The effect of surface treatments: no treatment (control), sandblasting, silane application and combinations of these treatments on the bond strength of resin cement to Turkom-Cera were also investigated. According to the luting cements and surface treatments used, seven different groups were evaluated.

Seventy Turkom-Cera ceramic discs with 10 mm diameter and 3 mm thickness were prepared (10 for each group). The ceramic discs were wet ground to ensure flat parallel surfaces. The luting cements were bonded, as per manufacturer instructions to Turkom-Cera discs using a bonding jig recommended by ISO TS 11405/2003. Then, the bonded specimens were stored in distilled water for 24 hours at 37°C. Shear bond strengths were determined using the universal testing machine (Instron) at 0.5 mm/min crosshead speed.

The results of this study indicated that selection of the appropriate luting cement is a key factor for achieving a strong bond to Turkom-Cera all-ceramic material. The phosphate-containing resin cement Panavia-F exhibited shear bond strength value significantly higher than all other cements tested. Within the results of this study, it was found that when using Panavia F resin cement and Clearfil silane; sandblasted Turkom-Cera specimens produced the highest mean shear bond strength values. Almost similar shear bond strength values were obtained for Turkom-Cera specimens when sandblasted

with  $50-\mu m$  Al<sub>2</sub>O<sub>3</sub> or silanated with clearfil silane. Therefore, these three surface treatments appeared to be the methods of choice for the cementation of Turkom-Cera restorations.

Strength is an important mechanical property that controls the clinical success of dental restorations. Usually, complex stress distributions that are induced by compressive, tensile, and shear stresses are present in most specimens under practical conditions. In general, tensile strength is easily determined for ductile materials such as metals. For convenience, compressive strength is often measured for brittle materials such as porcelains, cements, amalgams, and resin composites (Ban and Anusavice, 1990).

The occlusal fracture resistance of Turkom-Cera all-ceramic copings compared to Procera AllCeram and In-Ceram all-ceramic copings cemented with resin luting cement Panavia F was evaluated using metal dies as a supporting structure. The effect of different luting agents (Elite, Fuji I and Panavia F) on the occlusal fracture resistance of Turkom-Cera was also evaluated.

Ten ceramic copings of 0.6 mm thickness were fabricated for each group of ceramics. The copings were cemented to standardized metal dies according to manufacturer's instructions. After 24 hours of distilled water storage at 37°C, the copings were vertically compressed using Instron testing machine at a crosshead speed of 1 mm/min.

According to the results of this study, the mean load at fracture of Turkom-Cera (2184 N) was significantly more than Procera AllCeram (1953.5 N) (p<0.05). There was no significant difference between the mean load at fracture of In-Ceram (2041.7 N) and Procera AllCeram and also between Turkom-Cera and In-Ceram (p>0.05). There were

significant differences in the load at fracture between the three luting cements used (p<0.05). The mean load at fracture of Turkom-Cera copings cemented with Elite, Fuji I and Panavia F were 1537.4 N, 1294.4 N, and 2183.6 N, respectively. It was concluded that luting agents have an influence on the occlusal fracture resistance of Turkom-Cera copings.

Abutments made of metal do not reproduce the actual force distribution that may occur on crowns cemented to natural teeth. The fracture load of ceramic may be greater if crowns are supported by dies with a high modulus of elasticity (Scherrer & de Rijk, 1993). The advantages of using metal dies in fracture strength testing are the possibility of a standardized preparation and the identical physical properties of materials (Komine et al., 2004). However, using natural teeth or materials with a comparable modulus of elasticity are preferred for in vitro fracture strength tests (Rosentritt et al., 2000). Therefore, an in vitro study to investigate the occlusal fracture resistance of Turkom-Cera copings compared to Procera AllCeram and In-Ceram all-ceramic copings cemented to natural teeth was conducted.

Ten ceramic copings of 0.6 mm thickness were fabricated for each group of ceramics. The copings were cemented to their corresponding teeth with resin luting cement Panavia F according to manufacturer's instructions. After 24 hours of distilled water storage at 37 °C, the copings were vertically compressed using Shimadzu testing machine at a crosshead speed of 1mm/minute. The effect of marginal design (chamfer or shoulder) and artificial ageing (30-day water storage and 500 thermocycles) on the occlusal fracture resistance of Turkom-Cera copings were also investigated in this study.

Turkom-Cera showed significantly higher load at fracture (1341.9 N) than Procera AllCeram (975.0 N). No significant difference was detected between the load at fracture of In-Ceram (1151.6 N) and Procera AllCeram and also between Turkom-Cera and In-Ceram. In this study, there was no influence of the finish line design and artificial ageing on the load at fracture of Turkom-Cera all-ceramic copings (p>0.05). Furthermore, all of the investigated all-ceramic materials had higher mean value of load at fracture than the maximum biting forces in the molar and premolar regions, which justifies their application in those areas.

Marginal fit is one of the important criteria used in the clinical evaluation of fixed restorations. The presence of marginal discrepancies in the restoration exposes the luting agent to the oral environment. The larger the marginal discrepancy and subsequent exposure of the luting agent to oral fluids conditions, the more rapid is the rate of cement dissolution (Sulaiman et al., 1997; Jacobs and Windeler, 1991). According to McLean and von Fraunhofer (1971), a marginal opening of 120  $\mu$ m represents the maximum clinically acceptable gap size.

The marginal adaptation of Turkom-Cera In-Ceram and Procera AllCeram copings were investigated. The influence of the finish line design (chamfer or shoulder) on the marginal adaptation of Turkom-Cera all-ceramic copings was also evaluated. Ten ceramic copings of 0.6 mm thickness were fabricated for each group following manufacturer's instructions. The copings were seated on the abutments using a special holding device that facilitated uniform loading, and the marginal adaptation was assessed with a stereomicroscope. The mean marginal adaptation for Turkom-Cera, In-Ceram and Procera AllCeram were 49.19  $\mu$ m, 71.51  $\mu$ m and 34.38  $\mu$ m, respectively. It was verified that there were statistically significant differences among the marginal adaptation of the three all-ceramic systems (p<0.05). There was no significant difference in the mean marginal discrepancy of Turkom-Cera crowns among the chamfer and shoulder groups (p>0.05). Furthermore, it was concluded that the marginal discrepancies of all ceramic systems investigated in this study were all within the clinically acceptable standard.

It has been stated that the specimens used for in vitro testing of dental ceramics sometimes differ significantly in both size and structure from the restorations they represent (Kelly, 1995). Certain intraoral factors like stress corrosion and subcritical crack growth have significant effect on long-term survival of dental ceramics (Rekow and Thompson, 2001; Zhang and Lawn, 2005; Toksavul and Toman, 2007). Therefore, a preliminary prospective study to evaluate the durability of Turkom-Cera all-ceramic crowns through a two-year clinical trial was carried out.

In this study, twenty Turkom-Cera crowns (9 Premolars and 11 Molars) were placed in 16 patients at the Faculty of Dentistry, University of Malaya. They were evaluated from a minimum of 12 to a maximum of 31 months (mean, 21.5 months) after insertion. The Turkom-Cera crowns were evaluated by two examiners using the United States Public Health Service (USPHS) criteria. The crowns were evaluated for the following: (1) marginal integrity, (2) shade compatibility, (3) surface texture, (4) anatomic form, (5) secondary caries, (6) wear of crown and opposing dentition, and (7) cracks and fractures of the crowns. The modified USPHS criteria were used to assign a rating of Alpha, Bravo, or Charlie to each of the 7 categories of evaluation at baseline and subsequent recall appointments. In addition, subjects were asked about their experience with possible postoperative sensitivity and their general satisfaction with the restorations. During the whole observation period, 1 of the 20 Turkom-Cera crowns was found to have fractured after a service time of 14 months. The veneering porcelain chipped in 3 molar crowns, but did not compromise the integrity of the crowns. The other parameters were rated satisfactory according to the United States Public Health Service (USPHS) criteria. All patients expressed satisfaction with their restorations and did not report any sensitivity during or after treatment.

Turkom-Cera crowns demonstrated acceptable clinical behaviour and equal to or higher strength than currently accepted alumina-based all-ceramic materials. Therefore, it can be used for fabrication of all ceramic crowns both anteriorly and posteriorly.

# 9.2 Recommendations for further studies

The clinical behaviour of Turkom-Cera crowns were evaluated in a prospective study from a minimum of 12 months to a maximum of 31 months, with a mean of 21.5 months. However, because of fatigue phenomena for all-ceramic materials, further studies with a longer observation period are necessary to provide a definitive prognosis regarding long-term clinical behaviour of Turkom-Cera crowns.

In this study, only Turkom-Cera crowns were evaluated and the success rates of other types of restorations were not included. Further studies are required to evaluate the long term clinical behaviour of Turkom-Cera inlays, onlays and bridge restorations of probably more than 5 years.

The Turkom-Cera copings demonstrated equal to or higher fracture resistance than currently accepted all-ceramic materials. However, the specimens were loaded until failure in a single cycle, even though restorations may fail clinically through slow crack growth caused by cyclic fatigue loading. Subjecting the specimens to cycling fatigue loading could be considered in further investigation to give more information about the longevity and performance of Turkom-Cera crowns in conditions relatively resembling the clinical situation.

This study has evaluated the fracture resistance of only Turkom-Cera copings. It is recommended to conduct further studies on fracture resistance of Turkom-Cera all-ceramic crowns and bridges and compare it with other all-ceramic systems used in fabrication of all ceramic crowns and bridges.

In the present study, shear bond test was used to evaluate the bond strength of luting cements to Turkom-Cera discs. However, it can be questioned whether a tension test might be more appropriate for testing the bond strength of Turkom-Cera crowns and bridges using different luting cements. Further study to evaluate the tensile bond strength of Turkom-Cera crowns and bridges to natural teeth is needed.

This study has evaluated the bond strength of luting cements to Turkom-Cera. Another important issue is the bond strength of porcelain veneer to Turkom-Cera core. Combining the strength of ceramic cores and the aesthetics of veneering porcelains allows dental technicians to build an aesthetic restoration with an individual character. Bearing in mind that the bond strength of porcelain veneer has been considered a weak link in the layered all-ceramic restorations, future study aimed to evaluating the bond strength of different brands of porcelain veneers to Turkom-Cera core is recommended. The present study used natural teeth to measure the marginal discrepancy of Turkom-Cera crowns. However, the crowns were not cemented using luting cements. If the crowns were cemented, the marginal discrepancies might have been increased. In addition, the marginal discrepancy was measured, but the internal fit of the crowns was impossible to measure in this experimental design. Measuring the internal fit of artificial crowns requires cementation and sectioning of the specimens. Therefore, further studies are required to develop new experimental designs to measure both the marginal and internal fit of Turkom-Cera crowns and bridges. References
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APPENDICES
## APPENDIX I Turkom-Cera<sup>™</sup> system

### Material and system description

The equipment used for the Turkom-Cera all-ceramic system (Figure I.1) in a dental laboratory does not include more than standard laboratory furnace, propane gas flame and a standard laboratory micromotor.



Figure I.1: A Turkom-Cera Fused Alumina kit.

The Turkom-Cera Fused Alumina kit includes the following:

- Ø Ordinary silicon in a small glass jar
- Ø Metal holder for plastic foil
- Ø Isolation varnish
- Ø A plastic spacer foil of 0.1mm thickness (red)
- Ø A plastic spacer foil of 0.6mm thickness (transparent)
- Ø Turkom-Cera Solution
- Ø Turkom-Cera Alumina Gel in jar
- Ø Turkom-Cera Powder for crystal hardening (available in different shades)

- Ø High Performance Platinum Pins
- Ø Turkom-Cera Testing Liquid
- Ø Alumina blank

#### Fabrication of all-ceramic restorations using Turkom-Cera system

Fabricating restorations with Turkom-Cera system incorporates the following main steps:

1. After receiving the impression, a stone die must be prepared with high precision,

clearly indicating the shapes and margins (Figure I.2).



Figure I.2: Preparation of the stone die.

2. Place the red plastic foil (0.1 mm) atop the transparent plastic foil (0.6 mm), and then clamp both foils in the metal holder. Heat foils with a propane gas flame carefully until they are deformable (avoid overheating). When the plastic foils gain enough heat and start to deform, immerse the stone die deep in the silicon putty by bushing the die smoothly through the plastic foils. Hold on for 5-10 seconds to allow the foils to retain their rigidity. The foil should take the exact form of the stone model (Figure I.3A & B).



**Figure I.3A & B:** Holding and heating the foils (A), and dipping of the die in the silicone putty (B).

3. Pull out the die/foils from the silicon putty, and slowly release the die from the plastic foils. Start cutting out the double layer plastic coat using scissors paying special attention that the cut is done exactly along the upper line of the margin. Separate the transparent and red foils, removing off the transparent layer. Place the red plastic foil again on the stone die (Figure I.4A,B & C).



**Figure I.4A,B & C:** Removing the die (A), cutting the foils (B), and placing the red foil on the stone die (C).

4. Heat the remaining piece of the transparent foil, which was removed, and adapt it over the red foil along the margins and corners areas to ensure that the red plastic foil is tightly adjusted and aligned for perfect fitting (Figure I.5A & B).



Figure I.5A & B: Adapting the red foil on the die using the heated transparent foil.

N.B. (If the Turkom-Cera coping is to be used for a crown, one layer of the red foil will be sufficient to proceed to the next step. However, if coping is to be used for a bridge, repeat steps 2 through 4 to add a second layer of the red foil. The inner layer of the red foil should be 1.0 mm above the margin line, while the outer one should be accurate and aligned with the margin as described) (Figure I.6).



Figure I.6: The diagram showing the adaptation of a single or double red plastic foil.

5. Remove the plastic foil from the stone die. Using a brush, apply a layer of transparent varnish to isolate the surface of the stone about 10.0 mm lower from the margin line downwards. This is to prevent adhering of the alumina gel to the stone die. Let the varnish to dry for 10-15 seconds. Replace the red foil properly on the stone die and adapt it again. Use the separating oil to paint the stone die around the margin line downwards making sure that there is no oil on the plastic foil (Figure I.7A & B).



Figure I.7A & B: Isolating the stone die with varnish (A) and separating oil (B).

6. Stir the Turkom-Cera Alumina Gel gently. If necessary, add few drops of Turkom-Cera Solution to obtain the desired consistency. Using a nylon brush of appropriate size and shape, apply Turkom-Cera Alumina Gel carefully on the corner area of the plastic foil to avoid the formation of air bubbles at the margin. Then, apply Turkom-Cera Alumina Gel on the die fitted with red plastic foil by dipping into the jar containing Turkom-Cera Alumina Gel. The whole of the spacer foil and the adjacent area of the stone die should be immersed into the gel. Pull out the die gently, and hold it above the jar to get rid of the excess gel. Tap on the stone die with your index finger until the last drop of alumina gel is separated (Figure I.8A & B).





**Figure I.8A & B:** Applying the alumina gel to the margin area (A), and dipping the die in the gel (B).

7. Put the die aside and wait for 5-6 minutes until the Turkom-Cera Alumina Gel dries out. Then proceed to the next step to form a second layer of the alumina gel. Repeat the step once to apply a second layer of the Turkom-Cera Alumina Gel. Using a sharp blade mounted on a scalpel handle, remove the excess material from the die precisely below the margin. Carefully remove the coping from the stone model. For single crown Turkom-Cera Alumina Gel is applied in two layers (i.e. two dippings). However, for bridge work Turkom-Cera Alumina Gel is applied in three layers (i.e. three dippings) (Figure I.9A,B & C).



**Figure I.9A,B & C:** The die after applying alumina gel (A), removing the excess alumina (B), and removing the coping from the stone die (C).

8. Sintering is accomplished using a standard furnace. Place the coping on the firing tray. Then place the firing tray on the firing platform of the furnace and sintered for 5 minutes (1150°C). After sintering, finish the margin using silicon rubber. The contours of the sintered coping can be adjusted using a coarse-grain diamond bur at rotational speed of 2000-3000 rpm. The thickness of the coping can be also adjusted if necessary. It can be refined to as thin as 0.3mm (Figure I.10A,B & C).



Figure I.10A,B & C: Sintering (A), finishing (B), and adjusting the coping (C).

- 9. Before crystal hardening, check the accuracy of the sintered coping. Place it on the stone die to observe the need for possible adjustments. When this has been done, use Turkom-Cera Testing Liquid to check for any possible crack-lines. If there are no cracks detected, the Turkom-Cera coping is ready for crystal hardening.
- 10. Crystal hardening (Figure I.11A & B) is achieved using Turkom-Cera Crystal Powder. Mix Turkom-Cera Crystal Powder with distilled water to obtain a thick paste-like consistency. Apply 1 or 2 coats of the mixed powder (1-2mm thick) to the outer surface of the alumina coping by using a brush. Press on the coating with a dry tissue paper occasionally to ensure that there are no blank areas and

that the coating is well formed and structured. The margins must not be covered. Use the High Performance Platinum Pins to place the coping on the firing tray to perform crystal hardening. Then place the firing tray on the firing platform of the furnace. For crystal hardening, program the oven to run for 30 minutes at 1150°C for crowns and 40-50 minutes at 1180°C for bridge works.



Figure I.11A & B: Applying the crystal powder to the sintered alumina coping.

11. When crystal hardening is finished, remove the coping from the firing tray and wait until the coping cools down. Check carefully if all parts of the coping are properly hardened. If some areas are deficient of crystal hardening (usually appears as white spots/stains and may vary in size), the deficient areas should again be covered with a second layer of crystal and fired again. Once all of the areas have been properly hardened, crystal hardening is completed. Use coarse-grained diamond bur to remove excess crystal from the surface of the coping. After removing the excess crystal from the surface, the coping is ready to proceed with porcelain build-up. No opaque is needed with Turkom-Cera coping (Figure I.12A,B & C).



Figure 1.12A,B & C: Two views of the crystal hardened coping (A & B), and removing the excess crystal (C).

12. Prior to the starting of the build-up process, fire the coping for about 60 seconds at a temperature of 930°C to remove any traces of biological contacts (i.e. from the hands of the technician). All brands of alumina ceramic porcelain powder (with coefficient of thermal expansion of 6.5 to 7.2) existing in the market are compatible with Turkom-Cera fused alumina framework and can be used for the porcelain build-up.

## **APPENDIX II** STATISTICAL ANALYSIS TABLES FOR CHAPTER 3



Figure II.1: Histogram of biaxial flexural strength (MPa)

Table II.1:	Normality	test for	biaxial	flexural	strength
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	Kolmogo	-Smirnov <sup>a</sup>	Shapiro-Wilk			
	Statistic	Sig.	Statistic	df	Sig.	
<b>Biaxial Flexural Strength</b>	0.190	30	0.007	0.910	30	0.015

a. Lilliefors Significance Correction

**Table II.2:**Comparison of biaxial flexural strength (MPa) between Turkom-Cera and<br/>In-Ceram using Mann-Whitney Test with Bonferroni correction

Test	Statistics <sup>b</sup>
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	Biaxial Flexural Strength
Mann-Whitney U	2.000
Wilcoxon W	57.000
Z	-3.628
Asymp. Sig. (2-tailed)	0.000
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>

a. Not corrected for ties.

b. Grouping Variable: Ceramic

# **Table II.3:**Comparison of biaxial flexural strength (MPa) between Turkom-Cera and<br/>In-Ceram using Mann-Whitney Test with Bonferroni correction

#### Test Statistics<sup>b</sup>

	Biaxial Flexural Strength		
Mann-Whitney U	0.000		
Wilcoxon W	55.000		
Z	-3.780		
Asymp. Sig. (2-tailed)	0.000		
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>		

a. Not corrected for ties.

b. Grouping Variable: Ceramic

Table II.4:	Comparison of biaxial flexural strength (MPa) between In-Ceram and
	Vitadur N using Mann-Whitney Test with Bonferroni correction

Test Statistics <sup>b</sup>				
	Biaxial Flexural Strength			
Mann-Whitney U	0.000			
Wilcoxon W	55.000			
Z	-3.780			
Asymp. Sig. (2-tailed)	0.000			
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>			

a. Not corrected for ties.

b. Grouping Variable: Ceramic



Figure II.2: Histogram of Vickers microhardness (VHN)

### Table II.5: Normality test for Vickers hardness

	Kolmo	Shapiro-Wilk				
	Statistic	df	Sig.	Statistic	df	Sig.
Vickers hardness	0.126	15	$0.200^{*}$	0.937	15	0.343

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

Table II.6:	Test of Homogeneity of	Variances for	Vickers micrrohardness
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Levene Statistic	df1	df2	Sig.
1.389	2	12	0.287

# Table II.7: Multiple Comparisons of Vickers microhardness (VHN) mean values using Tukey HSD Test

					95% Confidence Interval		
		Mean Diff.	Std.		Lower	Upper	
(I) Ceramic	(J) Ceramic	( <b>I-J</b> )	Error	Sig.	Bound	Bound	
Tukom-Cera	In -Ceram	-114.1(*)	36.5	.022	-211.6	-16.6	
	Vitadur N	189.3(*)	36.5	.001	91.8	286.8	
In -Ceram	Turkom-Cera	114.1(*)	36.5	.022	16.6	211.6	
	Vitadur N	303.4(*)	36.5	.000	205.9	401.0	
Vitadur-N	Turkom-Cera	-189.3(*)	36.5	.001	-286.8	-91.8	
	In-Ceram	-303.4(*)	36.5	.000	-400.9	-205.9	

\* The mean difference is significant at the .05 level.

## **APPENDIX III** STATISTICAL ANALYSIS TABLES FOR CHAPTER 4



Figure III.1: Histogram of shear bond strength (MPa) (Effect of luting cements)

Table II.1: Normality test	for shear	bond strength	(effect of luting	cements)
	Те	ests of Normality		

	Kolmogorov-Smirnov <sup>a</sup>		Shapiro-Wilk			
	Statistic	df	Sig.	Statistic	df	Sig.
Shear bond Strength	0.289	40	0.000	0.771	40	0.000

a. Lilliefors Significance Correction

**Table III.2:**Comparison of shear bond strength (MPa) between Elite and Fuji I using<br/>Mann-Whitney U Test with Bonferroni correction

	Shear bond Strength	
Mann-Whitney U	11.000	
Wilcoxon W	66.000	
Z	-2.948	
Asymp. Sig. (2-tailed)	0.018	
Exact Sig. [2*(1-tailed Sig.)]	0.002 <sup>a</sup>	

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

# **Table III.3:**Comparison of shear bond strength (MPa) between Elite and Fuji Plus<br/>using Mann-Whitney U Test with Bonferroni correction

lest Statistics			
	Shear bond Strength		
Mann-Whitney U	0.000		
Wilcoxon W	55.000		
Z	-3.781		
Asymp. Sig. (2-tailed)	0.000		
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>		

Test Statistics<sup>b</sup>

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

# **Table III.4:** Comparison of shear bond strength (MPa) between Elite and Panavia F using Mann-Whitney U Test with Bonferroni correction

Test Statistics <sup>b</sup>				
	Shear bond Strength			
Mann-Whitney U	0.000			
Wilcoxon W	55.000			
Z	-3.780			
Asymp. Sig. (2-tailed)	0.000			
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>			

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

**Table III.5:**Comparison of shear bond strength (MPa) between Fuji I and Fuji Plus<br/>using Mann-Whitney U Test with Bonferroni correction

	Shear bond Strength	
Mann-Whitney U	2.000	
Wilcoxon W	57.000	
Z	-3.630	
Asymp. Sig. (2-tailed)	0.000	
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>	

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

# **Table III.6:**Comparison of shear bond strength (MPa) between Fuji I and Panavia F<br/>using Mann-Whitney U Test with Bonferroni correction

Test Statistics			
	Shear bond Strength		
Mann-Whitney U	0.000		
Wilcoxon W	55.000		
Z	-3.780		
Asymp. Sig. (2-tailed)	0.000		
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>		

Test Statistics<sup>b</sup>

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

# **Table III.7:** Comparison of shear bond strength (MPa) between Fuji Plus and PanaviaF using the non-parametric Mann-Whitney U Test

lest Statistics			
	Shear bond Strength		
Mann-Whitney U	0.000		
Wilcoxon W	55.000		
Z	-3.781		
Asymp. Sig. (2-tailed)	0.000		
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>		

### Fest Statistics<sup>b</sup>

a. Not corrected for ties.

b. Grouping Variable: Luting Cement

# **Table III.8:** Chi-square test between treatment groups ((Elite, Fuji I, Fuji Plus and<br/>Panavia F) and modes of failure

	Value	df	p-value
Pearson Chi-Square	9.730	3	0.021

4 (50.0%) cells have expected count less than 5. The minimum expected count is .75.



Figure III.2: Histogram of shear bond strength (MPa) (Effect of surface treatments)

Table III.9: Normality	test for shear bond	l strength (effect o	of surface treatments)
2		U V	

Tests o	f Nori	nality
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	Kolmogorov-Smirnov <sup>a</sup>		Shapiro-Wilk			
	Statistic	df	Sig.	Statistic	df	Sig.
Shear bond Strength	0.094	40	0.200 <sup>*</sup>	0.975	40	0.501

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

Levene's Statistic	df1	df2	Sig.
2.154	3	36	0.110

					95% Cor	nfidence
					Inte	erval
		Mean Difference	Std.		Lower	Upper
(I) Treatment	(J) Treatment	(I-J)	Error	Sig.	Bound	Bound
Control	Sand blasting	-5.59(*)	1.14	0.00	-8.65	-2.53
	Silane	-5.35(*)	1.14	0.00	-8.41	-2.29
	Sand blasting + S lane	-8.29(*)	1.14	0.00	-11.36	-5.23
Sand blasting	Control	5.59(*)	1.14	0.00	2.53	8.65
	Silane	0.24	1.14	0.10	-2.82	3.31
	Sand blasting + S lane	-2.70	1.14	0.10	-5.76	0.36
Silane	Control	5.35(*)	1.14	0.00	2.29	8.41
	Sand blasting	-0.24	1.14	0.10	-3.31	2.82
	Sand blasting + S lane	-2.94	1.14	0.06	-6.01	0.12
Sand blasting	Control	8.29(*)	1.14	0.00	5.23	11.36
+ Silane	Sand blasting	2.70	1.14	0.10	-0.36	5.76
	Silane	2.94	1.14	0.06	-0.12	6.01

 Table III.11:
 Multiple comparisons between the 4 treatment groups using Tukey HSD

\* The mean difference is significant at the .05 level.

Table III.12:	Chi-square tes	t between treatment	group and	mode of fracture
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	Value	df	p-value
Pearson Chi-Square	6.395	3	0.09

4 (50.0%) cells have expected count less than 5. The minimum expected count is 2.75.

## **APPENDIX IV** STATISTICAL ANALYSIS TABLES FOR CHAPTER 5



Figure IV.1: Histogram of the load at fracture (N) (Effect of ceramic materials).

Table IV.1:	Shapiro-Wilk test for the load at fracture (Effect of ceramic materials)
	Tests of Normality

	Kolmogorov-Smirnov <sup>a</sup>		Shapiro-Wilk			
	Statistic	df	Sig.	Statistic	df	Sig.
Fracture Strength	0.079	30	0.200*	0.978	30	0.781

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

Table IV.2: Levene's Test of equality of error variances

Levene's Statistic	df1	df2	Sig.
0.5184	2	27	0.601

		Mean			95% CI	
(I) Ceramic	(J) Ceramic	Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
Procera	Turkom-Cera	-230.10*	86.21	0.042	-453.39	-6.80
	In-Ceram	-88.20	86.21	0.598	-311.49	135.09
Turkom-Cera	Procera	$230.10^{*}$	86.21	0.042	6.80	453.39
	In-Ceram	141.90	86.21	0.275	-81.39	365.19
In-Ceram	Procera	88.20	86.21	0.598	-135.09	311.49
	Turkom-Cera	-141.90	86.21	0.275	-365.19	81.39

**Table IV.3:**Multiple Comparisons of load at fracture (N) between Procera, Turkom-<br/>Cera and In-Ceram copings using Scheffe's Test

\*. The mean difference is significant at the 0.05 level.

Table IV.4:	Chi-square test between treatment group (Procera, Turkom-Cera and In-
	Ceram) and modes of fracture

	Value	Df	p-value
Pearson Chi-Square	1.964	4	0.742
((6,70)) = 11 + 1 + 1 + 1 + 1 + 1 + 1 + 1 + 1 +			

6 (66.7%) cells have expected count less than 5. The minimum expected count is 1.00.



**Figure IV.2:** Histogram of the load at fracture (N) (Effect of luting cements).

Table IV.5:	Shapiro-Wilk test for the load at fracture (Effect of luting cements)
	Tests of Normality

	Kolmogorov-Smirnov <sup>a</sup>			Shapiro-Wilk				
	Statistic	df	Sig.	Statistic df Si				
Fracture Strength	0.189	30	0.008	0.917	30	0.022		

a. Lilliefors Significance Correction

 Table IV.6:
 Comparison of load at fracture (N) between Elite and Fuji I using Mann-Whitney Test with Bonferroni correction

Test Statistics<sup>D</sup>

	Fracture Strength		
Mann-Whitney U	16.000		
Wilcoxon W	71.000		
Z	-2.570		
Asymp. Sig. (2-tailed)	0.030		
Exact Sig. [2*(1-tailed Sig.)]	0.009 <sup>a</sup>		

a. Not corrected for ties.

b. Grouping Variable: Cement

 Table IV.7:
 Comparison of load at fracture (N) between Elite and Panavia F using Mann-Whitney Test with Bonferroni correction

Test	Statistics <sup>D</sup>
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	Fracture Strength		
Mann-Whitney U	0.000		
Wilcoxon W	55.000		
Z	-3.780		
Asymp. Sig. (2-tailed)	0.000		
Exact Sig. [2*(1-tailed Sig.)]	0.000		

a. Not corrected for ties.

b. Grouping Variable: Cement

 Table IV.8:
 Comparison of load at fracture (N) between Fuji I and Panavia F using Mann-Whitney Test with Bonferroni correction

 Test Statistics<sup>b</sup>

	Fracture Strength
Mann-Whitney U	0.000
Wilcoxon W	55.000
Ζ	-3.780
Asymp. Sig. (2-tailed)	0.000
Exact Sig. [2*(1-tailed Sig.)]	0.000 <sup>a</sup>

a. Not corrected for ties.

b. Grouping Variable: Cement

**Table IV.9:** Chi-square test between treatment group and modes of fracture

	Value	df	p-value
Pearson Chi-Square	4.109	4	0.391

6 (66.7%) cells have expected count less than 5. The minimum expected count is 1.00.

## **APPENDIX V** STATISTICAL ANALYSIS TABLES FOR CHAPTER 6

---- Normal



Figure V.1: Histogram of load at fracture (N) of Turkom-Cera, In-ceram and Procera.

Table V.1: Levene's Test of equality of error variances

Levene's Statistic	df1	df2	Sig.
1.943	2	27	0.163

 Table V.2:
 Multiple Comparisons of load at fracture (N) using Tukey's HSD

		Mean Difference		95% Confide	ence Interval
(I) Ceramic	(J) Ceramic	( <b>I-J</b> )	(I-J) Sig.		<b>Upper Bound</b>
Procera	Turkom-Cera	-366.93*	0.000	-561.12	-172.74
	In-Ceram	-176.60	0.080	-370.79	17.59
Turkom-Cera	Procera	366.93*	0.000	172.74	561.13
	In-Ceram	190.33	0.056	-3.86	384.52
In-Ceram	Procera	176.60	0.080	-17.59	370.79
	Turkom-Cera	-190.33	0.056	-384.52	3.86

\*. The mean difference is significant at the 0.05 level.

Table V.3: Chi-square test between treatment groups and modes of fracture

	Value	df	p-value			
Pearson Chi-Square	8.368	6	0.212			
a. 9 cells (75.0%) have expected count less than 5. The minimum expected count is 1.00.						



Load at fracture

1400.00

0.0 1200.00

**Figure V.2:** Histograms of the effect of finish line on load at fracture (N) of Turkom-Cera copings.

1600.00

1800.00

# **Table V.4:** Shapiro-Wilk test for the effect of finish line design on load at fracture of Turkom-Cera copings

Tests of Normality								
	-	Kolmogorov- Smirnov <sup>a</sup>			Shapiro-Wilk			
	Ceramic	Statistic	df	Sig.	Statistic	df	Sig.	
Load at fracture	Turkom-Cera Chamfer	.192	10	.200	.845	10	.049	
	Turkom-Cera Shoulder	.133	10	.200	.958	10	.766	

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

# **Table V.5:**Comparison of load at fracture (N) between Turkom-Cera (Chamfer) and<br/>Turkom-Cera (Shoulder) groups using the Mann-Whitney Test

#### Test Statistics<sup>b</sup>

	Load at fracture
Mann-Whitney U	25.000
Wilcoxon W	80.000
Z	-1.890
Asymp. Sig. (2-tailed)	0.059
Exact Sig. [2*(1-tailed Sig.)]	0.063 <sup>°</sup>

a. Not corrected for ties.

b. Grouping Variable: Ceramic



for ceramic= Turkom-Cera with aging

— Normal



- Figure V.3: Histograms of the effect of water storage and thermocycling on load at fracture (N) of Turkom-Cera copings.
- Table V.6: Shapiro-Wilk test for the effect of water storage and thermocycling on load at fracture of Turkom-Cera copings

Tests of Normality								
		Kolmogorov- Smirnov <sup>a</sup>			Shapiro-Wilk			
	Ceramic	Statistic	df	Sig.	Statistic	df	Sig.	
Load at fracture	Turkom-Cera no aging	.192	10	.200	.845	10	.049	
	Turkom-Cera with aging	.257	10	.059	.926	10	.414	

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

Table V.7: Comparison of load at fracture (N) between Turkom-Cera (no aging) and Turkom-Cera (with aging) groups using Mann-Whitney Test

Test Statistics
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	Load at fracture
Mann-Whitney U	25.000
Wilcoxon W	80.000
Z	-1.890
Asymp. Sig. (2-tailed)	0.059
Exact Sig. [2*(1-tailed Sig.)]	0.063 <sup>a</sup>

a. Not corrected for ties.

b. Grouping Variable: Ceramic

No	Tooth length	Mesio-distal width	<b>Bucco-lingual width</b>	Load at fracture
1	22.3	7.4	9.1	1093.15
2	22.1	7.5	8.9	896.77
3	21.5	7.3	9.3	997.28
4	22.3	7.2	9.3	965.36
5	21.7	7.2	9.4	1068.04
6	22.5	7.5	9.5	786.51
7	22.4	7.4	8.8	818.27
8	23.1	7.6	8.7	1093.44
9	22.8	7	9	1077.01
10	21.7	7.3	9.2	953.80

**Table V.8:** Teeth dimensions (mm) and load at fracture (N) for Procera group specimens with chamfer finish line

**Table V.9:** Teeth dimensions (mm) and mean load at fracture (N) for Turkom-Ceragroup specimens with chamfer finish line

No	Tooth length	Mesio-distal width	<b>Bucco-lingual width</b>	Load at fracture
1	21.8	7.4	9.1	1489.78
2	22.9	7.3	9.3	1269.19
3	22.8	7.1	8.7	1258.40
4	22.6	7.5	8.9	1532.30
5	22.5	7.3	9	1544.84
6	21.7	7.4	9.1	1543.40
7	22.8	7.6	9.4	989.15
8	21.9	7.3	9.2	1450.76
9	22.6	7.4	9.1	977.16
10	22.6	7.4	8.7	1363.99

**Table V.10:** Teeth dimensions (mm) and mean load at fracture (N) for In-Ceram group specimens with chamfer finish line

No	Tooth length	Mesio-distal width	<b>Bucco-lingual width</b>	Load at fracture
1	22.8	7.5	8.9	1116.01
2	22.6	7.3	9.5	1504.83
3	22.7	7.2	9.1	1043.79
4	22.6	7.2	8.7	982.36
5	22.4	7.1	9.5	1060.63
6	21.2	7.5	9.1	1387.98
7	22.7	7.6	9	1144.24
8	22.1	7.3	9.3	1077.61
9	21.9	7.6	9.4	940.43
10	22.7	7.4	9.3	1257.78

No	Tooth length	Mesio-distal width	<b>Bucco-lingual width</b>	Load at fracture
1	23	7.2	9.1	1588.69
2	21.7	7.5	8.7	1781.86
3	21.6	7	9.3	1661.83
4	22.8	7.6	9.1	1466.42
5	21.7	7.4	9	1661.01
6	22.2	7.6	9.4	1389.22
7	22.1	7.4	9.1	1326.18
8	22.9	7.3	9.3	1806.01
9	22.2	7.4	9.6	1261.15
10	22.1	7.5	9.4	1510.00

**Table V.11:** Teeth dimensions (mm) and mean load at fracture (N) for Turkom-Ceragroup specimens with shoulder finish line

**Table V.12:** Teeth dimensions (mm) and mean load at fracture (N) for Turkom-Ceragroup specimens subjected to artificial ageing

No	Tooth length	Mesio-distal width	<b>Bucco-lingual width</b>	Load at fracture
1	21.9	7.2	9.2	1102.24
2	22.2	7.1	9.2	1063.97
3	22.5	7.2	9.3	1154.08
4	22.6	7.4	8.7	1145.56
5	21.8	7	8.9	1415.76
6	22.3	7.1	8.4	1144.50
7	22.2	7.2	9.6	959.20
8	22.6	7.3	9.7	1099.53
9	22.3	7.1	9.9	1338.41
10	22.8	7	8.8	1320.08

## APPENDIX VI STATISTICAL ANALYSIS TABLES FOR CHAPTER 7



**Figure VI.1:** Histogram of marginal integrity (µm) of Turkom-Cera, In-ceram and Procera

# **Table VI.1:** Shapiro-Wilk test for marginal integrity of Turkom-Cera, In-ceram and Procera

#### Tests of Normality

	Kolmogorov-Smirnov <sup>a</sup>			Shapiro-Wilk			
	Statistic	df	Sig.	Statistic	df	Sig.	
Marginal integrity	0.108	30	0.200 <sup>*</sup>	0.957	30	0.262	

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

Table VI.2: Test of homogeneity of variances

Levene Statistic	df1	df2	Sig.
0.182	2	27	0.834

		Mean Difference		95% Confidence Interval		
(I) Ceramic	(J) Ceramic	(I-J)	Sig.	Lower Bound	Upper Bound	
Turkom-Cera	In-Ceram	-22.32 <sup>*</sup>	0.001	-35.99	-8.65	
	Procera	14.81 <sup>*</sup>	0.032	1.14	28.48	
In-Ceram	Turkom-Cera	22.32 <sup>*</sup>	0.001	8.65	35.99	
	Procera	37.13 <sup>*</sup>	0.000	23.46	50.80	
Procera	Turkom-Cera	-14.81 <sup>*</sup>	0.032	-28.48	-1.14	
	In-Ceram	-37.13 <sup>*</sup>	0.000	-50.80	-23.46	

**Table VI.3:** Multiple Comparisons of marginal integrity by Tukey's HSD Test

\*. The mean diference is significant at the 0.05 level.





Figure VI.2: Histograms of the effect of finish lines on marginal integrity (µm)

Table VI.4:	Shapiro-Wilk	est for the effect	t of finish lines	on marginal	integrity
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	-	Kolmogo	Kolmogorov-Smirnov <sup>a</sup>		Shapiro-Wilk		
	All-ceramic System	Statistic	df	Sig.	Statistic	df	Sig.
Marginal integrity	Turkom-Cera CH	0.149	10	0.200 <sup>*</sup>	0.941	10	0.566
	Turkom-Cera SH	0.129	10	0.200 <sup>*</sup>	0.979	10	0.962

**Tests of Normality** 

a. Lilliefors Significance Correction

\*. This is a lower bound of the true significance.

## **APPENDIX VII**

## PATIENT INFORMATION SHEET (English)

Please read the following information carefully. Do not hesitate to discuss any questions you may have with your doctor.

Study Title: Clinical and mechanical evaluation of a new all-ceramic restorative material Turkom-Cera

#### Introduction:

You are invited to participate in this project, which will be explained below. These pages contain information about a research project that we are inviting you to take part in. The purpose of this information is to explain to you clearly and openly all the steps and procedures of this project. The information is to help you decide whether you would like to take part in the research or not. Dr. Bandar AL-Makramani is a PhD student at Faculty of Dentistry, University of Malaya and the principal investigator in this project. Prof. Dato' Dr. Abdul Aziz Razak is his supervisor and the Head of Department of Conservative Dentistry, Faculty of Dentistry, University of Malaya. Prof. Dr. Mohamed Ibrahim Abu-Hassan is his second supervisor and the Dean of the Faculty of Dentistry, Universiti Teknologi MARA.

#### What is the purpose of this study?

As the demand for more natural-looking crowns has increased, dentists and porcelain manufacturers have investigated a number of methods to help reinforce ceramics with the goal of fabricating an all-ceramic restoration that delivers excellent aesthetics and good biocompatibility.

A new all-ceramic material Turkom-Cera, particularly with aluminium oxide, is being introduced in an attempt to provide high-quality with effective cost in coping and to improve clinical success. Independent studies of basic comparative data are necessary to characterize this new material in relation to mechanical properties. Therefore, the first part of my study is to evaluate mechanical properties of Turkom-Cera all-ceramic material. The second part of my study is to examine the clinical performance of Turkom-Cera all-ceramic crowns in the patient mouth for a period of 16 months.

#### What are the procedures to be followed?

The study will be explained to you and we will answer any questions you may have during your first appointment.

You will then be asked few questions about your past dental history and medical history to ensure that you are eligible to participate in this study. If you are eligible and you have agreed to participate in the study you will get a complete intraoral and radiographic examination. We will make impressions for the upper and lower jaws in order to prepare special tray and temporary crown/crowns

In the next appointment the abutment tooth/teeth will be prepared for all-ceramic crown using special preparation burs, then impression will be taken. The temporary crown/crowns will be fixed to maintain gingival health and prevent tooth/teeth sensitivity between visits. The impressions will be used for the fabrication of crown/crowns.

Once we receive the crown/crowns from the dental laboratory you will be called to come for the try-in and then for final cementation of the restoration.

After final cementation of the crown/crowns two examiners will evaluate the crown/crowns for the following:

(1) margin integrity, (2) shade compatibility, (3) surface texture, (4) anatomic form, (5) secondary caries, (6) wear of crown and opposing dentition, (7) cracks and fractures of the crowns and (8) Post-operative sensitivity. Modified USPHS criteria will be used to assign a rating of Alpha, Bravo, or Charlie to each of the 8 categories of evaluation.

Each restoration will be evaluated one to ten days after cementation (baseline), and thereafter every 6 months period. Evaluations will be performed by the two clinicians using a mirror, explorer and intraoral photographs.

At the end of the study you will also be asked to rate your restoration/restorations.

#### Who should not enter the study?

You are being asked to take part because of the following:

-You are over 18 years old with good general and dental health and with no active tooth decay (caries) present and no periodontal disease.

- You do not have any existing temporomandibular disorder, (e.g. clicking, popping, pain on opening) or parafunctional habits (e.g. bruxism, clenching).

- You have good oral hygiene and compliance.

### What will be the benefits of the study:

#### (a) to you as a subject?

The subject will receive a metal-free restoration which is strong, aesthetic, comfortable, not allergic and free of charge.

#### (b) to the investigator?

To test a new all-ceramic material which provide restorations with; perfect fitting, perfect margin, high quality work, hygienic and strong. This study is also conducted as a fulfilment of Dr. . Bandar AL-Makramani's PhD studies at Faculty of Dentistry, University of Malaya, Malaysia.

### What are the possible drawbacks?

None

### Can I refuse to take part in the study?

Yes, participation in this study is completely voluntary. You can refuse to take part in this study if you wish to do so, without any consequence on your future visits at Faculty of Dentistry, University of Malaya.

### Who shall I contact if I have additional questions during the course of the study?

Dr. Bandar AL-Makramani	Prof. Dato' Dr. Abdul Aziz bin Abdul Razak
B.D.S. (Jordan), H.D.D (Baghdad),	BDS (Mal), MSc (Manchester), PhD (Bristol)
MDSc. (Mal).	FICD, FADI, FICCDE
PhD student	Head
Department of Conservative Dentistry	Department of Conservative Dentistry
Faculty of Dentistry, University of Malaya	Faculty of Dentistry, University of Malaya
50603 Kuala Lumpur, Malaysia.	50603 Kuala Lumpur, Malaysia.
Tel. No.: 013-2508516	Tel. No.: 03-79674806

#### **BORANG MAKLUMAT PESAKIT** (Malay)

Sila baca maklumat berikut dengan teliti, dan sekiranya ada apa-apa soalan, sila bincangkan dengan doktor berkenaan.

#### **Tajuk Kajian:**

Penilaian klinikal dan mekanikal terhadap bahan seramik Turkom-Cera

#### **Pengenalan:**

Anda dipelawa untuk menyertai projek separti yang akan diterangkan di bawah. Lembaran ini mengandungi butir-butir berkenaan projek penyelidikan di mana seperti pelawaan yang dibuat kepada anda. Tujuan penerangan ini adalah untuk menerangkan dengan jelas serta secara terbuka kepada anda tentang langkah-langkah dan prosedur projek ini. Penerangan ini juga bertujuan bagi membantu anda bagi memilih sama ada bersetuju menyertai atau tidak di dalam projek yang dimaksudkan. Dr Bandar Al-Makramani ialah seorang penuntut PhD di Fakulti Pergigian, Universiti Malaya yang juga ketua penyelidik dalam projek ini. Penyelia beliau, Prof. Dato' Dr. Abdul Aziz Razak, Ketua Jabatan Pemliharaan Gigi, Fakulti Pergigian, Universiti Teknologi Mara, Shah Alam selaku penyelia bersama dalam projek ini.

#### Apakah tujuan kajian ini?

Terdapat peningkatan dalam permintaan korona yang lebih nampak keasliannya yang membuatkan Dr Gigi dan pengeluar-pengeluar bahan porselain, mengkaji cara-cara bagaimana dapat mengukuhkan bahan seramik bagi tujuan membentuk kesemua restorasi seramik yang boleh memberi estetik yang tinggi nilainya serta 'biocompatibilty' yang baik.

Bahan baru Turkom-Cera keseluruhannya berbentuk seramik, khusus yang mengandungi aluminum oksida, telah diperkenalkan. Ini adalah salah satu usaha bagi memberikan mutu terbaik dengan harga yang berpatutan untuk menyesuaikan dengan kehendak kejayaan klinikal. Data bebas dalam kajian komparatif asas adalah mustahak untuk memberikan sifat baru kepada bahan ini selaras dengan sifat sifat mekanikalnya. Oleh itu dalam peringkat awal kajian saya ini adalah bagi menilai sifat mekanikal bahan Turkom-Cera. Bahagian kedua kajian ini adalah bagi mengkaji keberkesanan korona dari bahan Turkom-Cera ini dari segi penggunaannya di klinik bagi pesakit-pesakit dalam jangka masa 16 bulan.

#### Apakah langkah-langkah perlu diikuti?

Kajian ini akan menerangkan kepada anda serta menjawab soalan-soalan anda semasa temujanji pertama nanti.

Anda akan disoal tentang sejarah kesihatan pergigian dan kesihatan anda bagi memastikan yang anda betul betul layak bagi menyertai dalam kajian ini. Sekiranya anda berjaya dan bersetuju menyertai kajian ini , maka anda akan diberikan rawatan 'intra-oral' serta kajian radiograf mulut anda. Seterusnya kami akan mengambil impresi atau acuan gigi atas serta bawah untuk membentuk ceper khas dan juga korona sementara.

Dalam temujanji kedua, gigi abutmen anda akan dibuat persediaan melalui pemotongan menggunakan bur, dan seterusnya impresi akan diambil buat kali kedua. Korona sementara dipasang bagi tujuan pemeliharaan sekitar kawasan gingiva serta mengelakkan rasa sensitif semasa dalam rawatan. Impresi tadi akan digunakan bagi penyediaan korona tetap kemudian nanti.

Apabila kami telah menerima prostesis korona tadi dari makmal, maka kami akan memanggil anda bagi tujuan pemasangan percubaan dan kemudiannya akan dipasang secara tetap di dalam mulut anda.

Setelah pemasangan krona dibuat secara tetap, maka dua orang pemeriksa akan menilai restorasi tersebut berdasarkan kepada aspek-aspek berikut:-

(1) intergriti pingir. (2) perbezaan warna. (3) tekstur permukaan. (4) bentuk anatomi. (5) karies sekunder. (6) hakisan korona serta gigitan bertentangan dan (7) keretakan serta pecahan korona tadi. Tiap-tiap restorasi korona nanti akan dinilai dari satu hingga sepuluh hari selepas disimen dan seterusnya bagi tempuh setiap enam bulan berikutnya. Penilaian akan dilakukan oleh dua orang pensyarah dengan menggunakan cermin, 'explorer' dan gambar 'intra oral'.

Dan diakhir kajian anda juga akan dikehendaki menilai sendiri tentang kebaikan restorasi tersebut.

#### Siapakah tidak layak diterima untuk kajian?

Anda dipelawa menyertai kajian ini diatas sebab-sebab berikut:

-Anda adalah berumur lebih 18 tahun dan mempunyai kesihatan gigi yang baik serta tiada terdapat karies serta penyakit periodontal yang lain.

-Anda tiada kesulitan mengenai kedudukan temporomandibular; contohnya 'clicking', 'popping', sakit ketika membuka mulut atau habitual parafunctional seperti 'bruxism' dan 'clenching'.

-Anda mempunyai kesihatan gigi yang baik dan patuh dengan cara pemeliharaannya.

### Apakah manfaat kajian ini: (a) kepada anda sebagai pesakit?

Anda akan memperolehi restorasi korona yang agak kukuh, cantik, selesa, tiada alergi, tiada bahan logam serta secara percuma pula.

#### (b) kepada penyelidik?

Kepada penyelidik, mereka akan memperolehi cara menguji bahan seramik baru yang memberi ketepatan padanan (perfect fit), ketepatan pingir restorasi, mutu kerja yang tinggi dan juga dari segi kebersihan dan kekuatan. Dapat mencuba dan mengkaji teknik baru restorasi keseluruhannya seramik yang mempunyai sifat-sifat kepadanan yang baik, pingiran bagus, berkualiti tinggi, teguh dan bersih.

### Apakah halangan kajian ini?

Tiada.

### Bolehkan saya menolak dari menyertai kajian ini?

Ya, anda boleh menolaknya kerana iannya bersifat sukarela. Anda boleh berbuat demikian tanpa menjejaskan peluang anda bagi mendapatkan sebarang rawatan di Fakulti Pergigian, Universiti Malaya ini.

# Siapakah patut saya berhubung sekiranya ada soalan tambahan sepanjang masa kajian ini?

Anda bolehlah berhubung dengan:

Dr. Bandar AL-Makramani B.D.S. (Jordan), H.D.D (Baghdad), MDSc. (Mal). Penuntut PhD Jabatan Pemeliharaan Gigi Fakulti Pergigian, Universiti Malaya 50603 Kuala Lumpur, Malaysia. Tel. No.: 013-2508516

### Prof. Dato' Dr. Abdul Aziz bin Abdul Razak BDS (Mal), MSc (Manchester), PhD (Bristol), FICD, FADI, FICCDE

Ketua Jabatan Pemeliharaan Gigi Fakulti Pergigian, Universiti Malaya 50603 Kuala Lumpur, Malaysia. Tel. No.: **03-79674806** 

# CONSENT BY PATIENT FOR CLINICAL RESEARCH FACULTY OF DENTISTRY, UM, KL.

I, Identity Card No		
of		
(Address) hereby agree to take part in the clinical research ( clinical study ) specified below : <u>Title of Study</u> : Clinical and mechanical evaluation of new all-ceramic material Turkom-Cera		
the nature and purpose of which has been explained to me by <i>Dr</i> . and interpreted by	Bandar M. A. AL-Makramani (PhD Candidate) (Name & designation of doctor) to the best of his/her ability inEnglish	
(Name & designation of interpreter) language/dialect.		
I have been told about the nature of the clinical research in terms of methodology, possible adverse effects and complications ( as per the patient information sheet). After knowing and understanding all the possible advantages and disadvantages of this clinical research, I voluntarily consent of my own free will to participate in the clinical research specified above.		
I understand that I can withdraw from this clinical research at any time without assigning my reason whatsoever and in such a situation shall not be denied the benefits of usual treatment by the attending doctors.		
Date	Signature or thumbprint	
IN THE PRESENCE OF		
Name,		
I/C No Signature	(Witness for signature of patient)	
Designation		
I confirm that I have explained to the patient the nature and purpose of the above mentioned clinical research.		
Date	Signature	
	(Attending doctor)	

CONSENT BY PATIENT FOR CLINICAL RESEARCH R.N. Name Sex Age Unit
# **Examination form**

Date:	
Patient Name:	
Patient No.:	
Tooth Number:	
Examiner Name:	Signature:

Rating Category	Alpha(A)	Bravo(B)	Charlie(C)
Marginal integrity			
Shade			
Surface texture			
Anatomic form			
Secondary caries			
Wear			
Presence of cracks and fracture			
Post-operative sensitivity			

# **APPENDIX VIII** LIST OF MATERIALS USED IN THIS STUDY

### MATERIAL

### MANUFACTURER

Retraction cords (Ultrapack®)	Ultradent, South Jordan, Utah, USA	
Polyether impression material (Impregum)	(3M ESPE, Seefeld, Germany)	
Self-curing acrylic resin	Ostron 100, GC Corp., Tokyo, Japan	
Die stone	Densite, Shufo, Japan	
Alginate impression material (Aroma Fine DF III)	GC Corp., Tokyo, Japan	
Interocclusal registration wax (Alminax)	Whip-Mix Corp., Louisville, Ky	
Chemically-polymerized composite resin (Trim)	Bosworth Co., Skokie, USA	
eugenol-free temporary cement (Temp Bond)	Kerr GmbH, Karlsruhe, Germany	
All-ceramic material (Turkom-Cera)	Turkom-Ceramic (M) Sdn. Bhd., Puchong, Malaysia	
Articulating film (Arti-Fol Metalic)	Dr. Jean Bauch KG, Köln, Germany	
articulating paper (Bausch Occlusionspapier)	Dr. Jean Bauch KG, Köln, Germany	
Self adhesive resin luting cement (RelyX U100)	3M ESPE, Seefeld, Germany	
Finishing discs (Soflex)	3M, St. Paul, MN	
All-ceramic material (In-Ceram)	Vita Zahnfabrik, Bad Säckingen, Germany	
All-ceramic material (Vitadur-N)	Vita Zahnfabrik, Bad Säckingen, Germany	

Diamond polishing paste (DIAMAT <sup>™</sup> )	PACE Technologies, Tucson, USA	
Lubricant (DIALUBE)	PACE Technologies, Tucson, USA	
silicon carbide (SiC) abrasive papers	Buehler Ltd., Lake Bluff, IL	
Zinc phosphate cement (Elite)	GC Corporation, Tokyo, Japan	
Glass ionomer cement (Fuji I)	GC Corporation, Tokyo, Japan	
resin modified glass ionomer cement (Fuji Plus)	GC Corporation, Tokyo, Japan	
Resin luting cement (Panavia F)	Kuraray Medical Inc., Okayama, Japan	
Silane coupling agent (Clearfil Silane Kit)	Kuraray Medical Inc., Okayama, Japan	
Silicon impression material (Express putty)	3M ESPE, St. Paul, MN, USA	
All-ceramic material Procera AllCeram	Nobel Biocare, Göteborg, Sweden	
Self-curing acrylic resin	Pattern Resin, GC, Corporation, Tokyo, Japan	
Dental wax (College Wax)	Metrodent Limited, England	
Non-precious metal alloy (Wiron 99)	BEGO, Brmen, Germany	
Silicon impression material	Optosil, Bayer Dental, Leverkusen, Germany	
Silicone impression material (Aquasil Monophase Ultra)	Dentsply Caulk, Dentsply International Inc., Milford, Germany	
Boxing wax (Boxing In Wax)	Metrodent Ltd., Huddersfield, Enland	

### APPENDIX IX LIST OF EQUIPMENTS AND INSTRUMENTS USED AND CITED IN THIS STUDY

EQUIPMENTS/ INSTRUMENTS	DISCRIPTION	MANUFACTRER
ESPE Pentamix	Polyether Mixing Machine	(Pentamix, 3M, ESPE AG, Germany)
Pindex system	Placing pinholes in the cast for die preparation	Coltene/Whaledent Inc., NY, USA
Articulator (Kavo)	Semiadjustable articulator	Kavo, Leutkirch, Germany
BEGO Topstar	Sandblaster	BEGO, Germany
Coltolux3	Visible light curing unit	Coltene/Whaledent Inc., Mahwah, NJ, USA
Programat p300	Porcelaim Furnace	Ivoclar Vivadent AG, Schaan, Liechtenstein
Vitasonic unit	Mixing machine	Vita Zahnfabrik, Bad Säckingen, Germany
In-Ceramat	Porcelaim Furnace	Vita Zahnfabrik, Bad Säckingen, Germany
Multimat-Touch & Press	Porcelaim Furnace	Dentsply, Dreieich, Germany
Metaserv <sup>®</sup> 2000	Grinder/polisher machine	Buehler, UK
Delta Ultrasonic Cleaner D150	Ultrasonic Cleaner	Taiwan Delta New Instrument Coltd., Dongguan, China
Mitutoyo	Digital caliper	Mitutoyo Corp, Tokyo, Japan
Memmert Oven	Incubator	Memmert GmbH & Co. KG, Germany

Instron 4302	Universal Testing Machine	Instron Corporation, England
BEGO Trinton	Steam cleaner	BEGO, Germany
HMV	Vickers microhardness testing machine	Shimadzu Corp., Tokyo, Japan
Shear bond test apparatus	Alignment apparatus for specimen bonding	Faculty of Engineering, University of Malaya
FEI Quanta 200	Scanning Electron Microscope	FEI Co., Eindhoven, Netherlands
Nobel Biocare <sup>TM</sup> Procera Piccolo	Procera-Scanning Machine	Renishaw, UK
Makramani-Load	Vertical Loading Apparatus	Faculty of Dentistry, University of Malaya
Piezon Master 400	Ultrasonic Scaler	EMS, Nyon, Switzerland
Olympus SZ61	Stereo microscope	Olympus Corp., Tokyo, Japan
Paralleling apparatus	For preparation of teeth	Faculty of Engineering, University of Malaya
Thermocycling machine	Thermocycling machine	Faculty of Engineering, University of Malaya
Shimadzu testing machine	Universal Testing Machine	Shimadzu Corp., Tokyo, Japan
Holding jig	For positioning the coping and the tooth model precisely	Faculty of Engineering, University of Malaya
Tecnika	Digital torque control motor	ATR, Dentsply, Pistoia, Italy

# **APPENDIX X**

# PAPERS PUBLISHED, ACCEPTED AND SUBMITTED

### FOR PUBLICATION

&

## **MEETING ABSTRACTS**

#### PAPER I

### (THE JOURNAL OF CONTEMPORARY DENTAL PRACTICE)

AL-MAKRAMANI BMA, RAZAK AAA, AND ABU-HASSAN MI. EFFECT OF LUTING CEMENTS ON THE COMPRESSIVE STRENGTH OF TURKOM-CERA ALL-ERAMIC COPINGS. J CONTEMP DENT PRACT 2008; 9 (2); 33-40.

#### PAPER II

#### (JOURNAL OF PROSTHODONTICS)

AL-MAKRAMANI BMA, RAZAK AAA, AND ABU-HASSAN MI. COMPARISON OF THE LOAD AT FRACTURE OF TURKOM-CERA TO PROCERA ALLCERAM AND IN-CERAM ALL-CERAMIC RESTORATIONS. J PROSTHODONT 2008; 18 (6): 484-488.

#### PAPER III

#### (JOURNAL OF APPLIED ORAL SCIENCE)

AL-MAKRAMANI BMA, RAZAK AAA, AND ABU-HASSAN MI. BIAXIAL FLEXURAL STRENGTH OF TURKOM-CERA CORE COMPARED TO TWO OTHER ALL-CERAMIC SYSTEMS. J APPL ORAL SCI; ACCEPTED FOR PUBLICATION August 11, 2009.

#### PAPER IV

### (INTERNATIONAL JOURNAL OF PROSTHODONTICS)

AL-MAKRAMANI BMA, RAZAK AAA, AND ABU-HASSAN MI, ESHAMSUL S, LUI JL, AND YAHYA NA. MARGINAL INTEGRITY OF TURKOM-CERA COMPARED TO OTHER ALL-CERAMIC MATERIALS: EFFECT OF FINISH LINE. INT J PROSTHODONT 2010; SUBMITTED FOR PUBLICATION.

# ABSTRACTS PUBLISHED IN JOURNAL OF DENTAL RESEARCH SPECIAL ISSUES (MEETING ABSTRACTS)

- 1. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. EFFECT OF SURFACE TREATMENTS ON SHEAR BOND STRENGTH OF TURKOM-CERA. J DENT RES 2009; 88 (SPEC ISS A): 98.
- 2. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. BIAXIAL FLEXURAL STRENGTH OF TURKOM-CERA COMPARED TO CORE- AND VENEER-CERAMICS. J DENT RES 2008; 87 (SPEC ISS C): 0021.
- 3. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. BOND STRENGTH OF LUTING CEMENTS TO TURKOM-CERA ALL-CERAMIC MATERIAL. J DENT RES 2008; 87 (SPEC ISS C): 0084.
- 4. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. FLEXURAL STRENGTH OF NEW ALL-CERAMIC CORE MATERIAL. J DENT RES 2008; 87 (SPEC ISS B): 2320.
- 5. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. LOAD AT FRACTURE OF TURKOM-CERA COPINGS USING DIFFERENT LUTING CEMENTS. J DENT RES 2007; 86 (SPEC ISS B): 0090.
- 6. <u>AL-MAKRAMANI BMA</u>, RAZAK AAA, AND ABU-HASSAN MI. FRACTURE STRENGTH OF TURKOM-CERA COMPARED TO TWO ALL-CERAMIC SYSTEMS. J DENT RES 2007; 86 (SPEC ISS B): 0015.

**AWARDS** 

1. TRAVEL AWARD

(IADR/MALAYSIAN DIVISION) 7<sup>TH</sup> SCIENTIFIC MEETING OF INTERNATIONAL ASSOCIATION FOR DENTAL RESEARCH (IADR) MALAYSIAN SECTION AND NINTH ANNUAL GENERAL MEETING, FEBRUARY 23, 2008.

- 2. <u>UNIVERSITY OF MALAYA FELLOWSHIP SCHEME AWARD</u> 2007/2008 UNIVERSITY OF MALAYA FELLOWSHIP SCHEME, JULY 2007- JULY 2009.
- 3. TRAVEL AWARD

(IADR/MALAYSIAN DIVISION) 6<sup>TH</sup> SCIENTIFIC MEETING OF INTERNATIONAL ASSOCIATION FOR DENTAL RESEARCH (IADR) MALAYSIAN SECTION AND EIGHTH ANNUAL GENERAL MEETING, MARCH 10, 2007.