Chapter One:
Introduction
and
Objectives of Study

1.1. Introduction:

In conservative dentistry, interests in the development of restorative materials and restorative material quality have been major interests (Kelly et al., 1996). Wide varieties of treatment options have been suggested for the restoration of damaged tooth structure, such as inlays, onlays, veneers, metal or ceramic crowns. The prognosis of these treatment options was reported to be dependent on multiple factors, such as the luting agent, the design of the restoration, or treatment technique (Blatz et al., 2003). Although ceramic materials are considered to be expensive and difficult to repair, they have been recommended for crown restorations because of their aesthetically pleasing appearance, intra-oral chemical stability and stronger bonding capabilities to bonding agents and tooth structure (Aboushelib et al., 2007). On the other hand, one of the most popular core materials is composite resin because it can bond to dentine, can be finished and contoured easily, having lowest failure rate after silver amalgam , and good aesthetic outcome under ceramic restorations (Craig & Powers, 2002; Walmsley et al., 2002).

In this study, dental zirconia ceramics was investigated because it is an extensively used material (Kim et al., 2005). The study was necessary to investigated the mechanical properties of ceramic material when bonded to different core materials by different luting cements. In the oral cavity, masticatory forces applied to prosthesis produce a combination of mechanical stresses that occur simultaneously. Tensile strength is more appropriate for evaluating the adhesive capability of composite resin to ceramics. Failure has been reported to always occur in the adhesive layer (El-Zohairy et al., 2003; Davidson et al., 1984).

Many factors affect the tensile bond strength of dental ceramics. Surface treatments (airborne-particle abrasion and hydrofluoric acid etching) and silanization after surface treatment appear crucial for bond strength, especially with resin cements (Yang et al., 2008; Peumans et al., 2007; Wang et al., 2007; Pisani-Proenca et al., 2006).

The null hypothesis for this study is that there is no difference in the tensile bond strength of various composite resin core materials luted to ceramic copings by different luting materials.

1.2. Objectives of study:

The objectives of this study are:

1. To investigate the tensile bond strength of ceramic copings luted to different composite resin core materials.
2. To investigate the effect of different luting materials on tensile bond strength of ceramic copings bonded to composite resin core materials.

Chapter Two:
Literature Review

2.1. Post crown (Historical developments):

A review paper by Morgano and Brackett (1999) mentioned that historically many efforts were made to develop crowns retained by posts. Progress was, however, limited because of endodontic therapy failure. Black, a 19th century dentist, favoured using metal posts in which a porcelain-faced crown was secured by a screw passing into a gold-lined root canal.

In 1878, there was the introduction of a one-piece post and crown, but there was a problem if removal of the crown was required after cementation. These difficulties led to the development of the post-and-core restoration as separate entity, with a crown cemented over the core.

With the beginning of scientific endodontic therapy in the 1950’s, the challenges increased for restorative dentistry. Then, cast posts and cores became routine methods for restoration of endodontically treated teeth.

2.2. Core materials:

2.2.1. Introduction:

A core replaces the lost tooth structure to provide retention for a cast restoration (Charbeneau, 1989). The development of materials capable of bonding to tooth structure has enhanced the dentist’s ability to rehabilitate compromised endodontically treated teeth. A number of techniques have been suggested for restoring endodontically treated teeth, such as cast or directly placed posts-and-cores (De-Goes et al., 2007).

On the other hand, core reconstruction without a post is accomplished for vital teeth. Small dentine defects can be restored with a bonded material and more extensive dentine defects that often occur with previous endodontic treatment generally require additional support for the core material. This support can be obtained by rigid all-ceramic zirconium or titanium post materials depending on the aesthetic and functional requirements. That is what leads to the decrease in the indications for a cast post and core as an indirect procedure for prosthodontic reconstructions (Paul and Schärer, 1997).

A long term clinical study carried out by Ellner et al., (2003) reported, “if recommended procedures are strictly followed, posts and cores can serve as abutments for fixed single crowns with satisfactory long-term results”.

2.2.2. Classification of core materials:

2.2.2.1. Silver amalgam:

Silver amalgam was the strongest core materials with the prefabricated post systems (Coltak et al., 2007), but silver amalgam with high-copper displayed lower mechanical properties than other core dental materials (Yüzügüllü et al., 2008). A ferrule preparation and the bonding agent designed for silver amalgam core-dentin bonding can each increase the fracture strength for teeth receiving cast crowns after endodontic therapy and post with amalgam core restorations (Aykent et al., 2006).

2.2.2.2. Titanium:

Titanium core is popular and it is used in combination with a titanium post as one unit. The tensile bond strength value of titanium and gold alloy when used to construct a post-and-core are similar when cemented with either conventional cement or resin bonding agent (Menani et al., 2008) and after using air-particle abrasion, silica coating and silanization surface treatments (Schmage et al., 2006). The surface etching of titanium and cast gold alloy post-and-cores have no significant influence on tensile bond strength (Menani et al., 2008).

2.2.2.3. Gold:

Cast gold has been used successfully for many years as it exhibits high strength, low solubility, and its coefficient of thermal expansion is similar to that of the tooth substance. It is also aesthetically pleasing under all-ceramic restorations (Cheung, 2005; Carrossa et al., 2001). Gold containing alloys should not be acid-etched to provide a microscopic area of retention. Airborne-particle abrasion with aluminium oxide is the alternative method for surface treatment for the metal to improve retention (McDonald et al., 2004).

2.2.2.4. Composite:

Composite resin core material has high physical properties (Walmsley et al., 2002). Santos et al. (2004) investigated the diametral tensile strength of non-metallic posts with composite resin cores, and reported high tensile strength when used with resin cement.

2.2.2.5. Glass fibre:

Fibre-reinforced materials have highly favourable mechanical properties, and their strength to weight ratio is superior to those of most alloys. When compared with metals, they offer many other advantages, for example: non-corrosiveness, translucency, good bonding properties, and ease of repair. Glass fibre is commonly used as a post in endodontically treated teeth and completes tooth preparation by the particular composite resin core (Freilich et al., 2000). Glass fibre luted to dentine by different resin modified luting agents showed high retentive value with RelyX luting than other resin modified glass ionomer luting agents when tested using tensile bond strength *in-vitro* study by Bonfante et al. (2007). On the other hand, in a long term retrospective study on fibre-reinforced posts carried out by Segerström et al. (2006), they reported 35% failure rate, not including the teeth that developed recurrent dental caries.

2.2.2.6. Glass ionomer:

Glass ionomer was one of adhesive restorative materials (Gladwin and Bagby, 2000; Jacobsen, 1998). However, it has limits to its application because it lacks fracture resistance. Although glass ionomer has many limitations when used as a core material (Matsuya et al., 1996) but the cement has many uses because it can bond to different alloys (Mojon et al., 1992; Mount, 1990). On the other hand, using the glass ionomer as core material should be avoided because of its low strength, low stiffness, poor bonding characteristics and high solubility (Cheung, 2005).

2.2.2.7. Compomer:

Compomer is a combination of glass ionomer cement and composite/dentinal bonding system (Gladwin and Bagby, 2000). Although compomer filling material is considered as a material that release fluoride during polymerization that lead to less caries development compared with other types of composite resin materials (Lennon et al., 2007), this fluoride release from compomer restorative material is not proven by prospective clinical studies whether the incidence of secondary caries can be significantly reduced (Weigand et al., 2007). Compomer and resin reinforced glass ionomer have less physical strength characteristics than composite resin restorative materials (Christensen, 1997). Compomer develops hygroscopic expansion when used as core material or for adhesive bonding, and can lead to all-ceramic crown failure (Sindel et al., 1999).

2.2.3. Composite resin cores:

2.2.3.1. Introduction:

Early composite resin materials contained methyl methacrylate. However, in the mid-1960s they were replaced with dimethacrylate polymers such as Bis-GMA (Walmsley et al., 2002). Bis-GMA continues to be the most-used monomer for manufacturing present day composite resin restorative materials (Garcia et al., 2006). The bond strength between composite resin and all-ceramic coping materials has not been studied extensively (Kim et al., 2005). Composite resin, when used as core material, is easy to manipulate in one visit (Charbeneau, 1989). Some composite resin core materials have a compressive and tensile strength similar to silver amalgam cores. Composite resin materials are becoming increasingly popular for core build-ups (Walmsley et al., 2002). Packable composite resin is not a total replacement for silver amalgam, but should possibly be in the future (Nash et al., 2001).

2.2.3.2. Types of composite resin core materials:

Walmsley et al. (2002) classified the composite resin restorative materials into:

2.2.3.2a. Conventional (macrofilled):

The conventional material is the first type of composite resin, containing fillers of between 5 and 10 μm. This type of conventional composite resin is difficult to polish and has poor wear resistance. Chamfered macrofilled composite restorations have lower fracture resistance than bevelled microfilled restorations (Donly and Browning, 1992).

2.2.3.2b. Microfilled:

Microfilled composite resins contains colloid silica filler particles of approximately 0.04 μm. Current materials may contain fillers of lithium aluminosilicates, crystalline quartz, or barium aluminoborate silica glasses which make the restoration easily polished (Walmsley et al., 2002). This material has its disadvantages. It has a higher rate of marginal discoloration (Reusens et al., 1999) and has higher thermal expansion, water sorption, and solubility, when compared with conventional composite resins (Hirasawa et al., 1981). These disadvantages can be overcome by increasing the filler levels that result in increased depth of polymerization, colour stability, hardness, compressive strength, and stiffness (St-Germain et al., 1985).

2.2.3.2c. Small particles:

The filler size of this type of small particle composite resin is 1-5 μm, which is developed to improve wear resistance (Gladwin and Bagby, 2000).

2.2.3.2d. Hybrid:

The main filler particle size of hybrid composite resin is typically in the range of 1-1.5 μm, and these particles contain a combination of barium glass and other fillers. Hybrid composite resin is the type of resin material which performs as an anterior restorative material (Reusens et al., 1999). A conventional hybrid composite resin has significantly greater diametral tensile strength and flexural strength when compared with other resin-based composite resins (Cobb et al., 2000). In a study that compared the effects of chemical and mechanical surface treatment on the microtensile bond strength of microfilled hybrid composite resins, airborne-particle abrasion was found to give satisfactory bond strength for composite resin repair by composite resin (Papacchini et al., 2007).

2.2.3.2e. Flowable:

Flowable composite resin has low viscosity, and has less abrasion resistance. Flowable composite resin is typically used as the initial addition of a composite restoration and then covered with hybrid material (Gladwin and Bagby, 2000).

2.2.3.2f. Condensable:

Condensable composite resins have filler particle features that inhibit the sliding of the filler particles with one another (Gladwin and Bagby, 2000). However, it seems that not all of these condensable materials qualify for stress-loaded posterior restorations (Manhart et al., 2001).

2.2.3.3. Physical and mechanical properties for composite resin core material:

2.2.3.3a. Polymerization shrinkage:

Polymerization shrinkage has a significant effect on tensile bond strength of the centre of the mass than on the periphery, for the composite attached to enamel (Cabrera and De la

Macorra, 2007). The factor which increases polymerization shrinkage is the low-viscosity resin (Walmsley et al., 2002). A high rate of bond strength failure appears when the material has high polymerization shrinkage (Kleverlaan and Feilzer, 2005). A commercially light-emitting diode is better than halogen-based light-curing units, but they may not adequately polymerize resin-based composites, which can lead to restoration failures and adverse pulpal responses to un-polymerized monomers (Dunn and Bush, 2002).

2.2.3.3b. Coefficient of thermal expansion:

The coefficient of thermal expansion is the measurement of the change in volume in relation to the change in temperature (Gladwin and Bagby, 2000). Some light-polymerizing composite resins with higher energy density are beneficial to lower the coefficient of thermal expansion (Baek et al., 2008). Restorative materials like beta-quartz glass ceramics and tetric-ceram resin, which have a coefficient of thermal expansion approximating that of enamel, would seem to be the material of choice in reducing the problem of marginal microleakage as a result of miss-match in thermal coefficients of expansion (Santini et al., 2006).

2.2.3.3c. Water sorption and solubility:

The weight gained when a dental material is immersed in water is water sorption. Solubility is the amount of material that dissolves in a given amount of liquid in a given time (Gladwin and Bagby, 2000). Water sorption level was the same for the nano-filled, mini-filled and micro-filled resins, but solubility was lower for the nano-filled than for mini-filled and micro-filled resins. The filler characteristics varied among the different composite materials (Berger et al., 2009). Poly-acid modified composite resin showed the lowest water solubility when compared with resin-modified glass ionomer and light-cured composite resin (Ayna et al., 2006).

2.2.3.3d. Elastic modulus:

The elastic modulus is the proportionality of constant stress and strain (Gladwin and Bagby, 2000). A high modulus of elasticity of composite restorative core materials placed in restrictive cavities cause tension in the material with possible subsequent distortion of the bond to tooth structure. A low modulus of elasticity is not necessarily associated with high bond strength (Ilie et al., 2006). Elastic modulus and polymerization shrinkage strain occur as a function of filler fraction (Sakaguchi et al., 2004). Composite resin has a low modulus of elasticity that can cause deformation of the core, which can result in failure of cement luting for a cast restoration (Kovarik, 1992). Composite resin also has a high potential of marginal leakage, especially in deep cervical margins. Non-eugenol containing cement should be used to cement the cast restoration to composite cores (Charbeneau, 1989). The elastic modulus of some dental materials, such as nano-filled composite resins, can result in low internal stress (Ilie et al., 2009). Dual-polymerization resin cement has a higher modulus of elasticity compared with conventional and self-etched cements, when photo-polymerized (Saskalauskaite et al., 2008; Meng et al., 2008).

2.2.3.3e. Strength:

This will be discussed in further detail in the tensile bond strength part of this study.

2.2.3.4. Biological properties of composite materials:

Un-polymerized composite resin materials at the floor of a cavity can induce long-term pulpal inflammation (Phillips, 1991).

2.2.3.5. Clinical considerations with the use of composite resin cores:

In a clinical study, Bausch et al., (1982) discussed how polymerization shrinkage occurred during the hardening process of activated composite resin. In chemically activated composite resin, the shrinkage is directed to the centre, but in photo-activated materials it contracts toward the light source. Light polymerized composite resins have low thermal conductivity, and they are available in colours that contrast with tooth tissue (Charbeneau, 1989). An observational clinical study carried out by Opdam et al. (2007) on the longevity of composite resin materials in comparison with silver amalgam reported no significant effect of operator, material as well as combination of material and operator was found.

2.3. Ceramics:

All-ceramic restorations with a ceramic coping are currently in high demand for aesthetic dentistry, and this type of ceramic restoration commonly requires surface conditioning to increase its retention to the tooth surface. Dental ceramics (i.e. metal-ceramic and all-ceramic crowns) have become the most widely used material for the construction of crowns in dentistry because of its excellent aesthetic properties especially so when it comes to its natural appearance (Garber et al., 1988).

McCabe and Walls (1998) mentioned that ceramics is a very rigid, hard, and brittle material whose strength is reduced by the presence of surface irregularities or initial voids and porosities.

2.3.1. In-Ceram coping material:

The In-Ceram with slip-casting technique produces a high strength coping material (Mclean, 2001). This property has led to all-ceramic crowns becoming popular. There is, however, one problem that can lead to failure of all-ceramic restorations and that is cracking of the ceramics (Lawn et al., 2002) and hygroscopic expansion of compomer materials under ceramic crowns (Sindel et al., 1999). In-Ceram with alumina core has suitable physical properties for clinical use as an all-ceramic crown system (Oh et al., 2007).

2.3.2. Ceramic surface treatment:

The first description for ceramic acid etching was in 1975 (Russell and Meiers, 1994). The etching was used for repair of failed all-ceramic restorations because complete removal of a fractured restoration is unpleasant and expensive for the patients (Frankenberger et al., 2000). The aim of acid etching of a ceramic surface is to produce micromechanical retention and enhance its bond to the luting cement (Russell and Meiers, 1994). Hydrofluoric acid was used as ceramic surface treatment for failed ceramic restorations with many protective means for the surrounding tissues (Gau and Krause, 1973).

De-Melo et al. (2007) mentioned that hydrofluoric acid etching or a tribochemical silica coating surface treatment, when used for leucite reinforced ceramics, produced the same microtensile bond strength results.

Airborne-particle abrasion of contaminated zirconia ceramic is more effective than alcohol cleaning. The airborne-particle abrasion for the zirconia ceramic surface gives high bond strength results at either short or long periods of incubation (Quaas et al., 2007) but airborne-particle abrasion may cause a damage on the cementation surface with zirconia-based restorations (Guess et al., 2010) Aboushelib et al., (2008) found that the infiltration etching technique is a reliable method for establishing a strong and durable bond with zirconia-based materials.

2.3.2.1. Types and properties of surface treatment:

Several studies using different methods of surface treatment, with variance of ceramic coping materials, gave different results of tensile bond strength.

2.3.2.1a. Chemical surface treatment:

Many studies used different concentrations of hydrofluoric acid, e.g., 4%, 4.9%, 8% and 10%. The result was higher bond strength at all concentratrions compared with other surface treatments and not etching the ceramics (different type of ceramic e.g. feldspathic, IPS Empress, CAD/CAM cercon and In-Ceram Alumina and Zirconia) (Peumans et al., 2007; El-Zohairy et al., 2003; Kim et al., 2005; Roulet et al., 1995). In another study, Kamada et al. (1998) measured the bond strength of bonding agents, after treating the Cerec 2 ceramic surface with phosphoric acid. The application of a silane coupling agent produced the highest value. The acid etching with phosphoric acid was not the determining variable with regard to bond strength. Silane coupling agents used with acid-etching on ceramic surfaces may create a bond stronger than the cohesive strength of the ceramics (Lacy et al., 1988).

2.3.2.1b. Mechanical surface treatment:

Mechanical roughening with a fine diamond to a ceramic surface has less effect on the bonding composite resin (Lacy et al., 1988). Sandblasting has the potential effect for both cleaning the ceramic surface and providing mechanical surface preparation for an adhesive bonding (Kato et al., 1996). On the other hand, intraoral sandblasting to repair ceramic restorations like the Cojet® silicate system (i.e., modified aluminium oxide Al_2O_3 particles) has the same efficacy as high-concentration hydrofluoric acid (De-Melo et al., 2007; Frankenberger et al., 2000). The Cojet® system is best, when compared to other types of sandblast systems, although all give approximately similar results (Valandro et al., 2005; Atsu et al., 2006).

2.3.2.1c. Silane coupling agents:

The silane coupling agent was effective in establishing a bond between composite resin and dental ceramics (Lacy et al., 1988). The Silica-silane bond forms at the filler interface of dental composite resins. Multiple layers of silane molecules form a film around the filler particles that is either physically or chemically attached to the filler particles (Söderholm and Shang, 1993).

2.4. Dentine bonding agent:

2.4.1. Introduction:

A bonding agent solves the problem of leakage around dental restorative materials. The mechanism of adhesion is simply a surface attachment. It is usually qualified by specifying that the phenomenon does involve some kind of intermolecular attraction between the luting cement and the substrate (Phillips, 1991). A bonding agent has an effect on the bond strength of luting cement to dentine, e.g., single-bond gives higher bond strength than the conventional bonding agent (Neelima et al., 2008). Water storage also has an effect on bonding agents (e.g,. a total-etch bonding agent has a high effect on water storage) (Abdalla et al., 2007).

2.5. Luting cements:

2.5.1. Introduction:

The luting agent is the mouldable substance to seal joints and cement two substances together (Phillips, 1991). There are two types of luting cements: conventional and resin composite cement (Mitchell et al., 1999). The clinical success of fixed prostheses is heavily dependent on the cementation procedure (Diaz-Arnold et al., 1999).

2.5.2. Types of luting cements:

Figure 2.1 illustrates the types of luting cements.

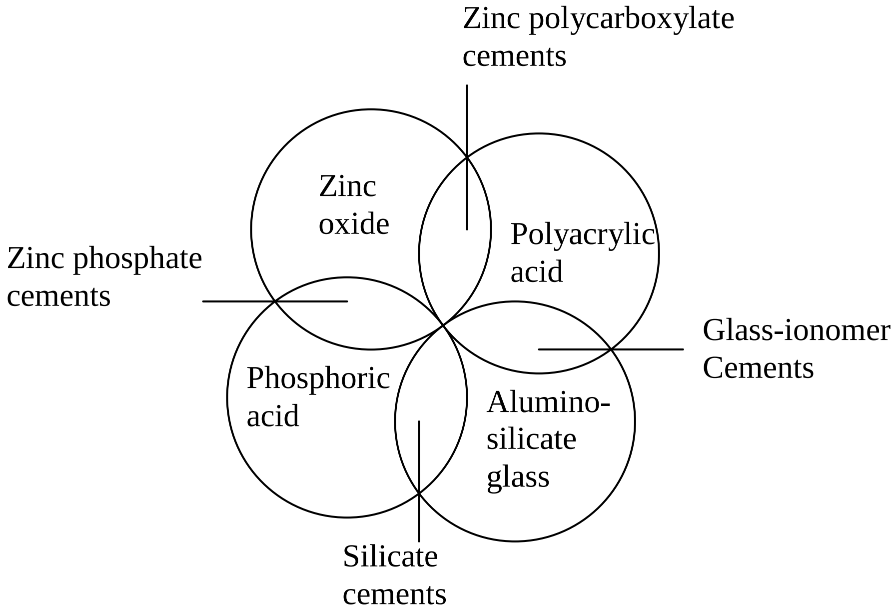

Figure 2.1
Schematics of the various dental cements based on powders of zinc oxide, alumina-silicate glass and liquids consisting of phosphoric acid and polyacrylic acid (Van-Noort, 1994).

2.5.2.1. Zinc phosphate:

Zinc phosphate cement (ZPC) is the oldest luting cement with the longest track record (Phillips, 1991). An increase of the liquid in ZPC mixing influences the setting rate reaction (Diaz-Arnold et al., 1999). Applying resin primer to dentine prior to crown cementation had no effect on its retention compared with a crown that was cemented by using ZPC or resin cement (Swift et al., 1997). ZPC and resin luting cement displayed similar tensile bond strength results when they were used for cementation of pure titanium and gold alloy post-and-core to root dentine (Menani et al., 2008). On the other hand, ZPC displayed higher tensile bond strength value compared with resin cement in a study that compared different post systems (Ertugrul and Ismail, 2005).

2.5.2.2. Glass ionomer cement:

Silicates and silicate-based compounds are frequently used materials in dentistry (Lührs and Geurtsen, 2009). In an *in-vitro* study by Snyder et al. (2003) reported a decrease in the fracture strength after long-term incubation for the glass ionomer cement used for luting an all-ceramic crown.

2.5.2.3. Resin cements:

The use of resin-based luting agents is ever expanding with the development of adhesive dentistry. A multitude of different adhesive systems are used with resin-based luting agents, and new products are introduced to the market frequently. Traditional adhesives generally require a multiple step bonding procedure prior to cementing with active resin-based luting materials.

However, combined agents offer a simple application procedure. Self-etching systems claim that there is no need for the use of a separate adhesive process (Carville and Quinn, 2008).

Cantoro et al., (2008) stored resin cements in a refrigerator then warmed them up to room temperature prior to an *in vitro* study on extracted teeth, in order to increase its bond strength.

2.5.3. Physical and mechanical properties of luting materials:

2.5.3.1. Introduction:

All cements exhibit certain properties during setting. The more important properties are included in ANSI/ADA Specification No. 96 (ISO 9917) for dental water-based cements. A summary of these requirements is given in Table 2.1. Selected mechanical and physical properties of all types of luting cements are listed in Tables 2.2 and 2.3 (Powers and Sakaguchi, 2006).

Table 2.1 Specification requirements for dental water-based cements (Powers and Sakaguchi, 2006)

Cement	Film thickness, maximum (µm)	Net setting time (min)	Compressive strength (MPa)	Acid erosion maximum (mm/h)	Opacity $C_{0.70}$	Acid soluble arsenic content (mg/kg)	Acid soluble load content (mg/kg)
Glass ionomer cement (luting)	25	2.5-8.0	70	0.05	-	2	100
Zinc phosphate (luting)	25	2.5-8.0	70	0.1	-	2	100
Zinc polycarboxylate (luting)	25	2.5-8.0	70	2.0	-	2	100
Glass ionomer (base/liner)	-	2.5-6.0	70	0.05	-	2	100
Zinc phosphate (base/liner)	-	2.5-6.0	70	0.1	-	2	100
Zinc polycarboxylate (base/liner)	-	2.5-6.0	70	2.0	-	2	100
Glass ionomer (restorative)	-	2.5-6.0	130	0.05	0.35 - 0.90	2	100

Modified from ANSI/ADA specification No. 96 for dental water-based cements

Table 2.2 Mechanical properties of luting cements* (Powers and Sakaguchi, 2006)

	Compressive strength (MPa)	**Tensile strength (MPa)**	**Elastic modulus (GPa)**	**Bond strength to dentine (MPa)**
Cement for final cementation				
Adhesive resin	52-224	37-41	1.2-10.7	11-24 with bonding agent
compomer	100	-	3.6	18-24 with bonding agent
Glass ionomer	93-226	4.2-5.3	3.5-6.4	3-5
Hybrid ionomer	85-126	13-24	2.5-7.8	10-12 with bonding agent
Resin composite	180-265	34-37	4.4-6.5	18-30 with bonding agent
Zinc oxide-eugenol (Type II) EBA-alumina	64	6.9	5.4	0
Polymer-modified	37	3.8	2.7	0
Zinc phosphate	96-133	3.1-4.5	9.3-13.4	0
Zinc polycarboxylate	57-99	3.6-6.3	4.0-4.7	2.1
Cement for temporary cementation				
Non-eugenol-zinc oxide	2.7-4.8	0.39-0.94	-	0
Resin composite	25-70	-	-	0
Zinc oxide-eugenol unmodified (Type I)	2.0-14	0.32-2.1	0.22	0

*Properties measured at 24 hours.

Table 2.3 Physical properties of luting cements (Powers and Sakaguchi, 2006)

Cement	Solubility in H_2O (% in 24 hr)	Setting time at 37°C (100% Humidity) (min)	Film thickness (µm)
Compomer	Low	3	-
Glass ionomer	0.4-1.5	6-8	22-24
Hybrid ionomer	0.07-0.40	5.5-6.0	10-22
Resin composite	0.13	4-5	13-20
Zinc oxide-eugenol polymer-modified	0.08	9	25
EBA-alumina	0.02-0.04	7-9	25
Zinc polyacrylate	<0.05	7-9	25-35
Zinc phosphate	0.2 maximum	5-9	25 maximum

2.5.3.2. Film thickness:

When the thickness of the luting material is constant, it will give nominal strength values (Neves et al., 2008). The film thickness depends on the particle size of the powder, the concentration of powder to liquid, the viscosity of the liquid, consistency of the cement and the amount and manner of force applied on the restoration during cementation (Powers and Sakaguchi, 2006).

2.5.3.3. Viscosity:

One of the physical properties a luting agent must exhibit is sufficiently low viscosity to flow along the interfaces between the hard tissue and prosthesis (Anusavice, 2003). In a study by De-Munck et al. (2004), pressure was applied on the prostheses during cementation to ensure that the relatively highly viscous cement intimately adapted to the cavity walls.

2.5.3.4. Setting time:

A sufficient period of time must be available after mixing to seat and finally adapt the margins of the restorations. Adequate working time is expressed by *net setting time*, which as determined by ANSI/ADA specification No. 96 and based on the luting consistency, which is between 2.5 to 8 minutes at a body temperature of 37°C. Light-polymerizing luting cement resolves the problem of limited working time (Powers and Sakaguchi, 2006) (Table 2.3).

2.5.3.5. Strength:

Silicates and silicate-based compounds are used as fillers in different dental restorative materials such as glass-ionomer cements, compomer, composite resins, and adhesive systems. In these materials, the fillers react with acids during the setting process or they improve the mechanical properties by increasing physical resistance, thermal expansion coefficient, and radiopacity in resin restorative materials. They also reduce polymerization shrinkage, and increase aesthetics as well as handling properties. The silicate layer formed in this process is the chemical basis for silane that forms a bond between this layer and the organic composite matrix. It also provides a micromechanical bond between the surface of the material and the composite resin matrix (Lührs and Geurtsen, 2009).

2.5.3.6. Solubility:

Solubility in water and oral fluids is also an important consideration in cement properties. In general, water-based cements are more soluble than resin- or oil-based cements according to ANSI/ADA specification No. 96 (Powers and Sakaguchi, 2006) (Table 2.1).

2.6. Tensile bond strength:

2.6.1. Introduction:

Tensile stress is the ratio of force to the original cross-sectional area perpendicular to the applied force (Anusavice, 2003). Testing the bond strength by tensile loading produced more adhesive failures which may favour the evaluation of true bond strength (El-Zohairy et al., 2003). The thermal expansion behaviour, post-firing shrinkage, interface toughness and roughness, and heating and cooling rates are all factors that must be handled carefully to prevent generation of undesired tensile stresses (Aboushelib et al., 2008). For identifying elementary properties of materials, the tensile test machine was developed. The tensile test machine consists of two parallel positioned springs to ensure the axial loading of the specimen, even if the pull rods of the testing machine should not be absolutely axial (Ilzhöfer et al., 1997). Conventional methods of measurement of tensile strength by using dumbbell-shaped specimens suffer from difficulties of specimen grip eccentricity and surface stress concentrations that are particularly severe with brittle materials (Williams and Smith, 1971).

2.6.2. Tensile stress in the oral cavity:

Tensile strength is the change in length per unit length of a material produced by tensile stress (Craig et al., 2004). In fixed prosthodontic clinics, tensile stress can be produced by bending forces. After applying bending stress, tensile stress will develop on the gingival side of a three-unit fixed partial denture (FPD) and on the occlusal side of a cantilever FPD (Anusavice, 2003).

2.6.3. Factors affecting the tensile bond strength test:

2.6.3.1. Quality of substrates:

2.6.3.1a. Dental tissues:

In Pastor et al., (1997), orthodontic brackets luted to enamel specimens that were acid etched were found to give a higher tensile bond strength than specimens treated with CO_2 laser. The tensile strength of enamel decreased after bleaching, because of alterations in the organic and mineral content of enamel after bleaching (Cavalli et al., 2004).

For dentine, the use of ethylana diamine tetraacetic acid (EDTA) or sodium hypochlorite (NaOCl) surface treatments lead to premature failure of the resin cement bond to dentine (Fuentes et al., 2004). Polar solvents such as acetone and methanol increased the tensile bond strength of dentine (Pashley et al., 2003).

Watanabe et al. (1998) recommended the use of the methacryloxythyl dihydrogen phosphate as a dentine surface treatment to give high bond strength. Dental fluid flow has damaging effects on the adhesive material to dentine surface (Özok et al., 2004). Different grit-sized diamond burs did not affect the mechanical properties of the dentine, except for the ultra-mild one step self-etch adhesive (Ermis et al., 2008).

2.6.3.1b. Dental materials:

Different types of dental materials need different types of surface treatment to increase the tensile strength (Kim et al., 2005). Saliva contamination for dental zirconia ceramics with a phosphate-monomer-resin significantly affected the bond strength. The most effective cleaning method is airborne-particle abrasion (Yang et al., 2008). Peumans et al. (2007) investigated hydrofluoric acid surface treatment for ceramics and found it gave higher mean bond strength than phosphoric acid. Phosphate monomers containing composite resin improve bond strength (Wolfart et al., 2007).

El-Zohairy et al. (2003) reported high tensile bond strength of resin cement with composite resin inlay restorations when compared with ceramic inlay restorations. The processed composite resin (i.e., indirect composite restoration), when luted to dentine by different luting resin cements produced a weak link with intermediate tensile strength values (Mak et al., 2002).

2.6.3.2. Bonded area and luting materials:

The bond strength value of a ceramic/cement combination was significantly lower for the 20 μm film than for thicker films (Molin et al., 1996). The adhesive thickness should remain constant when comparing different joints, as it facilitates the interpretation of nominal strength values (Neves et al., 2008). On the other hand, the application of sustained seating pressure during luting procedures improves the final bond strength of the resin cement (Chieffi et al., 2007).

2.6.3.3. Cross-section:

A study by Sano et al. (1994) tested the relationship between bonded surface area and tensile strength of adhesive materials and reported that using small surface areas decreased the cohesive failure of dentine.

Fowler et al. (1992) studied the effect of different specimen designs and different tensile test jigs, on the bond strength of human and bovine dentine. The result was higher tensile strength values with bovine than with human dentine. On the other hand, Phrukkanon et al.

(1998) found that using small surface areas produced higher bond strengths, and that the cross-sectional shape had little effect.

2.6.3.4. Duration of storage:

2.6.3.4a. Incubation:

Specimens tested in an *in-vitro* study should be incubated in an environment similar to the oral cavity. The incubation period either short-term or for a long-term in distilled water should be at 37˚ C. A short-term incubation period is 24 hours, whereas a long-term incubation period is 6 months. On other hand, to more closely simulate the environment of the oral cavity is recommended thermocycling (ISO/TR 11405). A study by Duarte et al. (2006) used times of incubation 10 min, 24 hours and 12 months, and there was no effect on the tensile result of resin cement when bonded to dentine by low-viscosity composite resins.

2.6.3.4b. Thermocycling:

With thermocycling specimens are storedfor 20 hours to 24 hours at 37˚C. Then the specimens are placed in a thermocycling apparatus with one bath of distilled water at 5˚C and another at 55˚C. The specimens are cycled with 500 exposures to each bath, at least 20 seconds in each bath. The transfer time from one bath to the other should be within 5 to 10 seconds (ISO/TR 1145). Thermocycling caused a reduction in bond strength to ceramics (Ishii et al., 2008; Kato et al., 1996). On the other hand, a resin bonding agent is more stable than silicophosphate cement after thermocycling (Bott and Hannig, 2003). In addition, Lüthy et al. (2006) studied the effect of thermocycling on bond strength. Theresult recorded a high value of bond strength for zirconia luted with Panavia 21 after 10,000 thermo-cycles. The bond strength of universal self-luting cement was higher than conventional luting cement after thermocycling (Abo-Hamar et al., 2005).

2.6.4. Methods for evaluation of quality of tensile test:

2.6.4.1. Mode of failure:

A microscopic inspection of the fractured surfaces can indicate the failure mode of an assembly (Ølio, 1993). Failure modes were categorized as: adhesive failure along the overlay-cement interface; cohesive failure within the resin cement; cohesive failure along the cement-adhesive interface; and adhesive failure along the dentine surface (Mak et al., 2002).

2.6.4.2. Pre-test failure:

Several studies excluded specimens that failed during the sectioning process. This may be incorrect, because there may have been a specific amount of force to produce the failure during sectioning, and this individual force is more than 0 MPa (Nikolaenko et al., 2004). The design ofthe micro-bar can contribute to a high rate of pre-test failure (El-Zohairy et al., 2003). Pre-test heating enhances the bonding potential of some types of luting materials (Cantoro et al., 2008).

High contraction stress and modulus of elasticity causes a more uniform stress distribution at the restorative composite resin-tooth interface. This uniform stress distribution is also evident in a reduced specimen loss during the different stages of specimen preparation (Ilie et al., 2006).

Chapter Three: Materials and Method

3.1. Materials:

In this study two types of conventional luting cement were used: Elite (Zinc phosphate cement) (GC Corporation, Tokyo, Japan) as control, and Fuji I (Glass ionomer cement) (GC Corporation, Tokyo, Japan). Two types of resin luting cement namely Calibra® (Dentsply, Konstanz, Germany), and Panavia™F 2.0 (Kuraray Medical Inc., Okayama, Japan) with Clearfil Silane Kit (Kuraray Medical Inc., Okayama, Japan) were used to lute Vita In-Ceram® Zirconia (Vita Zahnfabrik, Säckingen, Germany) coping material, with two types of composite resin materials, Spectrum®TPH®3 (hybrid composite) (Dentsply, Konstanz, Germany), and Composan Core DC (flowable composite) (Promedica, Neumünster, Germany). All the materials used in this study are listed in Table 3.1 and shown in Figures 3.1-3.8.

Table 3.1 List of materials used in this study

Material	**Product name**	**Manufacturer**	**Batch number**
Zinc phosphate cement	Elite	GC Corporation, Tokyo, Japan	Lot:0708161 ⌛ 2010-08
Glass ionomer cement	Fuji I	GC Corporation, Tokyo, Japan	Lot:0810021 ⌛ 2011-10
Resin luting cement	Calibra®	Dentspty, Konstanz, Germany	Lot:080520 ⌛ 2009-09
Resin luting cement	Panavia™F 2.0	Kuraray Medical Inc., Okayama, Japan	Lot:51622 ⌛ 2010-04
Silane coupling agent	Clearfil Silane Kit	Kuraray Medical Inc., Okayama, Japan	Lot:61165 ⌛ 2010-09
Hybrid composite resin	Spectrum®TPH®3	Dentsplay, Konstanz, Germany	Lot:0809000554 ⌛ 2011-08
Flowable composite resin	Composan Core DC	Promidica, Neumünster, Germany	Lot:0911471 ⌛ 2011-01
Ceramic coping material	Vita In-Ceram® Zirconia	Vita Zahnfabrik, Säckingen, Germany	Lot:24790 ⌛2010-08

Figure 3.1
Zinc phosphate cement (Elite).

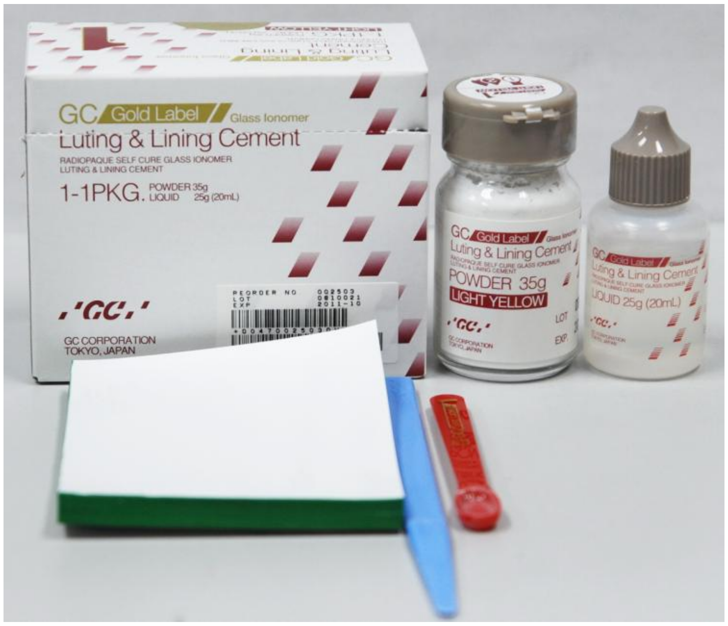

Figure 3.2
Glass ionomer cement (Fuji I).

Figure 3.3
Resin luting cement (Calibra®).

Figure 3.4
Resin luting cement (Panavia™F 2.0).

Figure 3.5
Silane coupling agent (Clearfil).

Figure 3.6
Hybrid composite resin (Spectrum®TPH®3).

Figure 3.7
Flowable composite resin (Composan Core DC).

Figure 3.8
Ceramic coping material (Vita In-Ceram$^{®}$ Zirconia).

3.2. Method:

3.2.1. Mould preparation:

3.2.1.1. Plastic mould preparation:

A plastic mould was designed to form the ceramic specimens. It was designed by the faculty of engineering, University of Malaya. The mould consists of two separate parts fixed together by four screws. The mould contained five separate holes that were created to hold 5 ceramic specimens. The dimensions of each hole were 8 mm in diameter and 5 mm in height as shown in Figure 3.9.

3.2.1.2. Stainless steel mould preparation:

For the composite resin specimen preparation, a stainless steel mould was designed. The upper part of the mould was designed exactly the same as the plastic mould for the ceramic specimens. The measurements for each hole were 6 mm in height and 5 mm in diameter for the first 3 mm height of the composite resin rod, then for the next 3 mm height, it increased gradually until it reached 8 mm in diameter at the top (to fix the composite resin rod inside the tensile jig). The lower part of this mould was prepared from transparent plastic to allow the light to pass through during polymerization as shown in Figure 3.10.

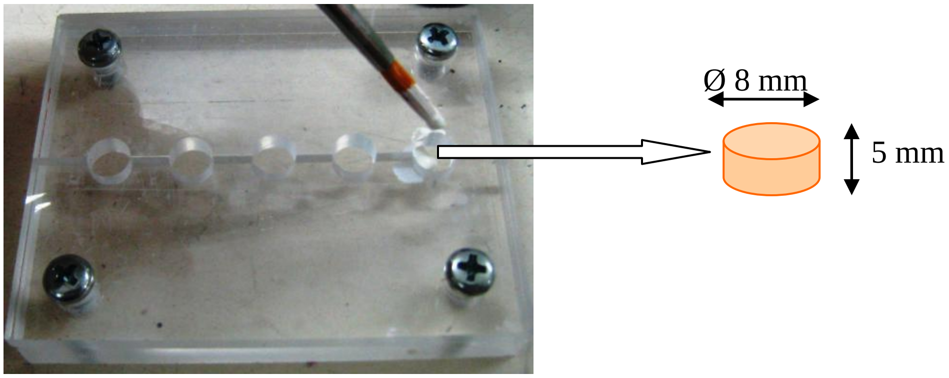

Figure 3.9
Plastic mould with the diagrammatic illustration for ceramic disc (white arrow).

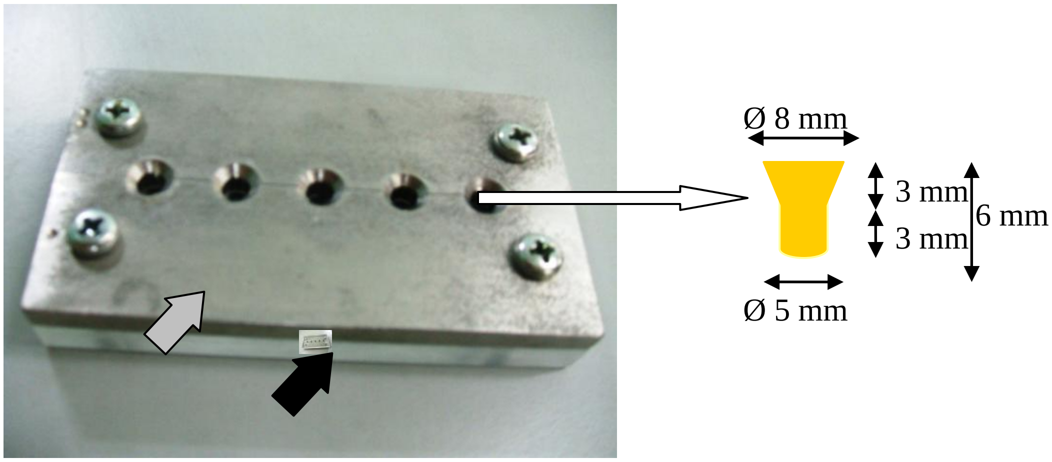

Figure 3.10
Stainless steel mould with metal side (grey arrow); and transparent side of stainless steel mould (black arrow); with the diagrammatic illustration for composite resin rod (white arrow).

3.2.2. Specimens preparation:

3.2.2.1. Ceramic disc preparation (Vita In-Ceram® Zirconia):

The fabrication of Vita In-Ceram® coping discs was accomplished in a dental laboratory*, by following the manufacturer’s instructions of Vita In-Ceram® coping preparation with some modifications:

1. The lower part of the mould was built from Vita In-Ceram® special plaster.
2. The slip preparation (weight- 45g) of Vita In-Ceram® Zirconia powder was weighed.
3. The coping material was prepared by mixing one ampoule of Vita In-Ceram® Alumina/Zirconia mixing liquid and 4 drops of Vita In-Ceram® Zirconia.
4. The mixture was poured into a glass beaker and premixed in the Vitasonic (Ultrasonic mixer for Vita In-Ceram® slip technique).
5. The glass beaker was placed on a vibrator and spatulated.
6. Vita In-Ceram® powder was poured slowly into the liquid in several small portions as shown in Figure 3.11(a).

The glass beaker was placed in chilled water inside the Vitasonic. After the entire amount of powder had been added, the glass beaker was placed in the Vitasonic mixer for 7 minutes as shown in Figures 3.11 (b) and (c).

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*Wilayah Dental Laboratory, Jalan Ramah, Kuala Lumpur, Malaysia.

After the slip exhibited a homogeneous consistency, the mixture was evacuated for 1 minute. The mixed slip was then poured into the enclosed plastic cup. The plastic mould hole was filled with the mixture (Figure 3.11(d)). The holes were filled rapidly and this process was accomplished without interruption, to prevent the outer layer from drying out, which might have led to an onion skin effect. After complete setting, the ceramic disc was carefully taken out from the mould.

After the first sinter firing, the discs were allowed to cool down to 400°C in the closed furnace and then to room temperature after the firing chamber was opened. Since the special plaster plate contacted during firing, the sintered disc could be easily removed.

The disc refining was accomplished in the Vita Inceramat furnace (Figures 3.11 (e), (f)) after the first sintering. In the second firing, the sintered disc was removed from the plaster plate and placed on the firing support. The second sinter firing time is shown in Figure 3.11 (j). After the second sinter firing, the discs were allowed to cool down to 400°C in a closed furnace. They were then allowed to cool to room temperature after the firing chamber was opened.

Vita In-Ceram® Zirconia glass powder was mixed with distilled water until it reached a thin consistency. A brush was used to apply a coat to surfaces of the sintered disc (Figures 3.11 (g) and (h)).

During glass infiltration the ceramic discs were laid on a sheet of platinum foil. The basal surface of the disc had not been coated with glass powder during the infiltration firing process, allowing the air to escape from the disc, to ensure complete glass infiltration. The glass-coated discs were placed on a sheet of platinum foil with a thickness of 0.1mm to perform the glass infiltration firing (Figure 3.11 (i)).

The discs with glass infiltration firing time’s programme of a Vita Inceramat are shown in Figure 3.11 (j). Just in case the infiltration was not completed, the infiltration process was repeated. The excess glass was removed by a coarse-grained diamond instrument (Vita Zahnfabrik, Säckingen, Germany). Then, the disc was airborne particle abraded with 50μm aluminium oxide at a maximum pressure of approximately 4 bar to remove excess glass (Vita Zahnfabrik, Säckingen, Germany).

Finally, each disc was placed on a fibrous pad (Vita Zahnfabrik, Säckingen, Germany) and glass control firing was accomplished according to the programme as shown in Figure 3.11 (j). The diagrammatic illustration of the ceramic disc preparation is shown in Figure 3.11 (j).

Figure 3.11 (a)
Vita In-Ceram® Zirconia powder, liquid and additive.

Figure 3.11 (b)
Vitasonic machine.

Figure 3.11 (c)
Mixing Vita In-Ceram® Zirconia mixture.

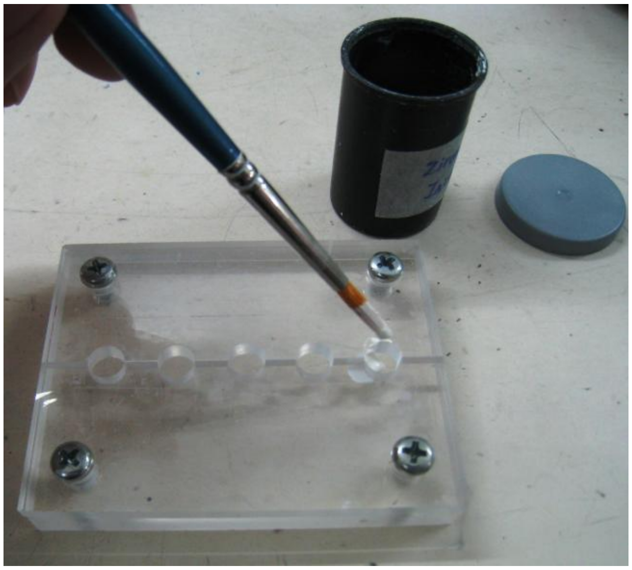


Figure 3.11 (d)
Vita In-Ceram® Zirconia mixture was poured into plastic mould.

Figure 3.11 (e)
Firing to sinter disc.

Figure 3.11 (f)
Vita Inceramat (furnace).

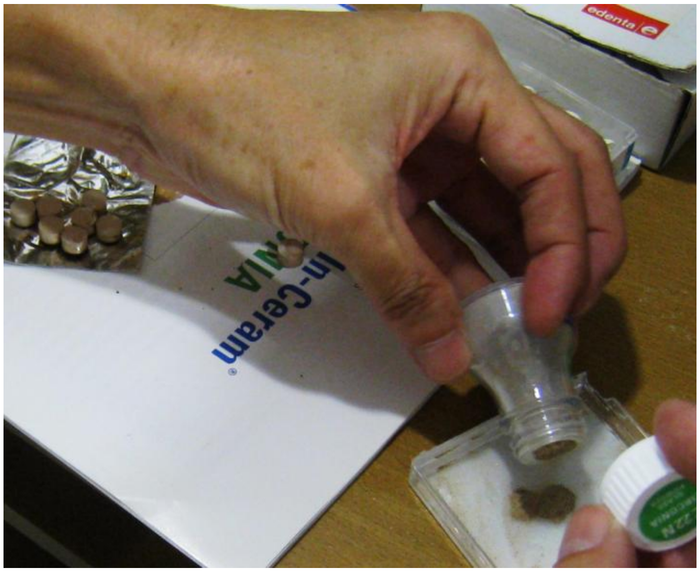
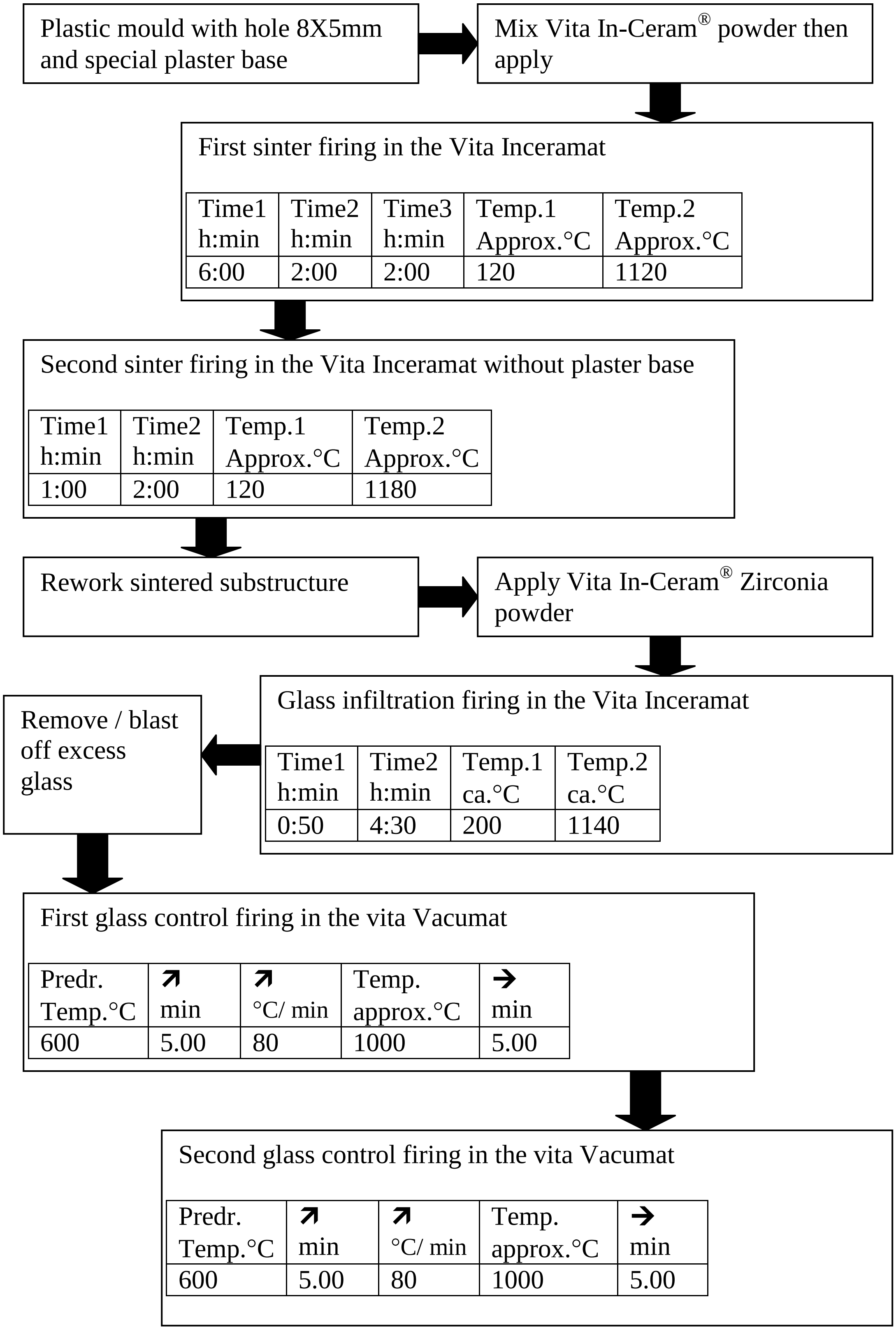


Figure 3.11 (g)
Mixing glass powder.

Figure 3.11 (h)
Applying glass powder on the ceramic disc.

Figure 3.11 (i)
Final shape of ceramic disc.



Plastic mould with hole 8X5mm and special plaster base

Mix Vita In-Ceram® powder then apply

First sinter firing in the Vita Inceramat

Time1 h:min	Time2 h:min	Time3 h:min	Temp.1 Approx.°C	Temp.2 Approx.°C
6:00	2:00	2:00	120	1120

Second sinter firing in the Vita Inceramat without plaster base

Time1 h:min	Time2 h:min	Temp.1 Approx.°C	Temp.2 Approx.°C
1:00	2:00	120	1180

Rework sintered substructure

Apply Vita In-Ceram® Zirconia powder

Glass infiltration firing in the Vita Inceramat

Time1 h:min	Time2 h:min	Temp.1 ca.°C	Temp.2 ca.°C
0:50	4:30	200	1140

Remove / blast off excess glass

First glass control firing in the vita Vacumat

Predr. Temp.°C	↗ min	↗ °C/ min	Temp. approx.°C	→ min
600	5.00	80	1000	5.00

Second glass control firing in the vita Vacumat

Predr. Temp.°C	↗ min	↗ °C/ min	Temp. approx.°C	→ min
600	5.00	80	1000	5.00

Figure 3.11 (j)
Diagrammatic illustration of the ceramic specimen preparation with sintring programme (Vita Zahnfabrik, Säckingen, Germany).

3.2.2.2. Composite resin rod preparation:

3.2.2.2a. Spectrum®TPH®3:

The stainless steel mould was cleaned and dried prior to the preparation of the composite resin rod. The first composite resin prepared in the mould was Spectrum® TPH®3 shade A3. A radiopaque sub-micron hybrid composite was activated by visible-light (Figure 3.12 (a)). The depth of polymerization for shade A3 used in this study was 3mm for 20 seconds from each side.

3.2.2.2b. Composan core DC:

The same stainless steel mould was used in preparing the composite resin specimen by using Composan DC dentine shade (Figure 3.12 (b)). The material was mixed automatically with the correct ratio, in a mixing tip and polymerized for 20 seconds.

Figure 3.12 (a)
Light polymerization unit.

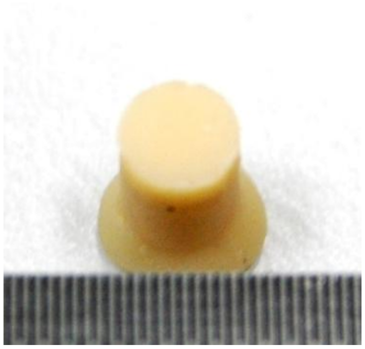


Figure 3.12 (b)
Composite resin specimen
after polymerization.

3.2.3. Incubation of composite resin specimens:

The incubation period for the composite resin specimens was 37°C for 24 h in distilled water, referring to ISO 11450.

3.2.4. Airborne-particle abrading ceramic disc by aluminium oxide:

The ceramic disc was airborne-particle abraded by using 50 µm aluminium oxide for a maximal pressure of approximately 4 bars to increase the micro-undercut as shown in Figure 3.13.

Figure 3.13
Airborne-particle abrasion machine.

3.2.5. Cementation:

Figures 3.14 (a), (b), and (c) display the specimens before, during, and after cementation respectively.

3.2.5.1. Luting cement mixing technique:

3.2.5.1a. Elite (ZPC as control):

For accurate dispensing of powder, the bottle was lightly tapped against the hand. Shaking the liquid bottle is undesirable. The bottle was held vertically, squeezed gently, then, closed after use. The powder and liquid ratio 1:3 (one level No.3 scoop with three drops of liquid) was dispensed onto a heavy glass slab. A stainless steel spatula was used to divide the powder into 3 equal portions. One portion of powder was incorporated into all the liquid and mixed thoroughly for 10 seconds. The total process took 60-90 seconds.

3.2.5.1b. Fuji I (GIC):

For accurate dispensing of powder, the bottle was lightly tapped against the hand. The liquid bottle was held vertically, squeezed gently and closed immediately after use. Powder and liquid ratio 1:2 (one level scoop of powder to 2 drops of liquid) was dispensed onto the pad. A plastic spatula was used to mix all the powder with the liquid. This cement was mixed rapidly for 20 seconds.

3.2.5.1c. Calibra® (resin cement):

The ceramic disc surface was cleaned thoroughly with a water spray and air drier. Calibra® silane coupling agent was applied to the ceramic disc according to the manufacturer's instructions. A single coat of Prime and Bond Adhesive was then applied to the ceramic and composite resin discs. The adhesive was immediately air dried for 5 seconds. A blue light was applied for 10 seconds for polymerization. The Calibra® base was applied directly onto the ceramic disc. The composite resin rod was seated on the ceramic disc slowly. The ceramic disc was tacked in place by briefly light polymerizing around the cemented area only, for no more than 10 seconds. Excess cement was lifted off around the margins. All the cemented areas were light polymerized for 20 seconds.

3.2.5.1d. Panavia™F 2.0 (resin cement):

The ceramic disc surface was rinsed with water and dried with an air syringe. The ceramic surface was silanated by using the Clearfil Silane Kit. Clearfil silane coupling agent was applied on ceramic disc, by using one drop of each of liquid A and liquid B, and dispensed onto a mixing dish. ED Primer was applied with a disposable brush tip to the ceramic surface and left in place for 60 seconds. The excess primer was removed by using a sponge to prevent pooling of the primer on the ceramic surface. Gentle air flow was used to dry the primer completely. A pooling of the primer must be avoid because it may cause a quick polymerization of the resin cement. A vacuum was used for drying to prevent the primer from splattering.

The mixed paste was then applied to the ceramic disc and cemented to the composite resin disc. Cementation was completed within 40 seconds. The resin cement was light polymerized for 10 seconds before the excess cement was removed.

Then the mixed paste was polymerized along the cement line for 20 seconds. If a large surface has to be light polymerized, it is advisable to divide the area into several sections before light polymerizing each section. Excess cement after polymerization was removed by polisher.

3.2.5.2. Load application:

A 5 kg load (Makramani load) was applied on cemented specimens for 5 minutes (Figure 3.14 (b)).

Figure 3.14 (a)
Ceramic and composite resin specimens before cementation.

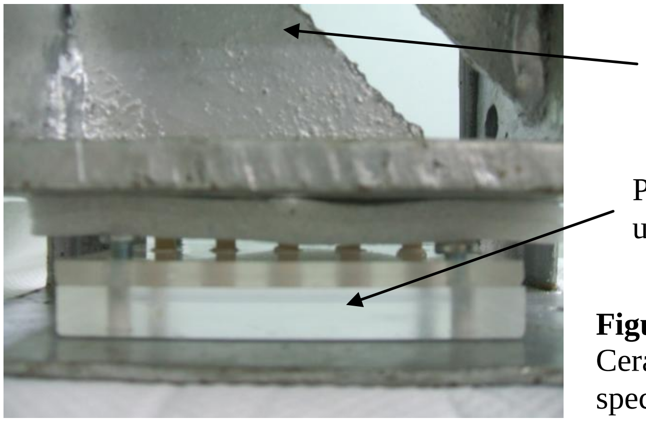


Figure 3.14 (b)
Ceramic and composite resin specimens during cementation.

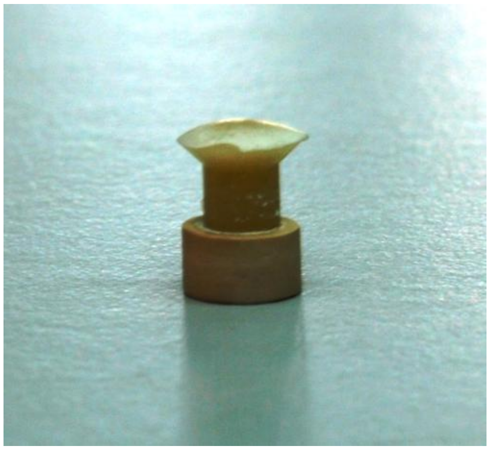


Figure 3.14 (c)
Ceramic and composite resin specimens after cementation.

3.2.6. Incubation of specimens after cementation:

After cementation, the specimens were incubated in distilled water at 37°C for 24h.

3.2.7. Testing of specimens:

3.2.7.1. Modified tensile jig (MJ):

3.2.7.1a. Upper part:

The modified tensile jig was set up to facilitate griping of the upper holder. It also allowed circular rotation. The ceramic part of specimen was fit inside the upper holder and held by using an aluminium retainer. The ceramic specimen holder was made of PVC, 25 mm in diameter and 40 mm in height. The lower part of that holder had a centre hole measuring 8 mm in diameter and 3.5 mm in height as shown in Figure 3.15.

3.2.7.1b. Lower part:

The lower part of the modified jig was a fixed part. It was used to hold the composite resin rod of specimens with the aid of an aluminium retainer as shown in the diagrammatic illustration, Figure 3.15. The composite resin specimen was griped inside the hole by an aluminium retainer. This holder was 15.8 mm in diameter and 25 mm in height. This was designed to fit inside the universal testing machine as is shown in Figures 3.16 (a) and 3.16 (b).

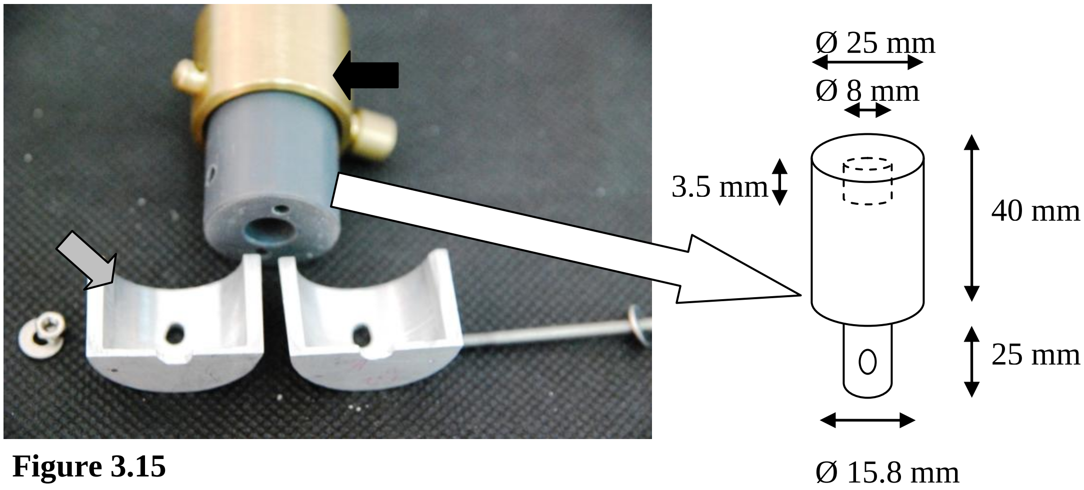


Figure 3.15
Plastic holder for ceramic and composite resin specimens with diagrammatical illustration (white arrow); the modified jig (black arrow); and aluminium retainer (gray arrow).

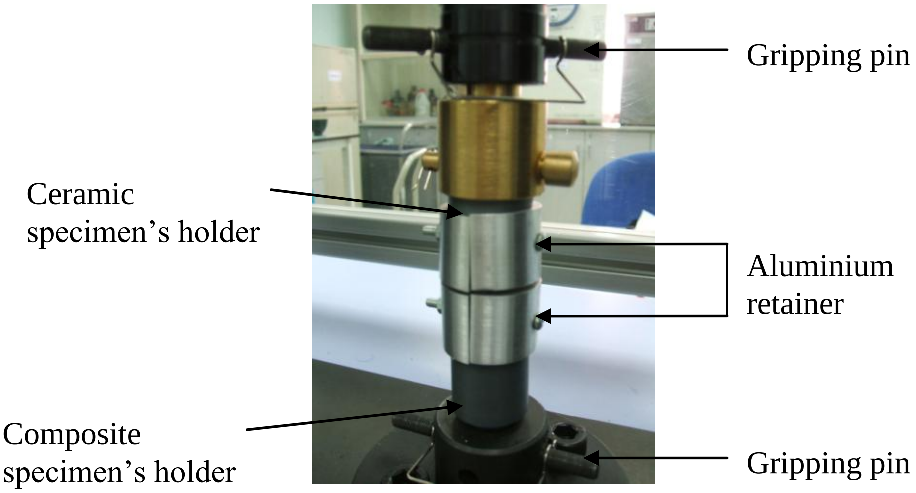


Figure 3.16 (a)
Modified tensile jig.

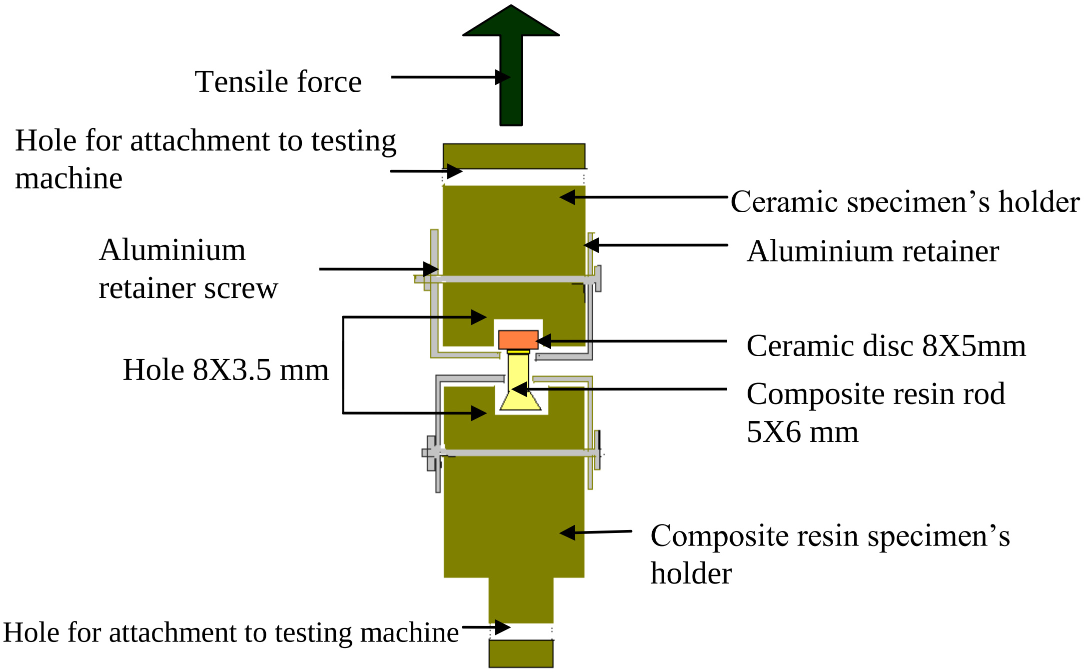


Figure 3.16 (b)
Diagrammatic illustration for modified tensile jig.

3.2.8. Tensile force application:

After 24 h of water storage, the tensile bond strength was tested by using a universal testing machine (Autograph AG-X series, Shimadzu, Precision Universal Tester; Japan) at a cross head speed of 0.5 mm/min (Figure 3.17). The ceramic part of the specimen was fixed in the upper holder and gripped by an aluminium retainer. Then, the composite resin part of the specimen was inserted in the lower part of the lower holder, and fixed by an aluminium retainer.

3.2.9. Data collection:

Data were collected by using TrabeziumX software that was supplied with the universal testing machine (Autograph AG-X series, Shimadzu, Precision Universal Tester, Japan) (Figure 3.18).

3.2.10. Statistical analysis:

The data were analysed by using SPSS 12.0 statistical software.

3.2.11. Mode of failure:

To determine the mode of failure, all specimens were observed the next day after fracture under a stereomicroscope (Olympus Corporation, Tokyo, Japan) (Figure 3.19). The type of failure was based on the percentage of luting materials remaining on the ceramic surface (Figure 3.20). If it was more than 75%, the mode of failure was considered to be cohesive (the surfaces of both adherents after debonding will be covered by fractured luting material). If it was more than 25%, and less than 75% the failure was mixed (characterised by a certain percentage of adhesive and cohesive areas). If the amount of cement was less than 25% the failure was considered to be adhesive (debonding occurs between luting material and the adherent). If the composite resin was fractured, the failure was considered cohesive in composite resin. The percentage of remaining luting material on the ceramic surface was obtained from a series of steps in the software supplied with the Olympus stereomicroscope (Olympus Corporation, Tokyo, Japan). Firstly, photographs of each ceramic disc were captured after failure. This procedure was accomplished with a camera supplied with the Olympus stereomicroscope and connected to a computer and processed by software (DigiAcquis 2.0) (Software supplied with Olympus stereomicroscope). Secondly, the surface area of luting material on each photograph was measured by using CellˆD* software. The software then determined the surface area, by drawing a boundary for the remaining luting materials on the ceramic disc. Finally, the surface area of the specimens was converted to percentage by using Microsoft Excel programme. All stages are summarized in Figure 3.21.

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*Analysis Image processing supplied with Olympus microscope (Olympus high resolution microscope model BX51 TRF Tokyo, Japan). See Appendix I page 98.

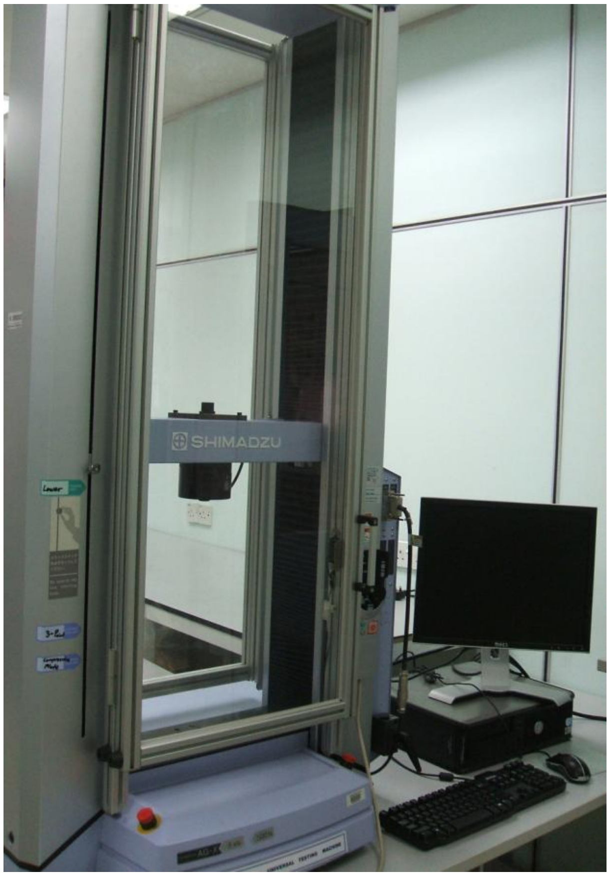
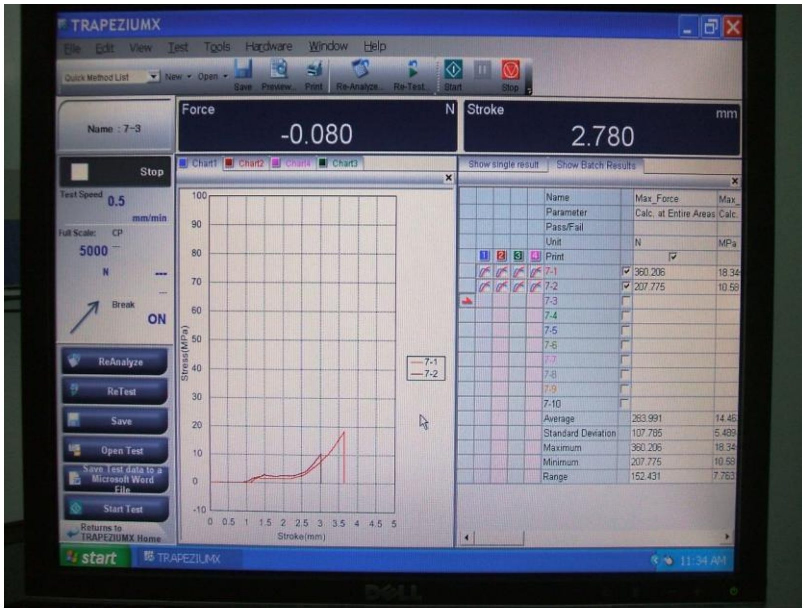


Figure 3.17
Universal testing machine.



Name : 7-3

Force -0.080 N

Stroke 2.780 mm

Stop

Test Speed 0.5 mm/min

Full Scale: CP 5000 N

Break ON

Name		Max_Force	Max_
Parameter		Calc. at Entire Areas	Calc.
Pass/Fail			
Unit		N	MPa
Print		☑	
7-1	☑	360.206	18.34
7-2	☑	207.775	10.58
7-3	☐		
7-4	☐		
7-5	☐		
7-6	☐		
7-7	☐		
7-8	☐		
7-9	☐		
7-10	☐		
Average		283.991	14.46
Standard Deviation		107.785	5.489
Maximum		360.206	18.34
Minimum		207.775	10.58
Range		152.431	7.763

Figure 3.18
TrabeziumX.

Figure 3.19
Stereomicroscope.

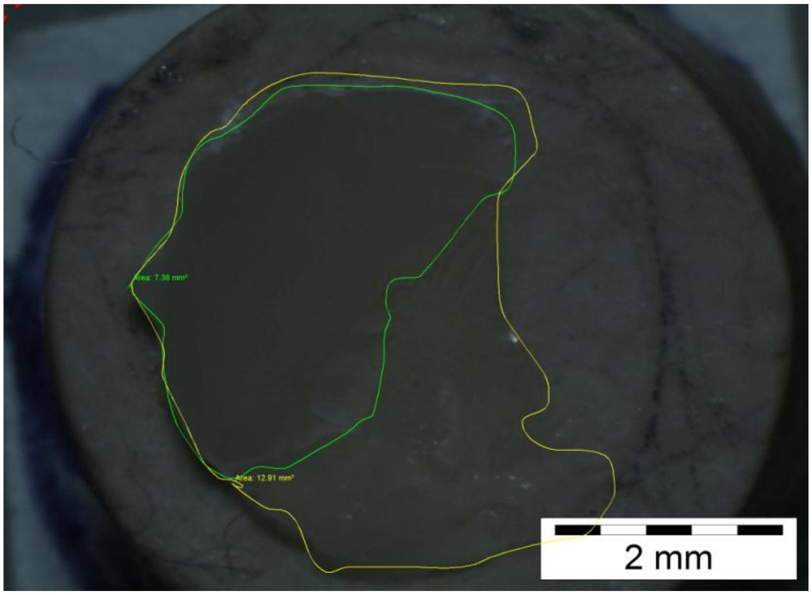


Figure 3.20
Ceramic specimen after tensile test.

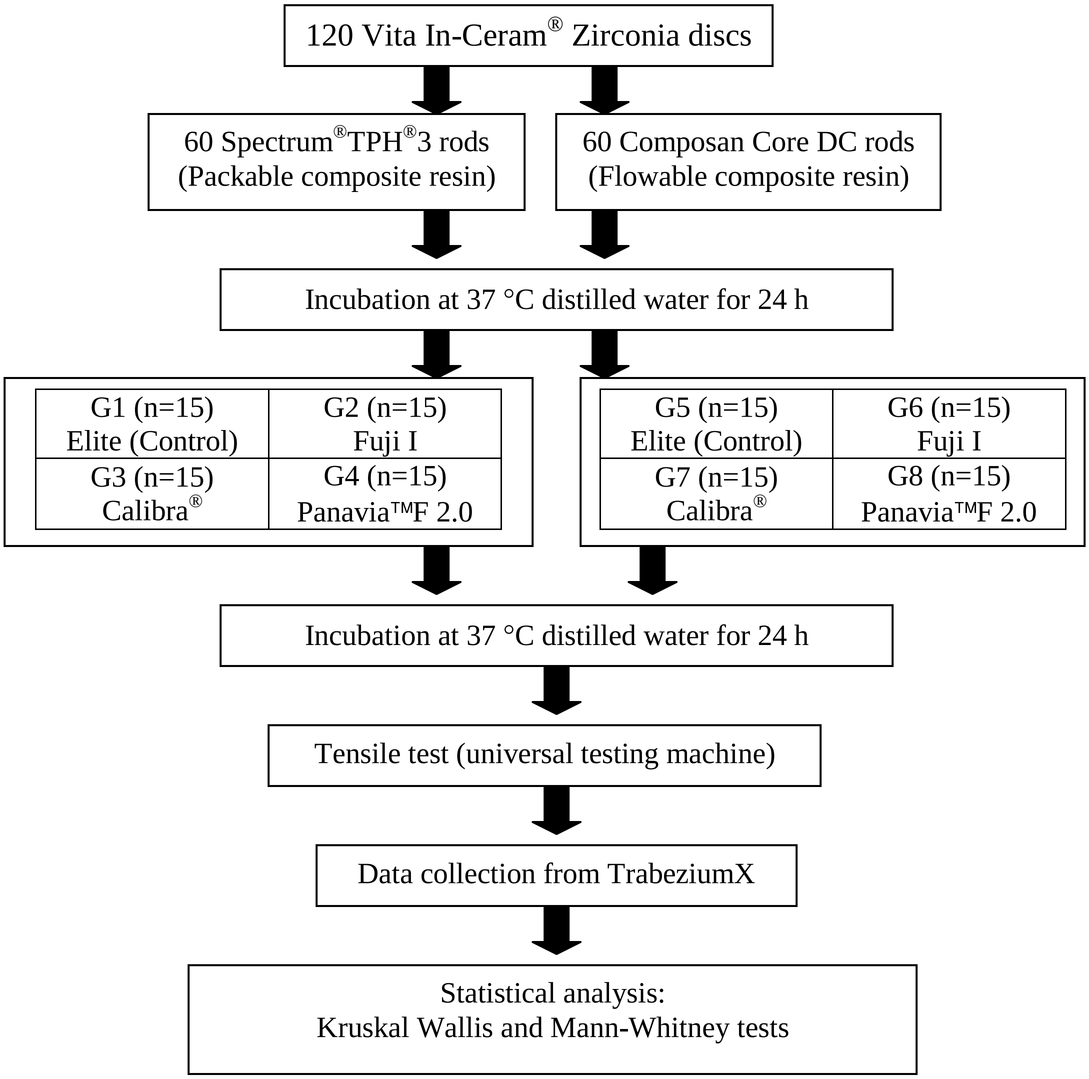


Figure 3.21
Diagrammatic illustration for the method.

Chapter Four:
Results

4.1. Mean values of TBS test:

The arrangement of groups in this study is shown in Table 4.1.

Table 4.1 Testing groups.

Vita In-Ceram® Zirconia								
Composite	**Spectrum®TPH®3**				**Composan Core DC**			
Cement	Elite	Fuji I	Calibra	Panavia	Elite	Fuji I	Calibra	Panavia
Group no.	G1	G2	G3	G4	G5	G6	G7	G8

The results are grouped and summarized in Table 4.2. The table displays the mean and standard deviation of the eight tested groups.

The mean and standard deviation are useful ways to describe a set of scores. The scores that appear vary in values. The frequencies for all specimens that appear are not normally distributed as shown in Figure 4.1.

The data in this study were subjected to homogeneity test (Levene's test). The result of Levene's test ($p < 0.05$) is shown in Table 4.3. This value must be greater than 0.05. This suggests that tensile bond strength values across groups are not equal.

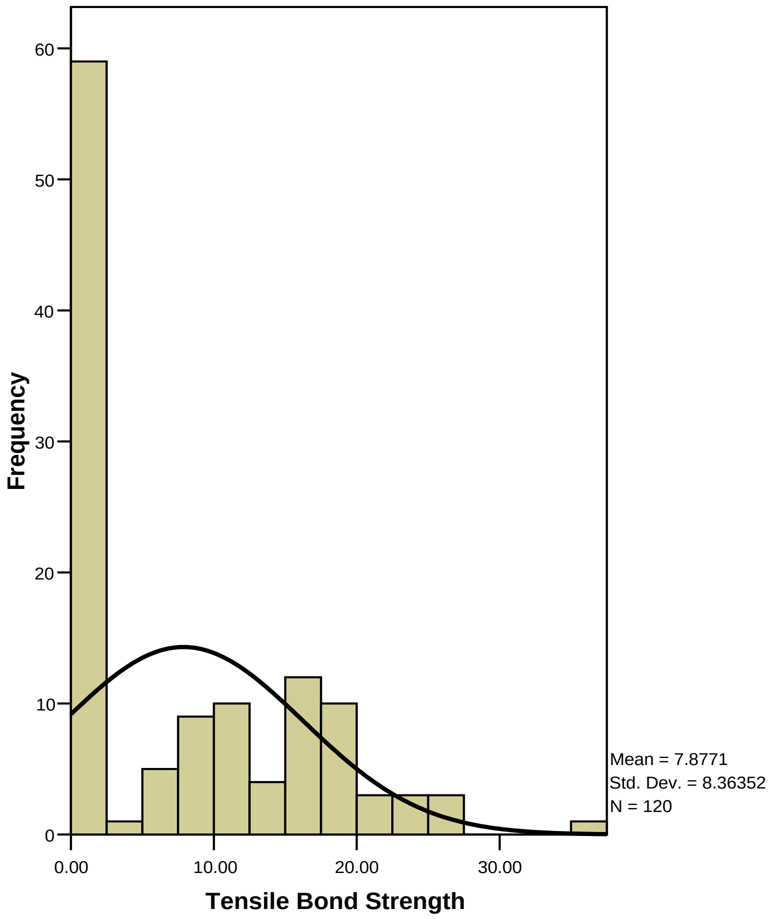
Table 4.2 Tensile bond strength of ceramic disc luted to composite resin cores by using different luting materials.

Composite	Cement	n	Minimum value (MPa)	Maximum value (MPa)	Mean	SD
Spectrum	**Elite**	15	0.01	0.97	0.4452	0.25684
	Fuji I	15	0.01	2.87	1.3292	0.86546
	Calibra®	15	5.80	19.39	13.7727	3.86550
	Panavia™	15	6.77	22.57	12.7543	4.37564
Composan Core DC	**Elite**	15	0.03	0.98	0.2231	0.22907
	Fuji I	15	0.12	1.71	0.9362	0.38125
	Calibra®	15	15.24	35.35	21.8747	5.43992
	Panavia™	15	5.19	19.68	11.6812	4.71255

Table 4.3 Levene's test.

F	df1	df2	Sig.*
14.582	7	112	<0.001

**The groups were not homogenous* $p > 0.05$.



MPa

Figure 4.1
Histogram displays lack of normal distribution of means and standard deviations for all test groups.

4.2. Statistical analysis:

After the data were subjected to SPSS, the values were not normally distributed and not homogenous. The non-parametric Mann-Whitney and Kruskal-Wallis were the statistical tests of choice. The descriptive values were calculated in median and inter-quartile range (IQR) as shown in Table 4.4.

Table 4.4 Median and IQR of TBS of luting materials for all experimental groups.

Groups	**Median (IQR) of TBS (MPa)**	
	Spectrum Groups	**Composan Groups**
Elite (Control)	0.39(0.27)	0.15(0.18)
Fuji I	1.48(1.42)	0.94(0.27)
Calibra	15.27(5.89)	20.35(7.57)
Panavia	11.68(6.74)	11.04(7.21)

4.3. Achievement of study objective statistically:

4.3.1. Effect of composite resin materials on TBS:

Mann-Whitney test was performed to analyse the effect of composite resin core materials and luting materials on TBS.

Mann-Whitney test (Table 4.5) indicated that there was no significant difference between the composite resin materials ($p > 0.05$).

Mann-Whitney was used to show the relationship between the composite resin groups to each other. This result is displayed in Table 4.6. When specimens were cemented with all tested groups, the median of bond strength of both composite resin materials were significantly higher compared with the control groups. Table 4.6 displays the *p* values with specimens cemented by Calibra® and the *p* values with specimens cemented by Fuji I and Panavia™F 2.0.

Table 4.5 Effect of Spectrum®TPH®3 and Composan Core DC on TBS of all luting materials.

Variable	**Spectrum (n=60) Median (IQR) (MPa)**	**Composan (n=60) Median (IQR) (MPa)**	**Z Statistic***	***P* value****
TBS	4.34(12.86)	3.45(17.55)	-0.110	0.912

**Non-Parametric Mann-Whitney U test was applied.*

***Not significantly different between groups (p>0.05).*

Table 4.6 Effect of Spectrum®TPH®3 and Composan Core DC on TBS of all luting materials show the difference between composite materials according to each luting cement.

Variable	**Spectrum (n=15) Median (IQR) (MPa)**	**Composan (n=15) Median (IQR) (MPa)**	**Z Statistic***	***P* value****
TBS of Elite	0.39(0.27)	0.15(0.18)	-2.841	0.004(a)
TBS of Fuji I	1.48(1.42)	0.94(0.27)	-1.265	0.217(b)
TBS of Calibra	15.27(5.89)	20.35(7.57)	-3.754	<0.001(a)
TBS of Panavia	11.68(6.74)	11.04(7.21)	-1.099	0.285(b)

**Non-Parametric Mann-Whitney U test was applied.*
***The significant difference between groups.*
(a)There is a significant difference $p<0.05$.
(b)There is no significant difference $p>0.05$.

4.3.2. Effect of luting materials on TBS:

Kruskal-Wallis test recorded that there was a significant difference among the luting materials regardless of the effect of composite resin materials ($p < 0.05$) (Table 4.7). The same test, shown in Table 4.8, also revealed that TBS was significantly affected by the different luting materials ($p < 0.05$). The post hoc multiple comparisons test with Bonferonni's correction, as shown in Table 4.8, recorded statistically significant differences between control and test groups ($p < 0.05$).

Table 4.7 Effect of luting materials on TBS.

Variable	**Luting cement**	**(n=30) Median (IQR) (MPa)**	**$\chi 2$ Statistic (df)***	***P* value****
TBS	Elite Fuji I Calibra Panavia	0.28(0.33) 0.96(0.97) 17.09(5.61) 11.42(6.94)	98.563(3)	<0.001

**Non-Parametric Kruskal-Wallis test was applied.*
***There is a significant difference p<0.05*

Table 4.8 Comparison of TBS values of different luting materials to each other regardless of composite resin materials.

Variable (TBS)	Luting cements (n=30) Median (IQR) (MPa)	Z Statistic*	*P* value**
Elite **Fuji I**	0.28(0.33) 0.96(0.97)	-4.879	<0.001
Elite **Calibra**	0.28(0.33) 17.09(5.61)	-6.653	<0.001
Elite **Panavia**	0.28(0.33) 11.42(6.94)	-6.653	<0.001
Fuji I **Calibra**	0.96(0.97) 17.09(5.61)	-6.653	<0.001
Fuji I **Panavia**	0.96(0.97) 11.42(6.94)	-6.653	<0.001
Calibra **Panavia**	17.09(5.61) 11.42(6.94)	-3.622	<0.001

**Mann-Whitney U test was applied.*
***There is a highly significant difference between groups p<0.05.*

Kruskal-Wallis test, shown in Table 4.9, recorded that there was a significant difference between the luting materials with Spectrum®TPH®3 regardless of the effect of Composan ($p < 0.05$). This result revealed that TBS was significantly affected by the different luting materials with Spectrum®TPH®3 ($p < 0.05$).

The post hoc multiple comparisons test with Bonferonni's correction, shown in Table 4.10, indicated statistically significant differences between control and test groups ($p < 0.05$).

Table 4.9 Effect of luting materials on TBS with Spectrum®TPH®3.

Variable	**Luting cement**	**(n=15) Median (IQR) (MPa)**	**χ2 Statistic (df)***	***P* value****
TBS with Spectrum	Elite Fuji I Calibra Panavia	0.39(0.27) 1.48(1.42) 15.27(5.89) 11.68(6.74)	46.742(3)	<0.001

**Non-Parametric Kruskal-Wallis test was applied.*

*** There is a highly significant difference p<0.05*

Table 4.10 Comparison of TBS values of different luting materials to each other with Spectrum®TPH®3.

Variable (TBS)	Luting cements (n=15) Median (IQR) (MPa)	Z Statistic*	*P* value
Elite **Fuji I**	0.39(0.27) 1.48(1.42)	-2.924	0.003 (a)
Elite **Calibra**	0.39(0.27) 15.09(5.89)	-4.666	<0.001 (a)
Elite **Panavia**	0.39(0.27) 11.68(6.74)	-4.666	<0.001 (a)
Fuji I **Calibra**	1.48(1.42) 15.09(5.89)	-4.666	<0.001 (a)
Fuji I **Panavia**	1.48(1.42) 11.68(6.74)	-4.666	<0.001 (a)
Calibra **Panavia**	15.09(5.89) 11.68(6.74)	-1.099	0.285 (b)

**Mann-Whitney U test was applied.*
(a) There is a significant difference $p < 0.05$.
(b) There is no significant difference $p > 0.05$.

Kruskal-Wallis test showed that there was a significant difference between the luting materials with Composan Core DC regardless of the effect of Spectrum®TPH®3 ($p < 0.05$) (Table 4.11). This result revealed that TBS was significantly affected by the different luting materials with Composan Core DC ($p < 0.05$).

The post hoc multiple comparisons test with Bonferonni's correction, shown in Table 4.12, indicated statistically significant differences between control and test groups ($p < 0.05$).

Table 4.11 Effect of luting materials on TBS with Composan Core DC.

Variable	**Luting cement**	**(n=15) Median (IQR) (MPa)**	**$\chi 2$ Statistic (df)***	***P* value****
TBS with Composan	Elite Fuji I Calibra Panavia	0.15(0.18) 0.94(0.27) 20.35(7.57) 11.04(7.21)	52.153(3)	<0.001

** Non-Parametric Kruskal-Wallis test was applied.*
*** There is a significant difference p<0.05*

Table 4.12 Comparison of TBS values of different luting materials to each other with Composan Core DC.

Variable (TBS)	Luting cements (n=15) Median (IQR) (MPa)	Z Statistic*	*P* value**
Elite **Fuji I**	0.15(0.18) 0.94(0.27)	-3.961	<0.001
Elite **Calibra**	0.15(0.18) 20.35(7.57)	-4.666	<0.001
Elite **Panavia**	0.15(0.18) 11.04(7.21)	-4.666	<0.001
Fuji I **Calibra**	0.94(0.27) 20.35(7.57)	-4.666	<0.001
Fuji I **Panavia**	0.94(0.27) 11.04(7.21)	-4.666	<0.001
Calibra **Panavia**	20.35(7.57) 11.04(7.21)	-3.920	<0.001

**Mann-Whitney U test was applied.*

***There is a highly significant difference between groups $p<0.05$.*

4.4. Testing mode of failure:

The percentage of mode of failure was measured as described in the last chapter. Cohesive failure of luting material occurred in most cases as shown in Figure 4.2.

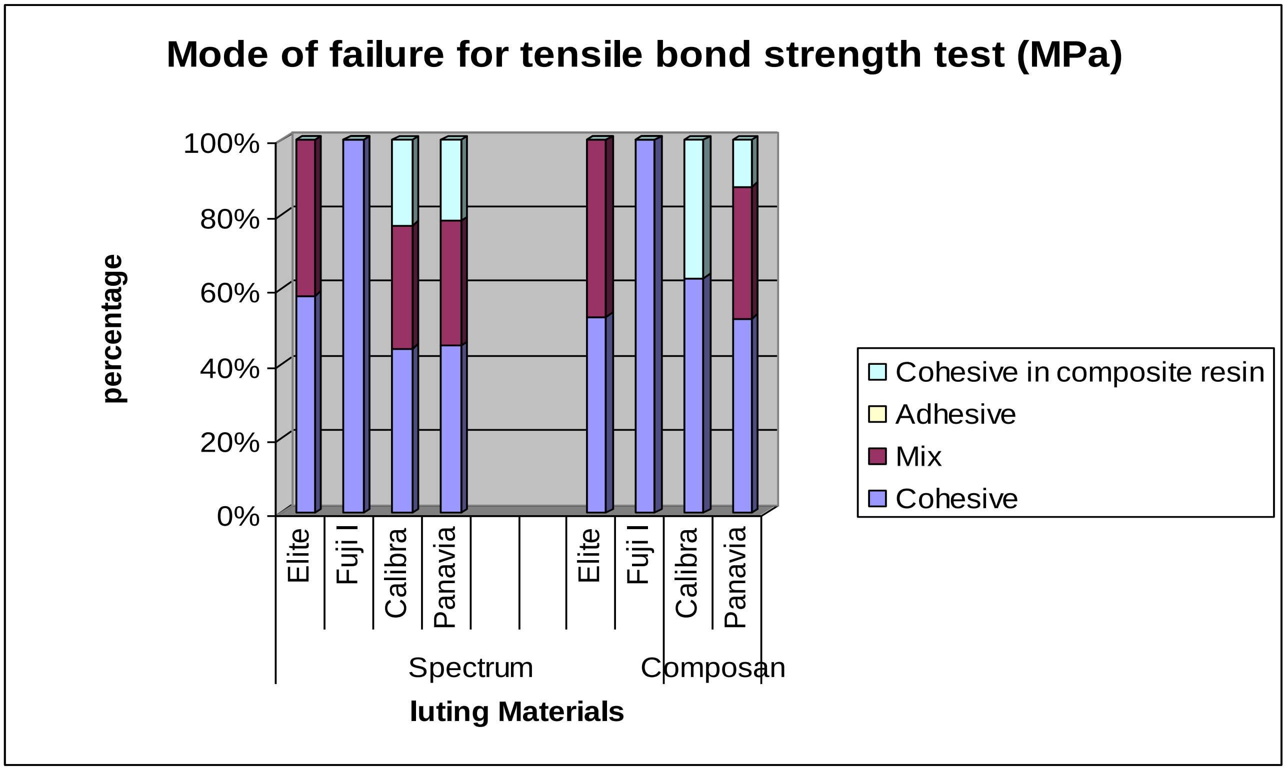


Figure 4.2
Mode of failure of tensile bond strength test of ceramic disc luted to composite resin cores by using different luting materials.

Chapter five:
Discussion

5.1. Methodology:

5.1.1. Ceramic disc preparation:

In this *in-vitro* study, ceramic discs were used to detect the tensile bond strength (TBS) of ceramics luted to composite resin core materials by using different luting agents. According to studies on mechanical properties of ceramics, zirconia ceramic specimens treated with airborne-particle abrasion and adding silane coupling agent resulted in a high value of tensile bond strength to the composite resin (Kim et al., 2005 and Blatz et al., 2003).

Vita In-Ceram® Zirconia is based on many years of clinical experience with Vita In-Ceram® displaying many advantages such as optimal aesthetics, and excellent biocompatibility, and excellent marginal fit. Other advantages included withstanding high functional stress, no thermal conductivity and standardized dental technical procedures (Vita Zahnfabrik, Säckingen, Germany).

According to Kim et al. (2005), ceramics with zirconia and alumina with different surface treatment has an influence on TBS, and preparing the ceramic coping into a disc facilitates standardized specimens designed for tensile test (Yang et al., 2008; Wolfart et al., 2007; Duart et al., 2006; Kim et al 2005). In the present study, a pilot study was accomplished to evaluate the possibility of cutting the ceramics into microbars. This pilot study resulted in premature failure for all ceramic microbars luted to composite resin (Nikoleanko et al., 2004; El-Zohairy et al., 2003).

Airborne-particle abrasion was used for cleaning and surface conditioning for the ceramic discs. According to Quaas et al. (2007) and Yang et al. (2008) the effective cleaning method for the ceramics is the airborne-particle abrasion. In addition, the manufacturer's instructions of Vita In-Ceram® Zirconia were also the use of airborne-particle abrasion as the surface treatment (Vita Zahnfabrik, Säckingen, Germany).

5.1.2. Composite resin rod preparation:

Rod-shaped composite resin specimens were used in many studies (Yang et al., 2008; Wolfart et al., 2007; Duart et al., 2006; Kim et al 2005; Fowler et al., 1992). In these studies the rod-shaped specimen was formed by using gelatinous tubes or capsules. On the other hand, orthodontic wire or fish swivels were necessary to connect the specimens to the pulling part of the tensile jig in the universal testing machine (UTM) (Figure 5.1) (Kim et al 2005; Fowler et al., 1992).

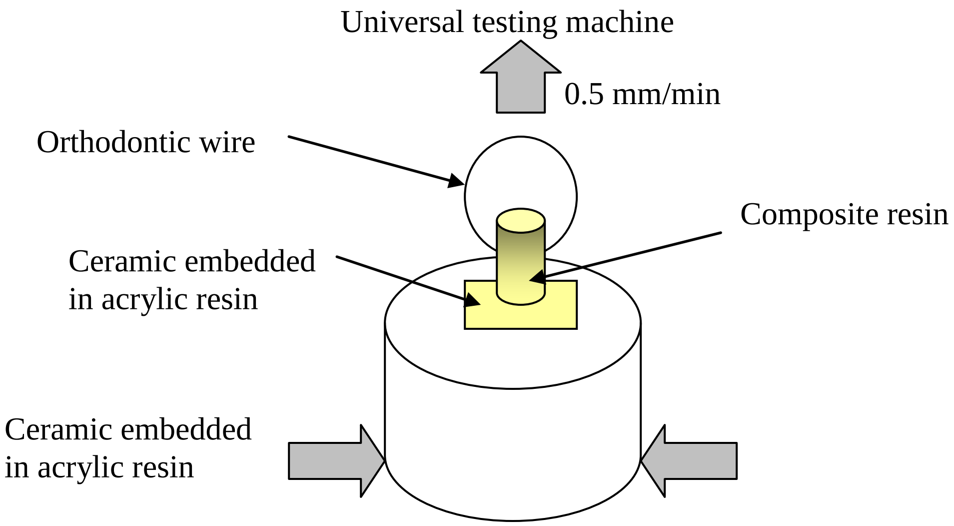


Figure 5.1
Specimen placed in UTM for TBS measurements (Kim et al., 2005).

In the present study, the top part of the rod-shaped composite resin specimen was modified to an inverted cone shape (Menani et al., 2008). Menani et al. (2008) reportedon a specimen was designed with the inverted-cone shape at the top of the post in the core position, in the present study the inverted cone was connected to the composite resin core. The function of this inverted cone was to facilitate gripping of the composite resin rod.

In addition, the use of the stainless steel mould in the present study helped to standardize the composite resin rod formation without any stickiness within the walls of the mould (Cobb et al., 2000). The hole in the stainless steel mould was 6 mm in height with transparent floor that allowed the light polymerization to reach the entire depth of the composite resin rod (Cobb et al., 2000) (Figure 5.2).

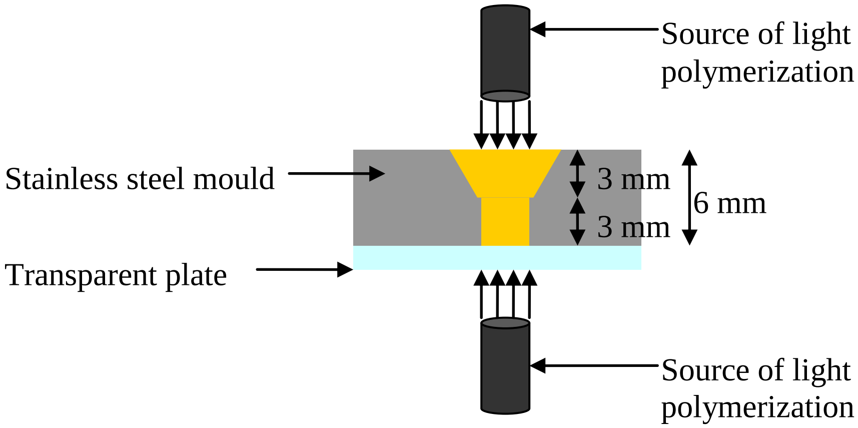


Figure 5.2
Schematic illustration displaying light polymerization for composite resin rod inside stainless steel mould.

5.1.3. Cementation:

In the present study, the cementation of the ceramic disc to the composite resin rod was accomplished following the manufacturer's instructions. The cement was applied directly on the composite resin rod by using a spatula. An even thickness of the luting cement was obtained by placing the specimens during cement setting under the load device (5 kg the weight (Al-makramani weight) that was available in the biomaterials laboratory, Faculty of Dentistry, University of Malaya) (Wolfart et al., 2007; De-Munck et al., 2004).

5.1.4. Incubation:

The minimum period of incubation is 24 hours as described by ISO/TS 11405. The incubation in distilled water at 37°C for 24 hours was to simulate the moisture and temperature in the oral environment. The period of incubation either with thermocycling or without had an effect on the decrease of the TBS of luting area (Wolfart et al., 2007; Lüthy et al., 2006; Kato et al., 1996).

5.1.5. Modified tensile jig:

The modified tensile jig was designed to facilitate gripping of the cylindrical shape specimens that were designed according to ISO/TS 11405. The TBS test for the current study was designed following a combination of specimen designs reported by Fowler et al. (1992). They compared the effect of different specimen designs and different substrates. Their results indicated no statistically significant difference between different TBSwith regard to specimen designs, but there was a significant difference between the substrate groups. Whereas, Phrukkanon et al. (1998) reported that, on small surface areas for TBS specimens higher bond strengths were produced. While Sano et al. (1994) reported that small surface areas of TBS specimens decreased the incidence of cohesive failure of dentine.

5.1.6. Tensile bond strength test:

In the present study the specimens were fitted inside the modified tensile jig, and then the test was accomplished with a cross-head speed of 0.5 mm/min (ISO/TR 11405).

5.2. Results:

5.2.1. Results of TBS of ceramic disc luted to composite resin core materials with different luting agents:

In view of the results reported previously in chapter four, it can be suggested that there is no significant difference between different composite resin core materials. This appears to be an important finding that may help the practitioner to use only tested two composite resin materials type of composite resin core materials.

The findings of the present study cannot be regarded as a follow up of previous studies that evaluated the TBS of different luting materials luted together under different conditions (Ølio, 1993). Other studies tested the influence of post-core systems using different core materials on TBS (Menani et al., 2008; Schmage et al., 2006)s.

However, the present study reported an agreement with previous studies of TBS of luting materials either using cement with the post or crown. In this study, resin cement Calibra® reported higher TBS value than the other cements; this finding was in agreement with Wang et al. (2007). While in Pisani-Proenca et al. (2006), Panavia F 2.0 recorded the highest TBS of all luting materials. The present study was based on recent papers which deal with dental material *in vitro* studies. Zinc phosphate cement (control groups) displayed the lowest TBS results in comparison with the other experimental groups. This finding was in agreement with a study by Mojon et al. (1992) that reported that the zinc phosphate cement was the weakest material, whereas the adhesive resin produced the strongest joints. On the other hand, Ertugrul and Ismail (2005) reported that zinc phosphate cement is the best luting material for cast metal post-and-core. The findings of the present study, the effect of luting materials on TBS was more significant in comparison with the effect of composite resin core materials. These findings are in agreement with the study by Ishii et al. (2008) which concluded that the light cure bonded system seems to have an effect on the core build-up..

In the experimental groups that were using the glass ionomer luting material theresults were very close to control groups.

The cohesive failure in composite resin was observed in the experimental groups cemented with resin-base luting materials. Those results were in agreement with the results of a study by El-Zohairy et al. (2003) who investigated the micro-TBS and failure mode of composite and ceramic CAD/CAM blocks following various surface treatments. They concluded that CAD/CAM restorations fabricated from processed composite blocks may have an advantage over the ceramic blocks related to the higher tensile bond strength with resin cement.

The results might be more significant by adding additional groups to compare the influence of different ceramic surface treatments materials (Peumans et al., 2007). This observation might be explained by the results of Kim et al. (2005) who reported different types of dental materials that need different types of surface treatment or cleaning methods (Yang et al., 2008; Quaas et al., 2007) to increase their tensile bond strength.

5.2.2. Mode of failure:

In the present study, the failure mode displayed that the weakest area was the luting materials (Ølio, 1993). The mode of failure for the present study was classified according to the observation by Mak et al. (2002), who reported the failure modes were categorized as: adhesive failure along the overlay-cement interface; cohesive failure within the resin cement; cohesive failure along the cement-adhesive interface; and adhesive failure along the dentine surface. The mode of failure for the present study displayed cohesive failure of luting materials within all test groups (Figure 4.2 page 71), this finding supports the finding of previous studies (Yang et al., 2008, Wolfart et al., 2007).

On the other hand, frequent forms of modes of failure that were reported in the present study may be the result of surface flaws, internal material flaws in substrate material and the luting material layer or the bonded composite resin and flaws in the interface, as reported in El-Zohairy et al. (2003).

5.6. Limitation of study:

In this *in-vitro* study, the test conditions were different from the intraoral conditions; therefore, it was difficult to relate the results directly to clinical practice. Only one type of coping material was used with a single system of surface treatment. Using different types of core materials such as gold alloy to compare it with the composite resin core materials might be more meaningful.

Chapter Six:
Conclusion

6.1. Conclusion:

(1) Vita In-Ceram® ceramic discs when cemented to composite cores by Calibra® gave high tensile strength value.

(2) The different composite resin core materials used in this study have no significant effect on tensile bond strengths.

6.2. Recommendation for further study:

(1) This study evaluated the TBS of luting materials between two types of composite core materials and Vita In-Ceram® Zirconia coping material. Therefore, further studies are suggested to investigate the TBS of luting materials between other core materials with dentine or other types of ceramic coping materials.

(2) Another study is suggested to evaluate the effect of thermocycling, incubation periods, and surface treatments for ceramics and dentine on TBS.

(3) An *in vitro* study is required to evaluate the other mechanical properties of the same materials used in this study.