Bond strength of metal orthodontic brackets to all ceramic crowns

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Summary

Aim

The aim of this study was to evaluate, in-vitro, the shear bond strength (SBS) and the resultant failure pattern after debonding of metal orthodontic brackets bonded with Transbond^{TM} XT adhesive resin cement and RelyX^{TM} Unicem 2 self-adhesive resin cement to pre-treated (35% ortho-phosphoric acid and silane coupling agent application) IPS eMax and porcelain veneered zirconia crowns.

Material and methodology

A Typhodont maxillary lateral incisor was used and prepared in a conventional manner to receive a full ceramic crown. A CAD (computer aided design)/ CAM (computer aided manufacturing) machine was used to scan the prepared tooth and manufacture 40 IPS eMax crowns and 40 porcelain veneered zirconia crowns. Half the number of IPS eMax crown specimens (ie. 20) and half the number of porcelain veneered zirconia crown specimens (ie. 20) were thermocycled (ie. to mimic thermal changes which occur in the mouth), from 5 to 55^{o} for 500 cycles as recommended by the International Organization for Standardization (ISO 6872, 2008). The remaining 20 IPS eMax crown specimens and 20 porcelain veneered zirconia crown specimens remained new and unexposed to thermal changes. The facial surfaces of all the thermocycled and non-thermocycled crown specimens were then etched. Etching of all the ceramic bonding surfaces was performed by the application of 35 per cent ortho-phosphoric acid liquid for 2 minutes, followed by a thin layer of a ceramic primer. A lateral incisor metal bracket with a bracket base area of 9mm^2 (as confirmed by the manufacturer) was bonded to each of the etched and silane treated ceramic crown specimens and separated in the following manner: ***Group 1:*** (10 thermocycled, etched and silane treated IPS eMax and 10

thermocycled, etched and silane treated porcelain veneered zirconia crown specimens) RelyX™ Unicem 2 self-adhesive resin cement was used to bond the bracket to the ceramic crown specimens, ***Group 2:*** (10 thermocycled, etched and silane treated IPS eMax and 10 thermocycled, etched and silane treated porcelain veneered zirconia crown specimens) Transbond™ XT light cure adhesive primer was first applied onto the bonding surface of the crowns and then Transbond™ XT adhesive resin was used to bond the bracket to the ceramic crown specimens, ***Group 3:*** (10 non-thermocycled, etched and silane treated IPS eMax and 10 non-thermocycled, etched and silane treated porcelain veneered zirconia crown specimens) RelyX™ Unicem 2 self-adhesive resin cement was used to bond the bracket to the ceramic crown specimens, ***Group 4:*** (10 non-thermocycled, etched and silane treated IPS eMax and 10 non-thermocycled, etched and silane treated porcelain veneered zirconia crown specimens) Transbond™ XT light cure adhesive primer was first applied onto the bonding surface of the crowns and then Transbond™ XT adhesive resin cement was used to bond the bracket to the ceramic crown specimens.

After bonding all samples were stored in distilled water for 24 hours before being submitted to the shear bond strength test. Debonding forces in Newtons (N) was determined by using a shear testing machine and converted into Mega Pascals (MPa).

Results

The results after debonding were compared. The mean shear bond strength for RelyX^{TM} Unicem 2 self-adhesive resin cement bonded to the all ceramic non-thermocycled crowns (Group 3) ranged from a low of 5.1 MPa (45.5 Newtons) when brackets were bonded to the IPS eMax crowns to a high of 5.8 MPa (51.9 Newtons) when brackets were bonded to the porcelain veneered zirconia crowns. The mean shear bond strength for Transbond XT adhesive resin cement bonded to the all ceramic non-thermocycled crowns (Group 4) ranged from a low of 6.4 MPa (57.3 Newtons) when brackets

were bonded to the porcelain veneered zirconia crowns to a high of 8.1 MPa (72.7 Newtons) when brackets were bonded to the IPS eMax crowns.

The side by side Box-and-Whisker plots of the shear bond strengths show wide and overlapping dispersions of the crown/adhesive resin combinations which consequently lessen the probability of significant differences between the crown/adhesive resin combinations in all 4 groups. According to the Kruskal-Wallis test ($p < 0.05$), and the Bonferroni Test the non-thermocycled crown/adhesive resin combinations do not differ significantly.

Study of the mean ARI (Adhesive Remnant Index) values for the non-thermocycled crown/adhesive combinations shows that brackets bonded with Rely-X^{TM} Unicem 2 to non-thermocycled porcelain veneered zirconia crowns failed entirely at the ceramic/adhesive interface and for all the other non-thermocycled ceramic/adhesive combinations most of the failures of the bond (70%) occurred at the bracket/adhesive interface, ie. cohesive fractures within the composite resin. No cohesive fractures of the porcelain crowns were noted.

The results of the thermocycled groups (Group 1 and Group 2) show the Transbond^{TM} XT/non-thermocycled IPS eMax crown combination yielded the highest overall mean shear bond strength of 8.1 MPa (72.7 Newtons) but dropped to a mean shear bond strength of 5.1 MPa (46.1 Newtons) (36.4% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The Transbond^{TM} XT/non-thermocycled porcelain veneerd zirconia crown combination yielded the second highest overall mean shear bond strength of 6.4 MPa (57.3 Newtons) and dropped to a mean shear bond strength of 5.1 MPa (45.8 Newtons) (19.3% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The RelyX^{TM} Unicem 2/non-thermocycled porcelain veneered zirconia crown combination yielded the third highest overall mean shear bond strength of 5.8 MPa (51.9 Newtons) but dropped significantly to a mean shear bond strength of 3.2 MPa (29.1 Newtons) (a significant 43.8% drop in shear bond strength) when the crowns were thermocycled

prior to bonding. Lastly, the RelyX^{TM} Unicem 2/non-thermocycled IPS eMax crown combination yielded the fourth highest mean shear bond strength of 5.1MPa (45.5 Newtons) but dropped to a mean shear bond strength of 4.9 MPa (44.5 Newtons) (a drop in shear bond strength of only 3%) when the crowns were thermocyled prior to bonding. Relaxing the significance level (p-value) somewhat demonstrates the negative influence of thermocycling on the shear bond strength of the crown/adhesive combinations.

The non-thermocycled all ceramic crown/adhesive combinations showed mean ARI values of between 1.3 and 2.1 indicating cohesive fractures within the composite resin and efficient bonding of the adhesive material to the porcelain surface. However, all the thermocycled all ceramic crown/adhesive treatment combinations showed mean ARI values of between 0 and 0.8 indicating a bond failure between adhesive and porcelain and highlighting the negative influence of thermocycling on bond strength of both adhesive resin cements.

Conclusion

Within the limitations of this study, it can be concluded that:

1.There was no significant difference in the shear bond strengths of metal orthodontic brackets bonded with RelyX^{TM} Unicem 2 self-adhesive resin cement and metal orthodontic brackets bonded with Transbond^{TM} XT adhesive resin cement to IPS eMax and porcelain-veneered zirconia crowns which were conditioned with 35 % phosphoric acid and a silane coupling agent.

2. Conditioning the porcelain surface with 35% phosphoric acid and a silane coupling agent (which is safer to use than Hydrofluoric acid) is sufficient for bonding metal orthodontic brackets to all ceramic crowns, and should make it simpler for clinicians to remove the remaining adhesive from the porcelain surface after debonding.

3. The negative influence of thermocycling prior to bonding can be seen on shear bond strength values.

4. Most of the failures of the bond occurred at the ceramic/adhesive interface and cohesive fractures within the composite resin. No cohesive fractures of the porcelain crowns were noted.

Keywords:

Bond strength

Self-adhesive resin

Metal Orthodontic Brackets

All Ceramic crowns

Thermocycling

Declaration

I the undersigned declare that *Bond strength of metal orthodontic brackets to all ceramic crowns* is my own work and that it has not been submitted before for any degree or examination to any other university.

Moosa Ismail **July 2016**

Signed:.....

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Introduction

There has been an increasing interest and demand in the use of all-ceramic materials to restore severely damaged teeth or to replace lost teeth, particularly in adult patients. This is largely due to its' non-metallic, biocompatible and improved aesthetic properties (Blatz *et al* 2003, Conrad *et al* 2007).

In the anterior region, the most commonly fabricated silica based ceramic crown is the IPS eMax crown and the most commonly fabricated high strength ceramic crown is the feldspathic porcelain veneered zirconia based crown. Although the veneered porcelain reduces the flexural strength of the zirconia based ceramic, its translucency is greatly improved making it more aesthetically pleasing in the anterior regions (Fradaeni 2012).

The demand for orthodontic treatment in adult patients has been increased considerably, together with the increase of patients' knowledge and change in modern lifestyle. As a result, orthodontists are required to attach orthodontic attachments or fixed retainers to teeth which may have been previously restored with ceramic restorations such as crowns or veneers.

Ceramic is an inert material and therefore does not adhere chemically to any of the currently available bonding resins. Therefore, in orthodontics, ceramic surface preparation is an essential step prior to bonding. Several methods like sandblasting (Zachrisson *et al* 1996), using diamond burs to roughen the surface, etching with hydrofluoric acid (Zachrisson *et al* 1996), using silane coupling agents (Kocadereli and Canay Sand Akea 2001), etching with lasers (Raji *et al* 2012), and curing with halogen and plasma arc lights (Toodehzaeim *et al* 2012), have been advocated to increase the bond strength of orthodontic brackets to the porcelain surface. However, mechanical alteration (sandblasting and using diamond burs) to roughen the surface of porcelain can cause irreversible damage and compromise the integrity of the porcelain crown (Ajlouni *et al* 2005). Anecdotal evidence suggests orthodontic brackets bonded with silane coupling agents and phosphoric acid or

hydrofluoric acid has sufficient bond strength for orthodontic treatment (Nebbe and Stein 1996, Schmage *et al* 2003, Ajlouni et al 2005, Lamour *et al* 2006, Abu Alhaija and Al-Wahadani 2007).

The overall time required to place an appliance is an important factor in the cost of the treatment (Ajlouni *et al* 2005). Newer, self-adhesive cements have the potential to further simplify the bonding process. This is done by reducing the bonding of orthodontic brackets to a one-step procedure, and thereby, reduce chair time and increase cost effectiveness, resulting in increased convenience and reduced costs for the patient (Hayakawa *et al* 1992). Reducing the steps during the bonding process will also reduce the risks of saliva contamination and the effects of humidity which could both have an adverse effect on the bond strength of the resin cement.

On the one hand, optimum bond strength is required for minimizing accessory bond failures during the treatment phase, and on the other hand, the porcelain on the restored tooth must return to its initial state of appearance, without any damage to its surface after the brackets are removed (Mattos and Capelli 2006).

Although there are innumerable protocols for bonding orthodontic brackets to porcelain, there is still no scientific consensus about which of the techniques would be the ideal standard protocol for the purpose of overcoming the two points of contrast mentioned above (Herion *et al* 2010).

Increasing demands of adults for orthodontic treatment and controversy of the results in efficient methods of bonding to ceramics require more investigations. Hence, the purpose of the present study was to test and compare the shear bond strength and the resultant failure pattern of 2 types of resin adhesive cements (RelyX™ Unicem 2, a self-adhesive resin cement and Transbond™ XT, a 2 step bonding resin cement) to etched and silane treated all ceramic crowns. Additionally, a further aim of this study was to examine an alternative to etching using hydrofluoric acid which is noxious and

harmful. Instead, etching with 35% ortho-phosphoric acid and silane coupling application as pre-treatment preparation of the all ceramic crown surfaces before bonding was investigated. Furthermore, examining the effect of thermocycling (ie. the ceramic specimens will be thermocycled to simulate the oral environment prior to bonding of the orthodontic bracket to the ceramic crown) on the shear bond strengths, which many studies have not included, was also tested.

1.1. Aim

The aim of this study was to evaluate, in-vitro, the shear bond strength (SBS) and the resultant failure pattern after debonding of metal orthodontic brackets bonded with Transbond™ XT and RelyX™ Unicem 2 self-adhesive resin cement to pre-treated (35% ortho-phosphoric acid and silane coupling agent application) IPS eMax and porcelain veneered zirconia crowns.

1.2. Objectives

The objectives of this study were to compare:

1. The **shear bond strengths** of the self-adhesive resin cement (RelyX™ Unicem 2) and the 2 step adhesive resin cement (Transbond™ XT light cure adhesive primer and Transbond™ XT adhesive resin cement) to the pre-treated (35% ortho-phosphoric acid and silane coupling agent application), non-thermocycled IPS eMax and porcelain veneered zirconia crowns.
2. The **resultant failure pattern** of all the tested groups.
3. The **effects of thermocycling** on the shear bond strengths of the tested groups.
4. The **surface integrity** of the IPS eMax and porcelain veneered zirconia crowns after debonding for each of the groups tested.

1.3. Hypothesis

Transbond^{TM} XT, a 2 step adhesive resin cement and RelyX^{TM} Unicem 2, a self-adhesive resin cement have bond strengths which will be sufficient to bond metal orthodontic brackets to prepared ceramic crowns (etched with 35% ortho-phosphoric acid and treated with a silane coupling agent) for the period of orthodontic treatment.

Chapter 2

Review of the Literature

Review of the Literature

Introduction

Ceramic material is believed to be the most aesthetically pleasing option for the replacement of a lost tooth, the repair of a damaged tooth or for masking of an unattractive enamel surface. The demand for naturally looking restorations has led to the development of more advanced porcelain systems.

Ceramics are made from the melting and fusion of non-metallic materials, like clay, after having fired them at high temperatures (850-13000^{o}C). Dental porcelains are a form of ceramic and can be classified according to (Anusavice 2003, Rosensteil *et al* 2006):

a. Indications: anterior or posterior crowns, veneers, post and cores, stain ceramics, glaze ceramics and bridges.

b. Composition: Pure alumina, alumina-based glass ceramic, pure zirconia, zirconia-based glass ceramics, silica glass ceramic, leucite-based glass ceramic and Lithia-based glass ceramic.

c. Processing methods: sintering, partial sintering and glass infiltration, copy-milling and CAD/CAM (computer aided design/ computer aided manufacture).

d. Firing temperature: ultra-low fusing, low-fusing, medium-fusing and high-fusing.

e. Microstructure: glass, crystalline, and crystal containing glass.

f. Translucency: opaque, translucent and transparent.

g. Fracture resistance.

h. Abrasiveness.

For the purpose of this thesis, a general understanding of the various currently available dental ceramics will aid in the understanding of the materials' different behaviours. There are essentially

two families of dental porcelain: the family of glass ceramics and the family of poly-crystalline ceramics in which physical properties vary greatly.

The family of glass ceramics can further be divided into three sub-groups of dental ceramics: feldspathic porcelain, leucite-reinforced porcelain and lithium-disilicate porcelain. Feldspathic porcelain is formed from clay or sand that has been fired at high temperatures. It becomes a vitreous dental ceramic formed of a glass matrix and one or more crystalline phases. Conventional feldspar porcelain contain a silica network (SiO_2, 52-62% by weight), alumina (Al_2O_3, 11-16% by weight), lithium oxide and barium oxide additives, either potash (K_2O_3, 9-11% in weight), soda (Na_2O_5, 5-7% by weight) or both. As dental feldspars are relatively colourless and pure, pigments, opacifiers and other types of glass modifiers are required to reproduce the hues of natural teeth, to control the fusion and sintering temperatures, and to control the coefficient of thermal contraction and solubility (Anusavice 2003).

Feldspathic porcelains are the most aesthetic type of porcelain, but have the weakest flexural strength of 90MPa; they contain less than 40% leucite by content. Leucite is a crystalline mineral formed from melting potassium feldspar or potash. In other words, leucite is a potassium-aluminium-silicate mineral (Rosensteil *et al* 2006). In contrast, leucite-reinforced or leucite-based glass ceramics contain more than 35-50% of leucite in weight dispersed in a glassy matrix, and have a higher flexural strength of 110MPa (Kelly and Benetti 2011). Lithium-disilicate or Lithia-based glass ceramics has a greater flexural strength of 300-400 MPa. These materials can be relatively translucent (Raigrodski 2004).

The family of poly-crystalline ceramics is divided into two sub-groups: alumina and zirconia ceramics. The alumina or zirconia may come in pure forms, or be dispersed in a glass matrix as alumina- or zirconia-based glass ceramics. Alumina ceramics have a high fracture toughness and hardness, with a flexural strength of 700-800 MPa.

Zirconia or zirconia oxide is also considered a core material, upon which aesthetic feldspathic porcelain can be layered, as it has the highest flexural strength of 1100-1300 MPa. Although this type of ceramic is the strongest and toughest, it has a disadvantage: zirconia can only be fabricated through computer aided design-computer aided manufacturing (CAD/CAM) technology, which can be expensive and technique sensitive (Rosensteil *et al* 2006).

2.1. Porcelain surface Preparation

In vivo the tooth surface is covered by a pellicle consisting of a protein film that forms on the surface of the enamel/porcelain by selective binding of the glycoproteins from saliva. In order to remove the pellicle from the enamel/porcelain surface, routine bonding clinical practice sees enamel/porcelain surfaces cleaned with a slurry of pumice and a brush or rubber cup, used in a slow handpiece, prior to etching. There is no reported difference in the failure rate of the bond in cases where the polishing of the enamel/porcelain surface was excluded (Barry 1995, Eliades 2006). Prophylaxis pastes are contra-indicated as the fluoride content or the oils and the flavouring agents added to the pastes are believed to have a detrimental effect on the bond strength (Garcia-Godoy *et al* 1991).

Ceramic being an inert material does not adhere chemically to any of the currently available bonding resins. It is therefore necessary to change the inert characteristics of the surface to achieve clinically acceptable bonding of orthodontic metal brackets to ceramic surfaces (Abu Alhaija *et al* 2010). There are distinct differences in the constituents; particle size and crystalline structure of the commercially available porcelains but usually have a similar chemical formula. Different results are expected regarding bonding orthodontic brackets to porcelain (Hayakawa *et al* 1992). Authors have suggested three different approaches: a) physical or mechanical preparation, b) chemical preparation

and c) combined mechanical and chemical preparation of the ceramic surface (Mair and Padipatvuthikul 2009).

2.1.1. Mechanical Preparation

Preparing the ceramic surface mechanically involves the removal of the porcelain's glaze and/or the roughening of the porcelain surface to provide more surface area for chemical retention. Several options are available and are relatively quick chair side procedures. The use of coarse diamond burs has been well documented, along with green stones, and abrasive discs (Bourke and Rock 1999, Schmage *et al* 2003, Karan *et al* 2007). Zachrisson *et al* (1996) found that intra-oral sandblasting with microscopic particles of aluminium oxide removes the porcelain glaze better than burs or stones, since only a small amount of surface is removed and the result is more uniform. Although this requires minimal chair side time, the aluminium oxide particles are difficult to contain within limits of the mouth and also requires thorough rinse of the area afterwards. Authors have found that fine diamond roughening and sandblasting showed the highest surface roughness when compared to surface roughness obtained by acid etching (Schmage *et al* 2003). A retention cavity can also be cut in the porcelain surface to assist with bonding of the resin cement to the porcelain surface (Wood *et al* 1986, Bourke and Rock 1999). Laser radiation of the porcelain surface has also been studied as an alternative conditioning technique, but it is a very costly procedure (Zachrisson *et al* 1996).

However, mechanical alteration of the surface of porcelain can cause irreversible damage to the porcelain glaze and compromise the original lustre and integrity of the porcelain crown (Ajlouni *et al* 2005).

2.1.2. Chemical Preparation

The acid used in the chemical preparation of a ceramic surface is meant to create a series of micro-retention pits by the preferential dissolution of the glass phase within the ceramic matrix (Bourke and Rock 1999). Although this procedure yields mechanical retention and not a chemical bond to the ceramic surface, it is included as part of a chemical preparation because it entails the application of a technique-sensitive liquid product.

2.1.2.1. Acid Etching

The most commonly used ceramic acid etchant is a 9.6 % hydrofluoric acid gel. A 2-4 minute application of hydrofluoric gel has been advocated (Kocadereli and Canay Sand Akea 2001). Clinically, there are drawbacks with the use of hydrofluoric acid. It is very acidic and must be used with great care, and it is extremely corrosive and capable of causing severe trauma to soft tissue and tooth substance (Hayakawa *et al* 1992). Careful isolation of the working area (use of the rubber dam) is required. Bourke and Rock (1999) have questioned the clinical relevance of bond strengths with hydrofluoric acid application. Their shear bond strength study found that the shear bond strength was similar when comparing the groups that have use hydrofluoric acid with those that used phosphoric acid. If there is no added advantage of using hydrofluoric acid, one should eliminate it for obvious reasons.

Another study found that 1.23% of acidulated phosphate fluoride (APF) was a suitable substitute to hydrofluoric acid etching, while being a safer product. However, a 10 minute etching time with acidulated phosphate fluoride (APF) provided shear bond strength to a 1 minute hydrofluoric acid etching time (Nelson 1989, Abu Alhaija *et al* 2010). There are also contradicting results in the

current literature on acidulated phosphate fluoride application which provide clinically unacceptable low bond strengths (Abu Alhaija *et al* 2010, Heravi *et al* 2010, Raji *et al* 2012).

There are also inconsistent findings on the effects of acid etching with ortho-phosphoric acid on porcelain bond strengths. It was demonstrated that ortho-phosphoric acid with a concentration of 37% is not able to etch a ceramic surface and, consequently, does not produce physical or topographical changes on porcelain (Aidam *et al* 1995). However, in a study by Lamour *et al* (2006), etching ceramic surfaces with 37% ortho-phosphoric acid was reported to produce clinically acceptable bond strength comparable with that produced by the application of hydrofluoric acid. Anecdotal evidence suggests orthodontic brackets bonded with silane coupling agents and ortho-phosphoric acid or hydrofluoric acid has sufficient bond strength for orthodontic treatment (Nebbe and Stein 1996, Schmage *et al* 2003, Aljouni *et al* 2005, Lamour *et al* 2006, Abu Alhaija and Al-Wahadani 2007).

2.1.2.2 Silane Application

Silane coupling agents have been reported to enhance bond strength to ceramic surfaces (Kao *et al* 1988, Kocadereli and Canay Sand Akea 2001). Silane molecules, after being hydrolysed to silanol, is able to form a polysiloxane network or hydroxyl groups to cover the silica surface. Monomeric ends of silane molecules react with the methacrylate groups of the adhesive resins by free radical polymerization (Gillis and Redlich 1998, Daub *et al* 2006). In a study by Faltermeier *et al* (2012), etching with 37% ortho-phosphoric acid for 2 minutes and followed by a silane coupling agent application (pre-treatment procedure of veneering ceramics before bonding of the bracket), seem to prepare the surface of the ceramic restoration sufficiently before bracket bonding. In another study by Guimaraes *et al* (2012), surface etching with phosphoric acid, followed by silane application provide adequate bond strength, capable of resisting the forces applied during orthodontic treatment,

without causing irreversible failures in porcelain. On the other hand, ortho-phosphoric acid has the ability to neutralize the alkalinity of the adsorbed water layer, which is present on all ceramic surfaces in the mouth and, thereby, improve the chemical activity of any silane primer when subsequently applied (Hayakawa *et al* 1992). Therefore, the use of ortho-phosphoric acid followed by silane application seems to be an acceptable protocol for bonding orthodontic accessories to porcelain surfaces.

2.2. Measuring Shear Bond Strength

There are clinically many variable factors that are associated with the shear bond strength of any adhesive material (Thomas *et al* 1999, Eliades and Brantley 2000, Aljouni *et al* 2005). These variable factors associated with shear bond testing need to be carefully analysed in order to produce a reliable testing protocol for the results to be in any way meaningful (Eliades and Brantley 2000). Comparing different materials through in vitro testing is a common place. However, attempting to gain any clinical significance from such tests remains controversial.

Values for Shear Bond Strengths (SBS) can only be determined and measured in a laboratory or in vitro environments. Bond force is usually measured in shear or tension on a universal testing machine, although torsional testing has been reported (Rossouw 2010). In shear testing, the brackets are loaded by a blade in compression or by a wire in tension, so that the brackets slide parallel off the substrate. Unfortunately, pure shear loading is difficult to achieve. Most shear testing includes components of peeling, tension and torsion as well. Both shear and tensile loading modes are valid tests for studying orthodontic bonding (Powers *et al* 1997). However, many investigators believe that testing in tension or torsion loading modes are less relevant for clinical application, and have thus placed focus on shear testing for ease of reproducibility of protocols (Eliades and Brantley 2000). The average force transmitted to a bracket during mastication has been reported to be between 40

and 120 Newtons (N) (Reynolds 1975). The bond strength tests are basically performed as follows: in all situations a bracket is bonded to a substrate, enamel or porcelain. After some time of storage, a force is applied to the bracket until fracture occurs. The bond strength of the material is presented in Mega Pascals (MPa) and is the amount of force at the time of fracture which is measured in Newtons (N) divided by the bonding area of the bracket base in mm^2, MPa= Newtons/mm^2 (Conrad *et al* 2007, Rossouw 2010). Directly after testing the fracture area is examined and scored using the Adhesive Remnant Index (ARI score), first described by Artun and Bergland (1984). With this test the amount of residual cement left at the bonding surface is scored on a 4-point scale as shown in the table below:

Score	Definition
0	No adhesive remained on the porcelain surface
1	less than 50% of the adhesive remained on the porcelain surface
2	more than 50% of the adhesive remained on the porcelain surface
3	All adhesive remained on the porcelain surface

Table: 1. Adhesive Remnant Index (ARI)

The scores are determined with an optical microscope at a magnification of 10-25x.

2.3. Factors Affecting Bond Strength

Many variables must be taken into account when interpreting data and results of bond test studies (Bishara *et al* 1999).

These include:

- Type of surface (teeth vs porcelain).
- Type of porcelain (leucite vs feldspathic vs alumina).
- The different bonding agents.
- Type of bracket base (mesh size and topography).
- Type of surface treatment (mechanical vs chemical preparation, and its various products and concentrations).
- Type of bonding method (direct or indirect).
- The duration, intensity and direction of the light cure source.
- The size, speed and direction of the debonding force.
- The time period between bonding and debonding.
- Type of aging process (water storage, thermocycling, etc).
- In the clinical scenario the clinician's skills as well as the management of the oral environment play an important role in the success of the bond between the enamel/porcelain and the bracket.

2.4. Porcelain: Shear Bond Strength Comparisons

The ideal rupture force for clinically successful orthodontic bonding is between 5.9 and 7.9 MPa (Guimaraes *et al* 2012).

In a study by Faltermeir *et al* (2012), two surface conditioning methods of 4 ceramic materials before bonding was examined (Group 1- air particle abrasion with 25µm aluminium trioxide and subsequently a silane coupling agent was applied, Group 2- samples were etched with 37% ortho-phosphoric acid followed by silane application). Self-ligating metal brackets were bonded to the

ceramic blocks with Transbond™ XT and thermocycled (5^0C-55^0C, 6000 cycles). Shear bond strength testing was performed using the universal testing machine at a cross-head speed of 1mm/min. The statistical analysis of the data obtained in this study revealed that there is no significant enhancement of shear bond strength using sandblasting with 25µm aluminium trioxide in comparison to using 37% ortho-phosphoric acid as a surface conditioning method of ceramic restorations. The results show that median values of 130-217,5 MPa could be reached using only 37 % ortho-phosphoric acid together with a silane coupling agent. This level of shear bond strength is described to be sufficient for bracket bonding and to avoid accidental bracket debonding (Faltermeier *et al* 2012).

Guimares *et al* (2012) evaluated in vitro, the shear bond strength of orthodontic accessories to porcelain, under different porcelain surface treatment protocols. The sample consisted of 80 feldspathic porcelain discs which were divided into 4 groups using different porcelain surface treatment protocols (Group 1- 37% phosphoric acid for 1 minute, only, Group 2- hydrofluoric acid only, Group 3- phosphoric acid and silane application, Group 4- hydrofluoric acid and silane application). After bonding with the adhesive system and resin composite, Transbond™ XT, all samples were stored in 0.9% physiological serum for 24 hours, at ambient temperature, before being submitted to shear bond strength test. A universal test machine was used. The test machine was calibrated with a 50 N load cell at a speed of 0.5 mm/min. Group 1 showed statistically lower results (mean bond strength value- 2.21 MPa), Group 4 showed statistically higher results than the other groups (mean bond strength- 21.93 MPa), Groups 2 and 3 showed statistically equal performance (mean bond strength- 7.24 MPa). The results of the study suggest that:

- The application of silane significantly increases the shear bond strength of orthodontic accessories to porcelain;
- Etching the porcelain surface with phosphoric acid alone does not provide adequate shear bond strength;

- Surface etching with hydrofluoric acid, with or without silane application, increases the occurrence of irreversible fractures in porcelain;
- Surface etching with phosphoric acid, followed by silane application provided adequate bond strength, without causing irreversible failures in porcelain.

2.5. Thermocycling

Orthodontic composite and adhesive resins are routinely exposed to temperature variations in the oral cavity. Intra-oral temperatures vary between 0^0C when eating ice cream to 60^0C when eating a hot cheese sandwich (Mair and Padipatvuthikul 2009). Thermocycling, usually between 5^0C and 55^0C water baths, thus simulates the temperature changes of the oral environment and recreates the artificial aging process. Bishara *et al* (1999) suggested that thermocycling be part of the testing protocol of new resins.

Studies that incorporated thermocycling demonstrated statistically significant reductions in SBS between orthodontic resins and tooth or porcelain surfaces, in both direct and indirect bonding studies (Smith *et al 1988,* Bourke and Rock 1999 Faltermeier *et al* 2012). In all the previous studies the thermocycling was done after the bonding of the orthodontic attachments and prior to debonding.

However, when an adult patient presents for orthodontic treatment, they may have teeth which were restored with ceramic restorations that have been in the oral cavity for a long time. These ceramic surfaces will be altered by variations in temperature, saliva, acidity and adsorptions of mucoproteins and mucopolysaccharides (Zachrisson *et al* 1996). Therefore, thermocycling (superficial aging and simulating the clinical environment) of the ceramic specimens prior to bonding of the orthodontic bracket onto the ceramic surface, and its effect on the shear bond strength of the orthodontic brackets

to the ceramic crowns, will help to simulate the clinical environment as closely as possible and hopefully overcome this limitation which is observed in previous in-vitro studies.

2.6. Bonding of Metal Orthodontic Brackets

2.6.1. The bracket base

Orthodontists choose orthodontic brackets according to various treatment related factors (Proffitt and Fields 3rd ed., Mosby Co, Alexander 1986). The effectiveness of the adhesive surface is just as important as any other consideration (ie, type of adhesive, treatment and preparation of ename/porcelain) as all treatment results depend on the success and stability of the bond between the enamel/porcelain and the bracket. In the attempt to improve bond strength the focus of development has been the adhesive pad (Matasa 2003). There are many variations in the adhesive surface design of orthodontic brackets. Some brackets are manufactured with grooves, some with perforations; some have stainless steel brazed onto the adhesive pad, while some bases are laser formed. Each manufacturer claims its own 'in house' adhesive surface design, trademarks and/or patents but at the same time providing very little information regarding their dimensions (Matasa 2003). The bracket base design, size and surface treatment are important variables when it comes to bond strength testing (Sharma-Sayal *et al* 20003).

The improvement in the bond strengths of the bonding agents since inception has been significant. This coupled with the aesthetic demands of an aesthetically conscious society and the refinement of the bracket base design has allowed manufacturers to decrease the size of the base, without sacrificing bond strength. Matasa (2003) claims the size of the adhesive surface of the bracket has been reduced by 75% in recent years. Orthodontic metal brackets have an average adhesive base size of 9 to 12 mm^2 (Alexander 1986, Bishara *et al* 1999, Sorel *et al* 2002), and rely on mechanical

retention for their bond strength. The size of the base is important because of the oral hygiene ramifications, the strength of the bond, and the aesthetic considerations (Sharma-Sayal *et al* 2003). The effectiveness (design) of the adhesive surface is important for the stability, the strength of the bond, the ease of debonding, as well as the amount of bonding agent left on the enamel/porcelain surface after debonding. MacColl *et al* (1998) found that the shear bond strength was independent of the base size once the surface of the bracket exceeded 7mm^2. Banks and Macfarlane (2007) claimed that there is no apparent relationship between the size of the adhesive pad and bond strength.

2.6.2. Composite Resin Cements

Intra-orally, dental cements and adhesive resin cements are used to bond orthodontic attachments to teeth. Since the pioneering studies of Buonocore in 1955, there have been many advances in the bonding of orthodontic attachments to natural teeth. Recent progress in materials and techniques has shown that direct bonding of orthodontic attachments to surfaces other than enamel is also possible, such as ceramic surfaces. Adhesive resins have the advantage of low solubility and improved physical characteristics over cements. The resins are less susceptible to fracture than the cements resulting in higher bond strengths (Hudson 2007). Adhesive resins, however, do not bond well as a result of moisture contamination.

Good bond strength, clinically, is dependent on (Sfondrini *et al* 2004):

- Avoiding moisture contamination of the etched enamel/porcelain.
- Undisturbed polymerization of the bonding agent.
- Using a bonding agent with sufficient strength.
- Minimising occlusal stress (Banks and Macfarlane 2007).

The method for direct bonding of the orthodontic brackets is well known and understood by clinicians. It begins with the isolation of the oral environment, preparation of the tooth or ceramic surface, application of uncured composite resin on the back of the bracket, and placement of the individual bracket in its correct position on the surface of the tooth or ceramic restoration. Photo-polymerization of the composite resin is initiated to secure the bracket onto the surface of the tooth or ceramic restoration. Ideally this step is performed for each bracket and tooth or ceramic restoration individually.

Composite resin cements are the most popular choices for bonding orthodontic brackets to substrates. In order to maximise their advantages, composite resin cements are a combination of materials of differing properties (Matasa 2005). These advantages are improved mechanical properties, aesthetics, reduced polymerization shrinkage and a reduced coefficient of expansion (Aljouni *et al* 2005).

A wide variety of orthodontic resin bonding agents are available and there are a formidable set of criteria required for them to be successful (Proffitt and Fields 3rd ed.). Ideally they should be:

- Dimensionally stable.
- Fluid enough to penetrate etched enamel/porcelain and the retentive part of the bracket base.
- Strong enough to withstand the forces experienced in the mouth.
- Viscous enough to prevent the bracket moving on the tooth/crown surface subsequent to placement and prior to curing.
- User friendly.

The main categories of composites are:

- Dispersion-strengthened

- Particle strengthened
- Laminar (sandwich) and
- Fibre re-inforced

Almost all composite resin dental cements are particle strengthened and the filler particles exceed 25% of the composition of the composite. These particles have a strengthening effect in the composite (Hudson 2007). Chemically these material components display distinct boundaries between their particles (Matasa 2005). Little has changed in the composition of the composite resins in the last 50 years because of the consistent reliability of the bond achieved. They are still a mixture of Bis-GMA (Bisphenylglycidal-methacrylate) diluted with a less viscous acrylate (Matasa 2003, Aljouni *et al* 2005).

The composite resins are thermo-cured, light-cured, or chemically-activated or dual-activated. Composite resin cements are a class of materials that do not inherently contain water. To obtain optimal adhesion, composite resin cements require acid-etched or roughened dry surfaces for the best mechanical retention. They are also more fracture resistant than glass ionomer cements. Unfortunately, composite resin cements have the disadvantages of not bonding well in the presence of moisture and their attachment to surfaces is primarily mechanical (Matasa 2005, Hudson 2007).

Thermo-cured composite resin cements are available for custom base fabrication. It is dispensed as a single-paste onto the bracket base, which is then placed onto the casts. The resin cement stays unpolymerised until the cast is cured with heat for at least 15 minutes (Klange 2007). Only thermo-cured or light-cured composite resin cements allow an unlimited working time before polymerization (Klocke *et al* 2003).

Light-cured composite resin cements are available and are dispensed as a single paste. These single-component materials are easier to manipulate. The composite resin cement is cured with a handheld curing light. Again, the bracket placement can be verified indefinitely before curing, provided that the brackets are not exposed to light (Read and O'Brien 1990).

Chemically-activated or auto-polymerizing or dual-activated composite resin cements are supplied as a two-part formulation with a base and catalyst. However, the newer dual-activated composite resin cements are supplied in an automix syringe thereby eliminating the need for hand-mixing the composite resin cement prior to application. Handling and applying these materials is less problematic and less time consuming.

2.6.2a. Light-cured Composite Resin Cement

Nowadays, Transbond™ XT of 3M Unitek is one of the most commonly used light curing composite resin cements for bonding orthodontic brackets to enamel/porcelain substrates. Transbond™ XT is composed of 14% volume Bis-FMA, 9% volume Bis-EMA, and 77% volume filler particles (Bishara *et al* 1997). Because of its clinical effectiveness Transbond™ XT is often used as a reference material in laboratory research (Bishara *et al* 1997).

2.6.2b. Dual-cured Composite Resin Cement

RelyX™ Unicem 2 is a dual curing, self-adhesive resin cement supplied in an automix syringe. It is used for the adhesive cementation of indirect all-ceramic, composite or metal restorations and for posts and screws. Kumbuloglu *et al* (2004) determined that Unicem had the highest compressive strength among the four resin composite luting cements tested. Additional research conducted by Piwowarczyk and Lauer (2003), determined that although not as strong as resin cements, Unicem

proved to have stronger flexural and compressive strength than resin-modified glass ionomer cements, glass ionomer cements, and zinc phosphate cements. Other studies have demonstrated that over long periods of time and after thermal cycling, Unicem retains its adhesion and strength properties better than other resin cements, suggesting the potential use of the adhesive for longer term applications (Porseld *et al*, Hecht *et al*, Piwowarczyk *et al* 2004).

The use of RelyX™ Unicem in operative and prosthodontic applications without etching the enamel has provided contradictory results (De Munck *et al* 2004, Kumbuloglu *et al* 2005). For example, in a study investigating the shear bond strengths of composite resin cements to lithium disilicate ceramics, there was a significant difference between the bond strengths of Unicem (with no acid etch step) and other adhesive resin cements that require an additional acid etch step, namely, Panavia F (Kumbuloglu *et al* 2005). However, in another study it was found that the tensile bond strength of Unicem was similar to Panavia F bonding system only when a separate acid etch step was used before the application of the Unicem adhesive resin (De Munck *et al* 2004).

Although numerous studies have been conducted in assessing Unicem's potential applicability in operative and prosthodontic procedures, very little data are available on Unicem's potential for the use as an orthodontic bracket bonding adhesive.

With such limited data, and with the newer Unicem 2 Automix dispensing tips which make dispensing of the Unicem cement onto the orthodontic brackets easier, there is a need to further assess the potential use of Unicem as a one-step orthodontic bracket bonding system.

2.7. The light curing process

Maximum conversion of monomer, in the bonding agent, to polymer is required to achieve optimum bond strength. The thickness of the adhesive layer (which is considerably thinner than 2mm) is

largely determined by the amount and size of the filler particles in the resin, its viscosity, tooth surface irregularities and the bracket placement technique (Hudson 2007). This thin adhesive layer between the bracket base surface and the enamel/porcelain surface should therefore convert to polymer easily.

Light cured bonding agents are now routinely used to bond orthodontic attachments to teeth/crowns because of their ease of use and the time saved (Klocke *et al* 2003). The conventional halogen light, which is commercially available, is the most common and most affordable light source, and thus the instrument of choice since the seventies. These halogen lights display a wide intensity spectrum ranging from, approximately, 400 mW/cm^2 to 1000 mW/cm^2 (Kauppi and Combe 2003, Swanson *et al* 2004). Kauppi and Combe (2003) found that conventional as well as high intensity halogen curing lights show a drop in light intensity after 30 seconds of continuous use. The bond strength depends on the composition of the bonding agent as well as the intensity of and the exposure to the light source as well as the time elapsed after exposure (Swanson *et al* 2004).

2.8. Time post-cure

Bishara *et al* (1999) demonstrated that the initial bond strength of Transbond^{TM} XT adhesive resin more than doubled in the first 24 hours. Sharma-Sayal *et al* (2003) also found an increase in the shear bond strength of Transbond^{TM} XT adhesive resin but not to the same extent as Bishara and co-workers. Okemwa *et al* (2002) showed that the shear bond strength of Transbond^{TM} XT after 24 hours and after 7 days remained constant at 123 Newtons on premolars.

2.9. Bracket removal

2.9.1. Bond strength testing

The literature contains a large number of publications on in vitro bond strength testing of materials, the results of which are quoted by manufactures in support of their products. However, little attention has been paid to the detail of the test procedures used. Fox *et al* (1994), published a critique of bond strength testing in orthodontics, which revealed a large variation in the methods used, and the case for a possible standard technique was suggested. Van Noort *et al* (1989), and Rueggeberg (1991), both suggested the need for standardization of test procedures for the measurement of bond strengths, to allow valid comparisons to be made between different bonding agents.

There has been confusion in the literature over the unit of measurement most appropriate for describing bond strength (Fox *et al* 1994). Units such as Pascals, MegaPascals, Newtons per millimetre squared or MegaNewtons per metre squared have been used. These units provide an indication of the force per unit area required to dislodge the bracket. The use of force as an indicator of bond strength is only appropriate where the area is well controlled, but difficult to measure. As long as the dimensions (surface area) of the bracket base are quoted, the use of Newtons or MegaPascals is appropriate in quoting bond strength.

Laboratory shear bond strength depends on several factors including the bracket base retention mechanisms, the bonding system, the type of enamel/porcelain conditioner used, the etch pattern of the enamel/porcelain, the point of force application, direction and crosshead speed of the force applied (Eliades and Brantley 2000, Klocke *et al* 2003). In vivo shear bond strength tests show significantly lower bond strengths than in vitro tests (Pickett *et al* 2001).

Shear bond forces should be applied to the base of the attachment (Klocke and Kahl-Nieke 2005), as forces applied to any part of the attachment may corrupt comparative results and in this way may be

a reflection of the bracket design variability (resulting in varying force vectors) and not the base design or the adhesive material (Eliades and Brantley 2000, Klocke and Kahl-Nieke 2005).

Normal orthodontic forces applied to the brackets are estimated to produce stresses in the region of 3 to 7.8 MPa. For an adhesive system to have a clinical acceptable performance, the in vitro bond strength should be between 6 and 8 MPa (Clarke *et al* 2003).

In 58 out of 66 papers (Fox *et al* 1994), an Instron machine or similar testing machine was used. Other devices used included a pair of specially designed opening pliers (Perry 1980), and various other testing machines (Newman 1965, Hirce 1980, O'Brien *et al* 1991). Forty four of the papers tested the specimens in shear mode, sixteen in tensile and six used a combination of directions.

The majority of research into shear bond strength with a universal testing machine has applied unilateral forces to the test specimen. The results cannot be applied to clinical debonding (Olsen *et al* 1996, Olsen *et al* 1997, Fernandez and Cnut 1999). Debonding with sharp-edged pliers that apply a bilateral force at the bracket base-adhesive interface has been found to be an effective method of debonding orthodontic brackets (Farquhar 1986), and its use in vitro simulates more closely the debonding forces applied in actual clinical situations (Bishara *et al* 1993, Bishara *et al* 1994, Bishara *et al* 1995).

Eliades and Brantley (2000) commented on in vitro debonding as follows; "The simulation of clinical conditions is a task that is not to be attainable in the near future."

2.9.2. Concerns when Debonding

Cohesive failures occur either within the ceramic or tooth substrate, the bracket or the adhesive system. Adhesive failures occur between the ceramic-adhesive resin and the bracket-adhesive resin interfaces. Unfortunately, cohesive fractures of ceramic restorations resulting from bracket removal

are common and unpredictable (Newman 1994, Andreason and Stieg 1998). These fractures pose problems of an aesthetic and financial nature if they are large or extensively deep. The clinician may attempt to repair the slight porcelain damage with polishing systems. One can further try to prevent further extension of the micro-cracks by finishing and polishing with a series of graded ceramist points or diamond impregnated polishing wheels. Wood *et al* (1986) and Kao and Johnston (1988), both agree that this procedure can yield an acceptable, although not ideal, aesthetic result if finalised with a diamond polishing paste.

Understanding the nature of cohesive failures can give clues on how to avoid them. Cohesive porcelain fractures occur when the adhesive strength at the metal bracket-porcelain interface exceeds the cohesive strength of the porcelain. Mechanical roughening with diamond burs or sandblasting can be guilty of weakening the cohesive strength of the porcelain by creating micro-cracks within the porcelain (Gillis and Redlich 1998, Abu Alhaijah and Al-Wahadani 2007). Therefore, Wood *et al* (1986) tried to avoid bur roughening and preserved the glaze.

As with mechanical preparation, silane treatment has also been blamed for porcelain fractures at debonding sites by excessively enhancing the bond strength. In Larmour *et al*.'s study (2006) all samples had silane treatment, without any mechanical preparation. They still found a high incidence of porcelain surface damage visible at debond, particularly in the groups using Transbond™ XT composite resin cement when compared with Fuji Ortho LC cement, where 37,5% of the ceramic samples had visible damage. Thus even without mechanical preparation, one may still obtain porcelain fracture.

From a clinical perspective, it would be prudent to warn patients about the risk of damage to porcelain surfaces prior to the commencement of treatment and of the possible need to repair or replace them following orthodontic treatment.

2.10. Adhesive Remnant Index (ARI)

2.10.1. Background and Relevance

ARI scores are used to define the site of bond failure between enamel-porcelain, the adhesive and the orthodontic bracket base. The index scores the amount of resin remaining on the tooth after debonding. Many studies have used the Adhesive Remnant Index (ARI) developed originally by Artun and Bergland in order to assess the amount of resin remaining on the tooth surface after orthodontic bracket debonding (Artun and Bergland 1984, Powers *et al* 1997, Heravi *et al* 2010). The original ARI scores were defined from 0 to 3 or a 4-point scoring scale. The ARI was then modified by Bishara *et al* (1999) who gave the scores from 0 to 4 to include a score representing porcelain fractures. Unfortunately, many studies use other variations of the ARI. Due to lack of methodology standards and variability in the ARI scores, the reader must be careful when interpreting ARI numeric scores and values. Therefore, for this study we have decided to use the original ARI by Artun and Bergland (1984) *(see Table: 1)*.

However, this original index did not include recording or assessing damage which may have occurred to the porcelain surface after debonding of the orthodontic bracket.

2.11. Porcelain Fracture Index (PFI)

Bonding of an orthodontic bracket to a ceramic crown with an adhesive resin cement should not only be strong enough to resist accidental debond during treatment but should also prevent irreversible damage to the ceramic crown when the brackets are removed at the completion of treatment (Zachrisson 1996). Therefore, in addition to the shear bond testing, it is important to evaluate the quality of the porcelain surface after the removal of the residual adhesive.

A Porcelain Fracture Index (PFI) which was developed by Bourke *et al* (1999) will be used to assess the surface integrity of the porcelain surfaces after debonding. The PFI uses a 0 to 3 or a 4-point scale to evaluate the quality or integrity of the porcelain surface after the removal of the residual adhesive *(see Table: 2).*

Score	Definition
0	Ceramic surface intact in the same condition as before the bonding procedure;
1	Surface damage limited to glaze layer or very superficial ceramic;
2	Surface damage which features significant loss of ceramic requiring restoration of the defect by composite resin or replacement of the restoration;
3	Surface damage where the core material has been exposed due to the depth of the cohesive failure.

Table: 2. Porcelain Fracture Index (PFI)

All orthodontic bonding systems involve at least three interfaces: the tooth or porcelain interface, the resin interface, and the bracket interface. As previously mentioned, cohesive failures can occur within any of these components. Adhesive failures occur between the tooth/porcelain-adhesive system and the bracket-adhesive system. An observation is that authors do not differentiate between the residual adhesive resin and residual composite resin on the tooth/porcelain when they score; it is all combined under ARI (Powers *et al* 1997). Some researchers prefer bond failures within the adhesive or at the bracket-adhesive interface because it decreases the shear force stress at the crown

surface and increases the probability of maintaining an undamaged crown (Alhuwalia *et al* 2013). However, considerable chair time is needed to remove the residual adhesive with the added possibility of damaging the enamel/porcelain surface during the cleaning process (Bishara *et al* 2000). Other researchers consider the bond failure at the adhesive/porcelain or adhesive/enamel interface more desirable because they do not leave residues on the surface where bonding occurred (Mattos and Capelli 2006). This type of failure in the adhesive/porcelain interface shows that the chemical and mechanical bonding was not equal to or exceeded the mechanical retention provided by the bracket base and the bond strength to the porcelain surface was lower than the cohesive strength of the adhesive bracket (Alhuwali *et al* 2013). However, the enamel/porcelain surface can be damaged when failure occurs in this mode (Britton *et al* 1998). Powers *et al* (1997) further highlighted that isolating the weak link is complicated if the bond failure occurs in two of the three interfaces which happens frequently.

2.12. Data analysis

Klocke and Kahl-Nieke (2005), cautioned against interpreting shear bond strength values from in vitro tests for clinical relevance, as these values may be affected more by the methodology of the tests than the materials.

Chapter 3

Materials

and

Methodology

Materials and Methodology

List of materials used (*see table 3.*).

Name of material	Manufacturer
Transbond™ XT adhesive resin cement	3M Unitek, Monrovia, USA.
Transbond™ XT light cure adhesive primer	3M Unitek, Monrovia, USA.
RelyX™ Unicem 2 self-adhesive resin cement	3M ESPE, USA.
35% ortho-phosphoric acid etchant	ULTRADENT™, Salt Lake City, USA.
RelyX ™ Ceramic Primer	3M ESPE, USA.
Cold-cured epoxy resin	Buchler, Lake Bluff, Ill

Table: 3. List of materials used.

3.1. Sample selection and distribution

A Typhodont maxillary lateral incisor was used and prepared in a conventional method to receive a full ceramic crown. A CAD (computer aided design)/ CAM (computer aided manufacturing) machine was used to scan the prepared tooth and manufacture 40 IPS eMax crowns and 40 zirconia crowns. A technician used the cut back technique to add feldspathic porcelain to the facial surface of the zirconia crowns.

Mounting procedure: A 10mm diameter PVC pipe was used to make 80 plastic cylinders 10mm high. These 80 plastic cylinders were placed on a glass surface and filled with self-curing epoxy resin (Buchler, Lake Bluff, Ill), and a single ceramic crown was embedded into each of the epoxy resin filled plastic cylinders with the facial surface exposed. ***Alignment of crowns***: The crowns were held in place with a piece of Prestik® attached to the facial surface of the crown and a matchstick

was attached to the Prestik® and suspended across the two ends of the plastic cylinder. This helped to align the facial surface of the crown perpendicular to the base and this also helped to orientate the bonding surface to be parallel to the force applied during the shearing strength test. The mounted teeth were kept overnight in distilled water for the epoxy resin to set completely. Half the number of IPS eMax crown specimens (ie.20) and half the number of porcelain veneered zirconia crown specimens (ie. 20) – were thermocycled prior to bonding (ie. to mimic thermal changes which occur in the mouth), from 5 to 55^{o} for 500 cycles as recommended by the International Organization for Standardization (ISO 6872, 2008) (*see Figure: 1.*).

Figure: 1. The thermocycling apparatus used

The remaining 20 IPS eMax crown specimens and 20 porcelain veneered zirconia crown specimens remained new and unexposed to thermal changes.

Bonding process: The facial surfaces of all the thermocycled and non-thermocycled crown specimens were polished using pumice and water slurry in a rubber cup for 10 seconds. The crowns were then washed with distilled water for 15 seconds and dried thoroughly with oil-free air. Etching

of all the ceramic bonding surfaces was performed by the application of 35 per cent ortho-phosphoric acid liquid (ULTRADENT™) for 2 minutes (Faltermeier *et al* 2012). The crowns were then rinsed with distilled water for 15 seconds and dried with oil-free air. A thin layer of RelyX™ Ceramic Primer (3M, ESPE) was applied to the etched surface and allowed to react for 5 seconds, and then air dried so that the solvent evaporated completely (*see Figure: 2.*).

Figure: 2. 35 % phosphoric acid etch and a ceramic primer.

Before bonding, the etched ceramic crown specimens were divided into 4 groups. Group 1 and Group 2 consisted of 10 thermocycled, etched and silane treated IPS eMax crown specimens and 10 thermocycled, etched and silane treated porcelain veneered zirconia crown specimens each. Group 3 and Group 4 consisted of 10 non-thermocycled, etched and silane treated IPS eMax crown specimens

and 10 non-thermocycled, etched and silane treated porcelain veneered zirconia crown specimens each. *The decision to use 10 specimens per group was made partly because of the high cost of the ceramic crowns. A higher number of specimens per group have been recommended for tests involving enamel surfaces (Kalange 2007), where it is possible that greater specimen variation would occur than that seen with ceramic crowns made to one die by a CAD/CAM machine and a skilled porcelain technician.* A lateral incisor metal bracket (Octi^{R}, Dentsply) (*see Figure: 3.*), with a bracket base area of 9mm^{2} (as confirmed by manufacturer) was bonded to each of the etched and silane treated ceramic crown specimens in the following manner:

Group 1: (10 thermocycled, etched and silane treated IPS eMax and 10 thermocycled, etched and silane treated porcelain veneered zirconia crown specimens). A small amount RelyX™ Unicem 2 Automix (3M, ESPE) (*see Figure: 4.*) was placed on the base of the bracket and the bracket was placed onto the bonding surface of the embedded crowns. The bracket was compressed onto each crown using a 300g force (Correx force gauge, Bern, Switzerland). Excess resin was removed with an explorer and the resin was light cured for 40 seconds (20 seconds mesial and 20 seconds distal) with a curing light (Ortholux LED, 3M Unitek).

Group 2: (10 thermocycled, etched and silane treated IPS eMax and 10 thermocycled, etched and silane treated porcelain veneered zirconia crown specimens). A thin layer of Transbond™ XT light cure adhesive primer (3M, Unitek) (*see Figure: 5.*) was applied onto the bonding surface of the crowns and air dried. A small amount of Transbond™ XT adhesive resin (3M, Unitek) (*see Figure: 6.*) was applied to the base of the bracket and the bracket was positioned onto the bonding surface of the embedded crowns. The bracket was compressed onto each crown using a 300g force (Correx force gauge, Bern, Switzerland). The excess resin was removed with an explorer and the resin was light cured for 40 seconds (20 seconds mesial and 20 seconds distal) with a curing light (Ortholux LED, 3M Unitek).

Group 3: (10 non-thermocycled, etched and silane treated IPS eMax and 10 non-thermocycled, etched and silane treated porcelain veneered zirconia crown specimens). A small amount RelyX™ Unicem 2 Automix (3M, ESPE) (*see Figure: 4.*) was placed on the base of the bracket and the bracket was placed onto bonding surface of the embedded crowns. The bracket was compressed onto each crown using a 300g force (Correx force gauge, Bern, Switzerland). Excess resin was removed with an explorer and the resin was light cured for 40 seconds (20 seconds mesial and 20 seconds distal) with a curing light (Ortholux LED, 3M Unitek).

Group 4: (10 non-thermocycled, etched and silane treated IPS eMax and 10 non-thermocycled, etched and silane treated porcelain veneered zirconia crown specimens). A thin layer of Transbond™ XT light cure adhesive primer (3M, Unitek) (*see Figure: 5.*) was applied onto the bonding surface of the crowns and air dried. A small amount of Transbond™ XT adhesive resin (3M, Unitek) (*see Figure: 6.*) was applied to the base of the bracket and the bracket was positioned onto the bonding surface of the crowns. The bracket was compressed onto each crown using a 300g force (Correx force gauge, Bern, Switzerland). The excess resin was removed with an explorer and the resin was light cured for 40 seconds (20 seconds mesial and 20 seconds distal) with a curing light (Ortholux LED, 3M Unitek) (*see Figure 7.*).

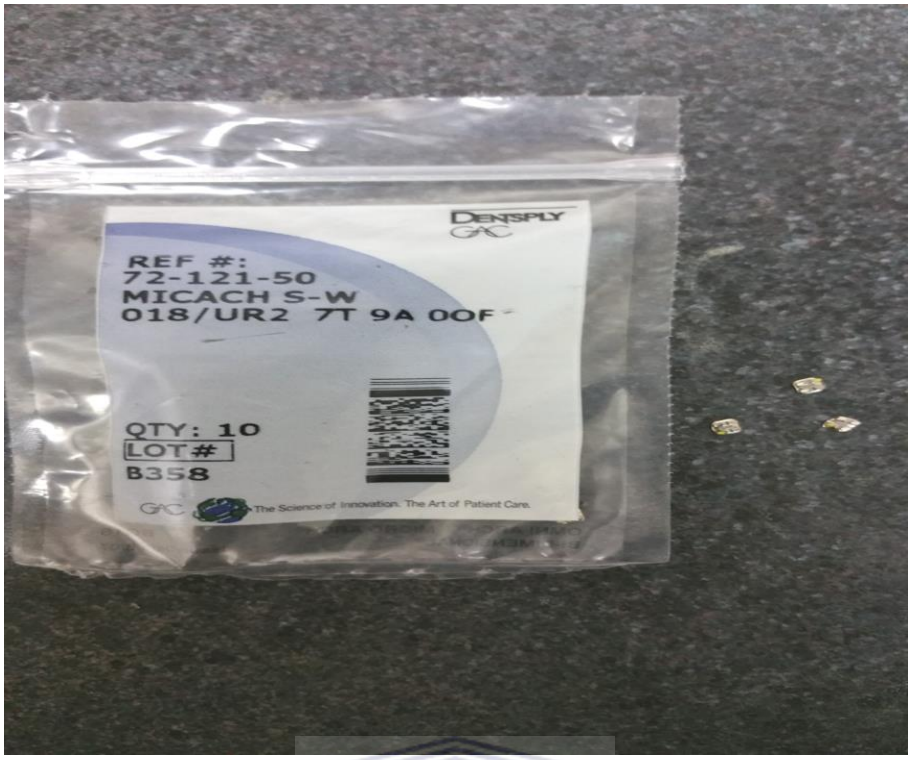

Figure: 3. The metal orthodontic brackets used.

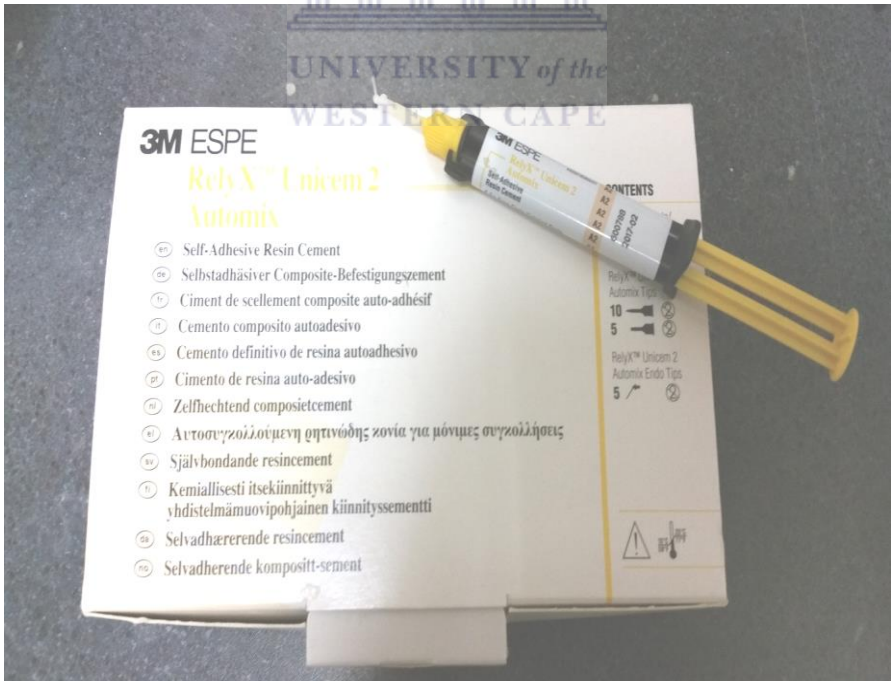

Figure: 4. RelyX Unicem™ 2 self-adhesive resin cement.

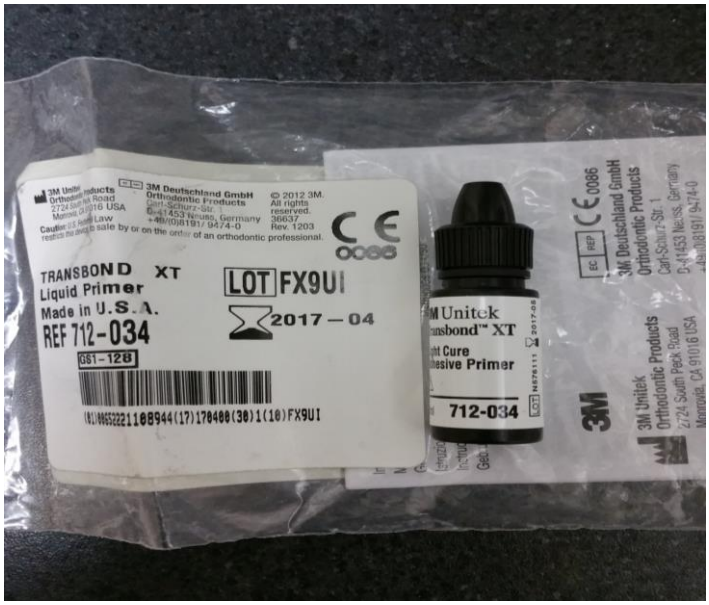

Figure: 5. Transbond™ XT adhesive primer

Figure: 6. Transbond™ XT adhesive resin cement.

Figure: 7. Crown embedded and bonded with a metal bracket. Labelled according to particular groups

3.2. Debonding Procedure

After bonding all samples were stored in distilled water for 24 hours before being submitted to the shear bond strength test. Debonding forces in Newtons was determined by using a testing machine (Instron, Canton, Mass.) operating at a crosshead speed of 1 mm/min. The embedded ceramic crown and adhesively fixed bracket was positioned in the machine so that the bracket slot was aligned horizontally. A knife-edged shearing rod was used to deliver a shearing force at the bracket-ceramic interface (*see Figure: 8.*).

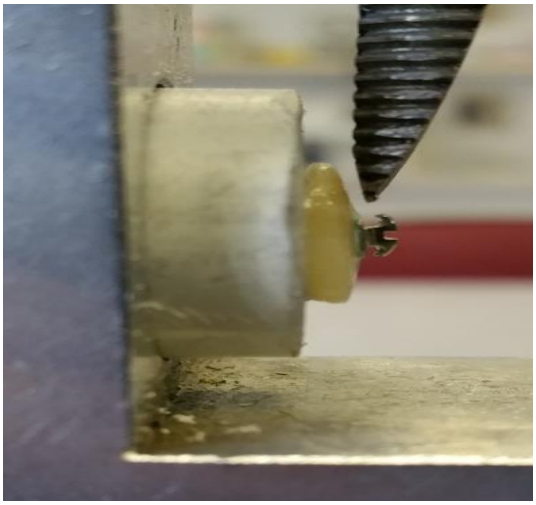

Figure: 8. The knife-edged rod of the shearing machine positioned at the bracket-ceramic interface.

The shear bond strength (MPa) was determined using the following formula:

Shear bond strength (MPa) =Shearing force (Newtons) / Bracket base surface area (mm^2)

3.3. Evaluation of bracket-crown interface

After debonding the amount adhesive left on the crown surface was examined with an optical microscope at a magnification of 10-25x (*see Figure: 9.*).

Figure: 9. After debonding the surface of the crown was examined.

3.3.1. Adhesive Remnant Index (ARI)

An adhesive remnant index (ARI), by Artun and Bergland (1984), was used to evaluate the adhesive remaining on the crown surface after debonding. Description of each category of the adhesive remnant index *(see Table: 1.).*

3.3.2. Porcelain Fracture Index (PFI)

All composite remnants were then removed from the ceramic crown specimens (with ARI scores of 1 or more) using scaling instruments after bulk reduction with a twelve-fluted tungsten carbide bur in a slow-speed handpiece. The ceramic surfaces were examined before bonding and the re-examined after debonding with an optical microscope at a magnification of 10-25x to assess damage which may have occurred to the porcelain and recorded using the Porcelain Fracture Index (PFI) (*see Table 2.*). For this study we used the Porcelain Fracture Index (PFI) developed by Bourke *et al* (1999). The

PFI uses a 0 to 3 or a 4-point scale to evaluate the quality or integrity of the porcelain surface after the removal of the residual adhesive.

Chapter 4

Results

Results

4.1. Presentation of raw data

Each of the adhesive resin cement/ crown combinations were grouped and presented in groups (*see Tables: 5-16.*). The abbreviations used are explained in *Table: 4.*

#	Specimen number
T.	Thermocycled specimens
NT.	Non-Thermocycled specimens
SBS-N	Shear bond Strength value in Newtons
SBS-MPa	Shear bond Strength value in Mega Pascals
ARI	Adhesive Remnant Index
PFI	Porcelain fracture Index
R-X U	RelyX™ Unicem 2 dual-cure resin cement (3M, ESPE)
Tb XT	Transbond™ XT light cure adhesive resin cement (3M, Unitek)
E	IPS eMax crown
Z	Porcelain-veneered zirconia crown

Table: 4. List of abbreviations used.

Group 1: Results of the Thermocycled eMax/ RelyX™ Unicem 2 and Porcelain-veneered zirconia crown/RelyX™ Unicem 2 adhesive combinations (*see Table: 5.*).

	Adhesive Agent	Type of crown	T./NT.	SBS N	SBS MPa	ARI score	PFI score
1	R-X U	E	T	26.7	3.0	0	0
2	R-X U	E	T	15.4	1.7	1	0
3	R-X U	E	T	46.9	5.2	0	0
4	R-X U	E	T	55.3	6.1	1	0
5	R-X U	E	T	90.9	10.1	1	0
6	R-X U	E	T	73.1	8.1	1	0
7	R-X U	E	T	13.2	1.5	2	0
8	R-X U	E	T	61.2	6.8	2	0
9	R-X U	E	T	20.1	2.2	0	0
10	R-X U	E	T	41.9	4.7	0	0
11	R-X U	Z	T	13.4	1.5	0	0
12	R-X U	Z	T	12.3	1.4	0	0
13	R-X U	Z	T	60.4	6.7	0	0
14	R-X U	Z	T	40	4.4	0	0
15	R-X U	Z	T	17.6	2.0	0	0
16	R-X U	Z	T	52.8	5.9	0	0
17	R-X U	Z	T	23.4	2.6	0	0
18	R-X U	Z	T	12.7	1.4	0	0
19	R-X U	Z	T	0 (defective)	------	--------	-------
20	R-X U	Z	T	0 (defective)	-------	-------	-------

Table: 5. Results: Group 1.

Group 1: Sorted from highest to lowest (SBS) (*see Tables: 6 and 7*).

Adhesive Agent/Type of Crown	SBS-N	SBS-MPa
R-X U/E-T	90.9	10.1
R-X U/E- T	73.1	8.1
R-X U/E-T	61.2	6.8
R-X U/E-T	55.3	6.1
R-X U/E-T	46.9	5.2
R-X U/E-T	41.9	4.7
R-X U/E-T	26.7	3.0
R-X U/E-T	20.1	2.2
R-X U/E-T	15.4	1.7
R-X U/E-T	13.2	1.5
Average/Mean	44.5	4.9
Median	44.4	5

Table: 6.

Adhesive Agent/Type of Crown	SBS-N	SBS-MPa
R-X U/Z-T	60.4	6.7
R-X U/Z-T	52.8	5.9
R-X U/Z-T	40	4.4
R-X U/Z-T	23.4	2.6
R-X U/Z-T	17.6	2.0
R-X U/Z-T	13.4	1.5
R-X U/Z-T	12.7	1.4
R-X U/Z-T	12.3	1.4
R-X U/Z-T	0	0
R-X U/Z-T	0	0
Average/Mean	29.1	3.2
Median	20.5	2.3

Table: 7.

Group 2: Results of Thermocycled eMax/Transbond XT adhesive and Porcelain-veneered zirconia crown/Transbond XT adhesive combinations (*see Table 8.*)

#	Adhesive Agent	Type of crown	T./NT.	SBS N	SBS MPa	ARI score	PFI score
21	Tb XT	E	T	38.3	4.3	0	0
22	Tb XT	E	T	25.1	2.8	0	0
23	Tb XT	E	T	54.5	6.1	0	0
24	Tb XT	E	T	72.1	8.0	0	0
25	Tb XT	E	T	56.7	6.3	0	0
26	Tb XT	E	T	87.2	9.7	0	0
27	Tb XT	E	T	25.5	2.8	0	0
28	Tb XT	E	T	42.1	4.7	0	0
29	Tb XT	E	T	44.2	4.9	0	0
30	Tb XT	E	T	15.6	1.7	0	0
31	Tb XT	Z	T	15.8	1.8	0	0
32	Tb XT	Z	T	32.7	3.6	0	0
33	Tb XT	Z	T	27.6	3.1	0	0
34	Tb XT	Z	T	70.9	7.9	0	0
35	Tb XT	Z	T	55.9	6.2	0	0
36	Tb XT	Z	T	26.7	3.0	0	3
37	Tb XT	Z	T	60.9	6.8	0	0
38	Tb XT	Z	T	49.2	5.5	0	0
39	Tb XT	Z	T	65.5	7.3	0	0
40	Tb XT	Z	T	51.1	5.7	0	0

Table: 8.

Group 2: sorted from highest to lowest (SBS) (*see Tables: 9 and 10.*).

Adhesive agent/ Type of crown	SBS-N	SBS-MPa
Tb XT/E-T	87.2	9.7
Tb XT/E-T	72.1	8.0
Tb XT/E-T	56.7	6.3
Tb XT/E-T	54.5	6.1
Tb XT/E-T	44.2	4.9
Tb XT/E-T	42.1	4.7
Tb XT/E-T	38.3	4.3
Tb XT/E-T	25.5	2.8
Tb XT/E-T	25.1	2.8
Tb XT/E-T	15.6	1.7
Average/Mean	46.1	5.1
Median	43.2	4.8

Table: 9.

Adhesive agent/Type of crown	SBS-N	SBS-MPa
Tb XT/Z-T	70.9	7.9
Tb XT/Z-T	65.5	7.3
Tb XT/Z-T	60.9	6.8
Tb XT/Z-T	55.9	6.2
Tb XT/Z-T	51.1	5.7
Tb XT/Z-T	49.2	5.5
Tb XT/Z-T	32.7	3.6
Tb XT/Z-T	27.6	3.1
Tb XT/Z-T	26.7	3.0
Tb XT/Z-T	15.8	1.8
Average/Mean	45.8	5.1
Median	50.2	5.4

Table: 10.

Group 3: Results of the Non –Thermocycled eMax/RelyX Unicem 2 and Porcelain-veneered zirconia Crown/ RelyX Unicem 2 adhesive combinations (*see Table: 11.*).

#	Adhesive Agent	Type of crown	T./NT.	SBS N	SBS MPa	ARI score	PFI score
41	R-X U	E	NT	40.3	4.5	2	0
42	R-X U	E	NT	65.3	7.3	3	0
43	R-X U	E	NT	26.6	3.0	0	0
44	R-X U	E	NT	45.8	5.1	3	0
45	R-X U	E	NT	22.2	2.5	1	0
46	R-X U	E	NT	49.4	5.5	1	0
47	R-X U	E	NT	52.2	5.8	2	0
48	R-X U	E	NT	51.4	5.7	3	0
49	R-X U	E	NT	55.1	6.1	3	0
50	R-X U	E	NT	45.5	5.1	3	0
51	R-X U	Z	NT	38.8	4.3	0	0
52	R-X U	Z	NT	40.8	4.5	0	0
53	R-X U	Z	NT	27.2	3.0	0	0
54	R-X U	Z	NT	52.5	5.8	0	0
55	R-X U	Z	NT	34.6	3.8	0	0
56	R-X U	Z	NT	68.2	7.6	0	0
57	R-X U	Z	NT	58.5	6.5	0	0
58	R-X U	Z	NT	65.9	7.3	0	0
59	R-X U	Z	NT	81	9	0	0
60	R-X U	Z	NT	0 (defective)	-------	------	------

Table: 11.

Group 3: sorted from highest to lowest (SBS) (*see Tables: 12 and 13.*).

Adhesive Agent/Type of crown	SBS-N	SBS-MPa
R-X U/E-NT	65.3	7.3
R-X U/E-NT	55.1	6.1
R-X U/E-NT	52.2	5.8
R-X U/E-NT	51.4	5.7
R-X U/E-NT	49.4	5.5
R-X U/E-NT	45.8	5.1
R-X U/E-NT	45.5	5.1
R-X U/E-NT	40.3	4.5
R-X U/E-NT	26.6	3.0
R-X U/E-NT	22.2	2.5
Average/Mean	45.5	5.1
Median	47.6	5.3

Table: 12.

Adhesive agent/Type of crown	SBS-N	SBS-MPa
R-X U/Z-NT	81	9
R-X U/Z-NT	68.2	7.6
R-X U/Z-NT	65.9	7.3
R-X U/Z-NT	58.5	6.5
R-X U/Z-NT	52.5	5.8
R-X U/Z-NT	40.8	4.5
R-X U/Z-NT	38.8	4.3
R-X U/Z-NT	34.6	3.8
R-X U/Z-NT	27.2	3.0
R-X U/Z-NT	0	0
Average/Mean	51.9	5.8
Median	52.5	5.8

Table: 13.

Group 4: Results of the Non-Thermocycled eMax/Transbond XT adhesive and Porcelain-veneered zirconia crown/ Transbond XT adhesive combinations (*see Table: 14.*).

#	Adhesive Agent	Type of crown	T./NT.	SBS N	SBS MPa	ARI score	PFI Score
61	Tb XT	E	NT	65.4	7.3	3	0
62	Tb XT	E	NT	172.2	19.1	0	0
63	Tb XT	E	NT	63.3	7.0	0	0
64	Tb XT	E	NT	65.7	7.3	0	0
65	Tb XT	E	NT	43.7	4.9	3	0
66	Tb XT	E	NT	68.8	7.6	3	0
67	Tb XT	E	NT	43.4	4.8	3	0
68	Tb XT	E	NT	103.9	11.5	0	0
69	Tb XT	E	NT	50.6	5.6	1	0
70	Tb XT	E	NT	50.2	5.6	0	0
71	Tb XT	Z	NT	57.3	6.4	0	0
72	Tb XT	Z	NT	52.2	5.8	2	0
73	Tb XT	Z	NT	49.6	5.5	3	0
74	Tb XT	Z	NT	52.2	5.8	3	0
75	Tb XT	Z	NT	62.5	6.9	1	0
76	Tb XT	Z	NT	54.2	6.0	0	0
77	Tb XT	Z	NT	41.2	4.6	3	0
78	Tb XT	Z	NT	72.1	8.0	3	0
79	Tb XT	Z	NT	71.9	8.0	0	0
80	Tb XT	Z	NT	59.8	6.6	3	0

Table: 14.

Group 4: sorted from highest to lowest (SBS) (*see Table 15 and 16.*).

Adhesive agent/Type of crown	SBS-N	SBS-MPa
Tb XT/E-NT	172.2	19.1
Tb XT/E-NT	103.9	11.5
Tb XT/E-NT	68.8	7.6
Tb XT/E-NT	65.7	7.3
Tb XT/E-NT	65.4	7.3
Tb XT/E-NT	63.3	7.0
Tb XT/E-NT	50.6	5.6
Tb XT/E-NT	50.2	5.6
Tb XT/E-NT	43.7	4.9
Tb XT/E-NT	43.4	4.8
Average/Mean	72.7	8.1
Median	64.4	7.2

Table: 15.

Adhesive agent/Type of Crown	SBS-N	SBS-MPa
Tb XT/Z-NT	72.1	8.0
Tb XT/Z-NT	71.9	8.0
Tb XT/Z-NT	62.5	6.9
Tb XT/Z-NT	59.8	6.6
Tb XT/Z-NT	57.3	6.4
Tb XT/Z-NT	54.2	6.0
Tb XT/Z-NT	52.2	5.8
Tb XT/Z-NT	52.2	5.8
Tb XT/Z-NT	49.6	5.5
Tb XT/Z-NT	41.2	4.6
Average/Mean	57.3	6.4
Median	55.75	6.2

Table: 16.

4.2. Statistical Analysis of shear bond strengths in Newtons (N) and Mega Pascals (MPa)

The following layout for IPS eMax (E) and Zirconia (Z**)** crowns were used.

Group A-

Thermocycled (T); IPS eMax (E) and Zirconia (Z**)** crowns bonded with RelyX^{TM} Unicem 2 (R-X U)

Group B-

Thermocycled (T); IPS eMax (E) and Zirconia (Z) crowns bonded with Transbond^{TM} XT (Tb XT)

Group C-

Non-Thermocycled (NT); IPS eMax (E) and Zirconia (Z**)** crowns bonded with RelyX^{TM} Unicem (R-X U)

Group D-

Non-Thermocycled (NT); eMax (E) and Zirconia (Z) crowns bonded with Transbond^{TM} XT (Tb XT)

This is also known as a factorial layout and the aim was to have ten replicates for each crown/adhesive treatment combination, eight combinations in total. It was analysed as a one-way analysis of variance, ANOVA.

The data in Newtons (N) which was obtained from the shear bond strength test performed with the Instron testing machine was converted into Mega Pascals using the following equation:

Shear bond strength (MPa) =Shearing force (Newtons) / Bracket base surface area (mm^2)

The orthodontic bracket used had a surface area of 9mm^2, as confirmed by the manufacturer.

A/C T/NT	Sample size	Average N	Median N	S.D	Average MPa	Median MPa	S.D
R-X U/ E-T	10	44.46	44.55	26.07	4.94	4.95	2.89
R-X U/ Z-T	10	29.14	20.70	19.36	3.24	2.30	2.14
TbXT/ E-T	10	46.17	43.20	22.17	5.13	4.80	2.47
TbXT/ Z-T	10	45.81	50.40	18.72	5.13	5.60	2.08
R-X U/ E-NT	10	45.54	47.70	12.94	5.09	5.30	1.43
R-X U/ Z-NT	10	51.80	52.20	17.88	5.76	5.80	2
TbXT/ E-NT	10	72.63	64.35	39.12	8.07	7.15	4.33
TbXT/ Z-NT	10	57.24	55.80	9.69	6.36	6.20	1.07

Table:17: Data analysed

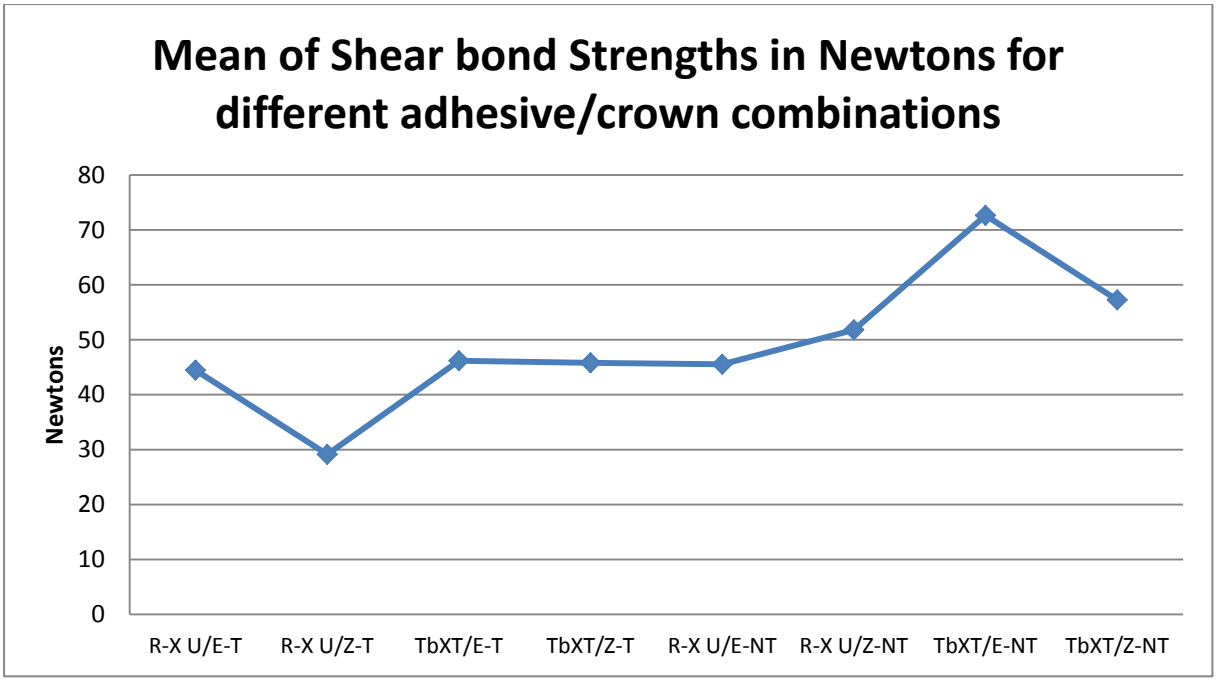

Figure: 10: Line graph: Mean shear bond strengths in Newtons for the 8 combinations.

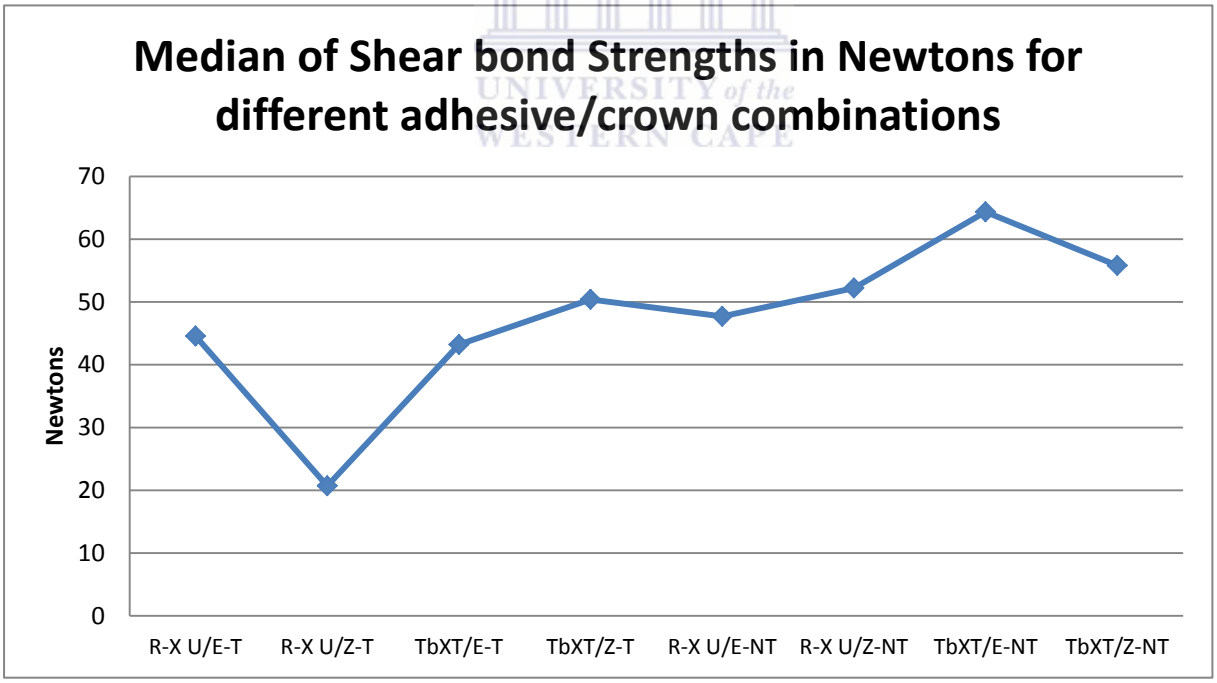

Figure: 11: Line graph showing the Median shear bond strengths in Newtons for each adhesive/crown combination.

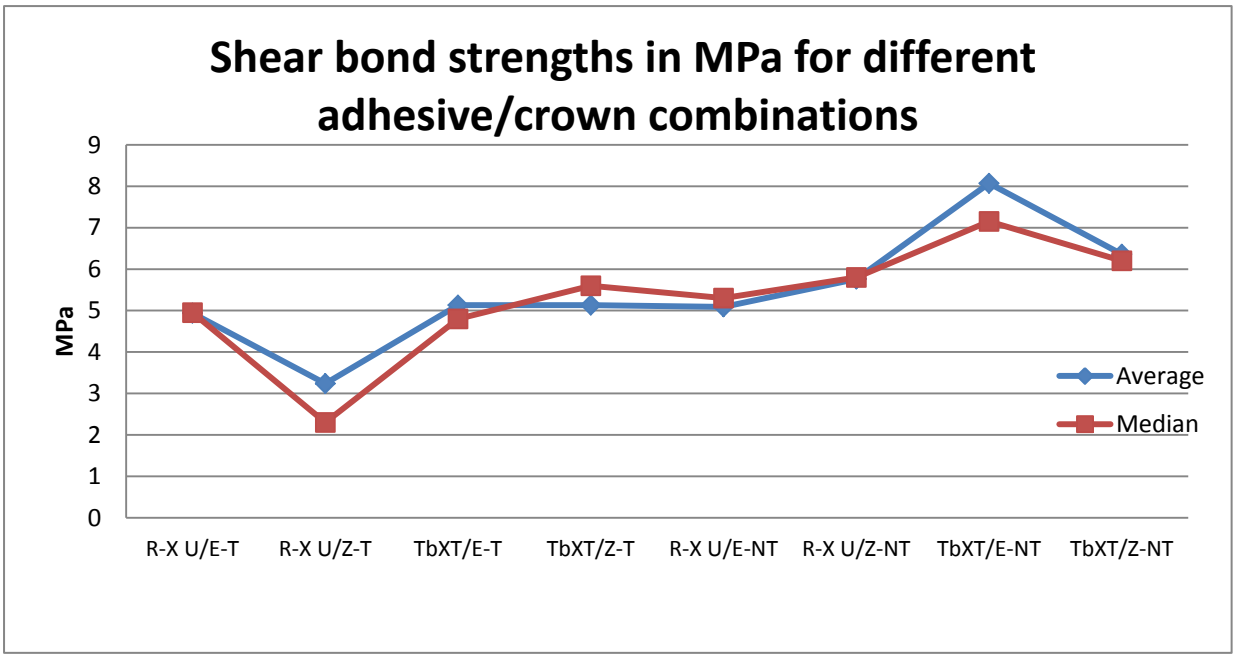

Figure: 12: Line graph showing the shear bond strengths in Mega Pascals (MPa) for each adhesive/crown combination.

The red line represents the median.

The blue line represents the average/mean.

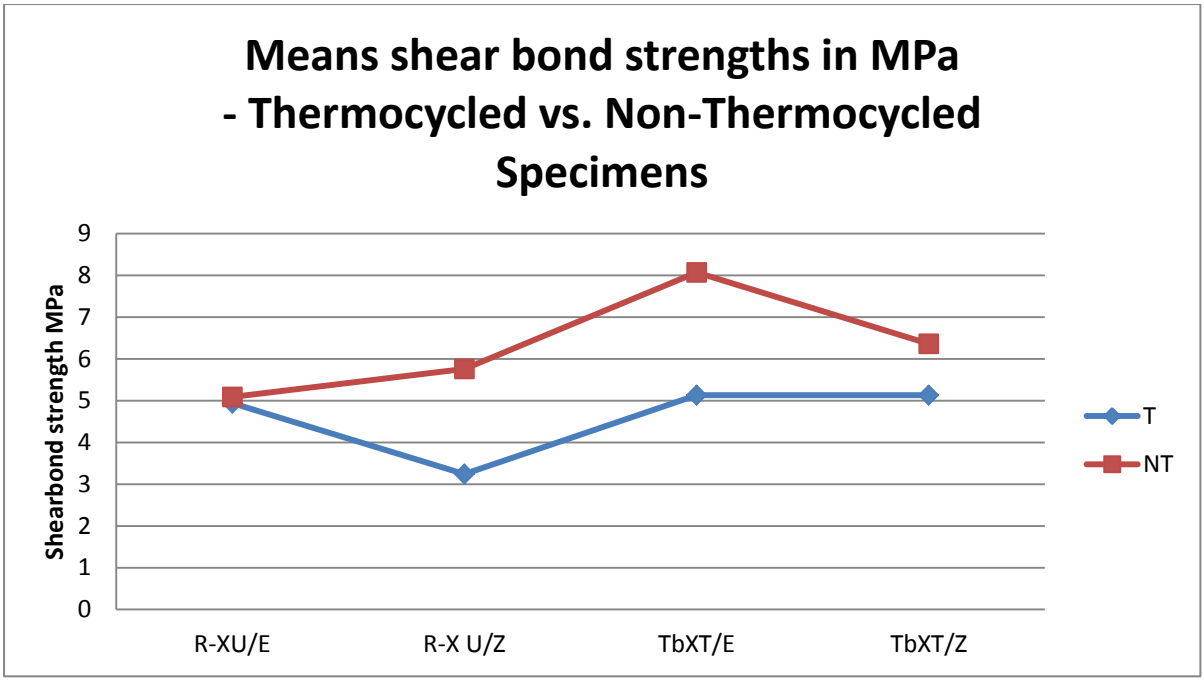

Figure: 13: Line graph showing the Mean shear bond strengths in MPa for the adhesive/crown combinations.

The blue line shows the Thermocycled specimens.

The red line shows the Non –Thermocycled specimens.

For a graphical display of the SBS determinations for each of the eight adhesive/crown combinations a Stem-and-Leaf was constructed (see Tables: 18-21.)

The bold digits in the **Leaves**-columns represents the individual observations (SBS in Newtons).

E-max- E	
Thermo Cycled	Rely X Unicem

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9	1	1
8		
7	3	1
6	1	1
5	5	1
4	27	2
3		
2	07	2
1	35	2
0		

E-max – E	
Thermo cycled	Transbond XT

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8	7	1
7	2	1
6		
5	57	2
4	27	2
3	8	1
2	56	2
1	6	1
0		

Distribution very flat	Distribution very flat

*Table: 18. Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** ≡ Frequency).*

E-max - E	
Non-Thermo Cycled	Rely X Unicem

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7		
6	**5**	1
5	**125**	3
4	**0669**	4
3		
2	**27**	2
1		
0		

E-max - E	
Non-Thermo cycled	Transbond XT

Stem-	Leaves	Freq
17	**2**	1
16		
15		
14		
13		
12		
11		
10	**4**	1
9		
8		
7		
6	**3569**	4
5	**01**	2
4	**34**	2
3		
2		
1		
0		

Dispersion narrow	Contains two outliers 172 &104

*Table 19. Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** ≡ Frequency).*

Zirconia – Z	
Thermo Cycled	Rely X Unicem

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7		
6	**0**	1
5	**3**	1
4	**0**	1
3		
2	**3**	1
1	**238**	3
0	**003**	3

Zirconia - Z	
Thermo cycled	Transbond XT

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7	**1**	1
6	**16**	2
5	**16**	2
4	**9**	1
3	**3**	1
2	**78**	2
1	**6**	1
0		

Distribution; skewed towards the larger values	Dispersion wide

*Table: 20: Stem-and-Leaf Diagram for two of the eight treatment combinations (**Freq** ≡ Frequency).*

Zirconia – Z	
Non-Thermo Cycled	Rely X Unicem

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8	**1**	1
7		
6	**68**	2
5	**39**	2
4	**1**	1
3	**59**	2
2	**7**	1
1		
0	**0**	1

Zirconia - Z	
Non-Thermo cycled	Transbond XT

Stem-	Leaves	Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7	**22**	2
6	**03**	2
5	**02247**	5
4	**1**	1
3		
2		
1		
0		

Dispersion wide	Dispersion narrow

*Table: 21: Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** ≡ Frequency).*

Group	SBS N	**Group**	SBS Mpa
R-X U-Z-T	20.70	R-X U-Z-T	2.30
Tb XT-E-T	43.20	Tb XT-E-T	4.80
R-X U-E-T	44.55	R-X U-E-T	4.95
R-X U-E-NT	47.70	R-X U-E-NT	5.30
Tb XT-Z- T	50.40	Tb XT-Z-T	5.60
R-X U-Z-NT	52.20	R-X U-Z-NT	5.80
Tb XT-Z-NT	55.80	Tb XT-Z-NT	6.20
Tb XT-E-NT	64.35	Tb XT-E-NT	7.15

*Table: 22.: The SBS in Newtons and MPa **Medians** of the eight adhesive/crown combinations sorted from small to large.*

Group	SBS N	**Group**	SBS Mpa
R-X U-Z-T	29.14	R-X U-Z-T	3.24
R-X U-E-T	44.46	R-X U-E-T	4.94
R-X U-E-NT	45.54	R-X U-E-NT	5.06
Tb XT-Z-T	45.81	Tb XT-Z-T	5.09
Tb XT-E-T	46.17	Tb XT-E-T	5.13
R-X U-Z-NT	51.80	R-X U-Z-NT	5.76
Tb XT-Z-NT	57.24	Tb XT-Z-NT	6.36
Tb XT-E-NT	72.63	Tb XT-E-NT	8.07

Table: 23.: The SBS in Newtons and MPa ***Means*** *of the eight adhesive/crown combinations sorted from small to large.*

The order for the two measures are exactly the same because the two units are linearly related

In the two Tables above (*see Tables: 22 and 23.*) there is a strong correspondence between the two rankings (Medians and the Means). The smallest shear bond (SBS) values remain the same as well

as the last three at the higher end of the spectrum. Towards the middle section of the rankings R-X U-E-T, R-X U-E-NT, Tb XT-Z-T and Tb XT-E-T are only slightly rearranged.

Group		R-X U-Z-T	Tb XT-E-T	R-X U-E-T	R-X U-E-NT	Tb XT-Z-T	R-X U-Z-NT	Tb XT-Z-NT	Tb XT-E-NT
Median	**SBS N**	20.70	43.20	44.55	47.70	50.40	52.20	55.80	64.35
Mean	**SBS N**	29.14	46.17	44.46	45.54	45.81	51.80	57.24	72.63

Table: 24: The Medians and Means of the SBS in Newtons of the eight different adhesive/crown combinations.

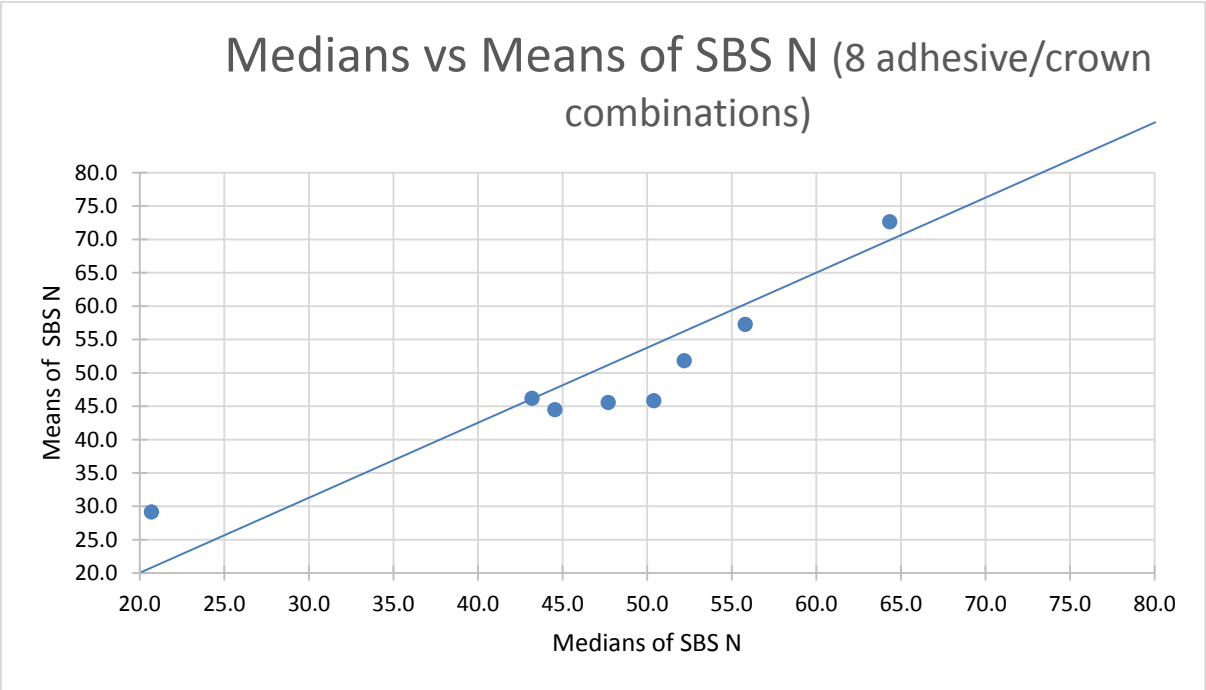

Figure: 14: Scatter plot of the Medians vs Means of the SBS in Newtons of the eight adhesive/crown combinations.

In the above graph of the eight adhesive/crown combinations Medians vs Means of SBS N are displayed and the symmetry of the statistical distribution of SBS-N is confirmed. The smallest SBS N is much less than the second smallest observation.

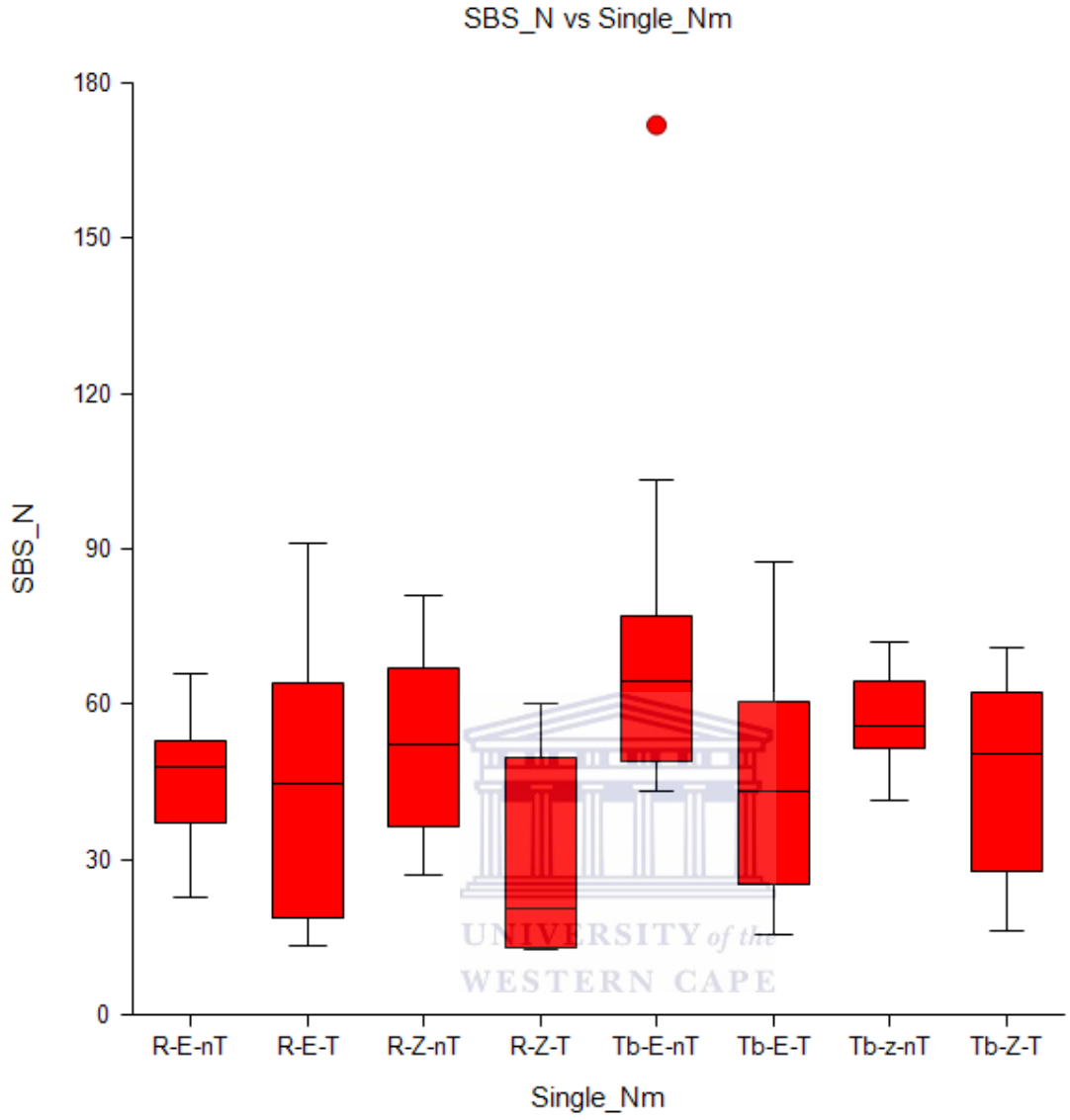

Figure: 15: Side by side Box-and-Whisker Plots of the SBS (N) values for the eight adhesive/crown combinations (Single combination Names).

Each box represents the interquartile area (50% of the readings for each combination).

The red line in each box represents the medians.

The red dot is representative of an extreme value obtained.

The wide and overlapping dispersion (interquartile ranges, the red boxes) of the adhesive/crown combinations will consequently lessen the probability of significant differences between the eight adhesive/crown combinations. The excessive outlier in the treatment combination Tb XT-E-NT would not affect the analysis because non-parametric methods were used.

From the Kruskal-Wallis test with respect to the Medians, the following Table (*see Table: 25.*) (for all the pairwise comparisons) can be constructed ($p < 0.05$).

Group	R-Z-T	Tb-E-T	R-E-T	R-E-NT	Tb-Z-T	R-Z-NT	Tb-Z-NT	Tb-E-NT
Median	20.7	43.2	44.55	47.7	50.4	52.2	55.8	64.35

Table: 25. Medians in Newtons of the eight adhesive/crown combinations.

From this Table we could learn that SBS N for the treatment R-X U-Z-T which has a median of 20.7 N is different from Tb XT-E-T, R-X U-E-T, R-X U-E-NT, Tb XT-Z-T, R-X U-Z-NT, Tb XT-Z-NT and Tb XT-E-NT with corresponding medians 43.2, 44.55, 47.7, 50.4, 52.2, 55.8 and 64.35. Using the Bonferroni Test for Medians, it implies that those adhesive/crown combinations linked by the dark horizontal line do not differ significantly.

Relaxing the significance level (p-value) somewhat one could arrive at the following Table (*see Table 26.*).

Group	R-Z-T	Tb-E-T	R-E-T	R-E-NT	Tb-Z-T	R-Z-NT	Tb-Z-NT	Tb-E-NT
Median	20.7	43.2	44.55	47.7	50.4	52.2	55.8	64.35

Table:26. Relaxing p-value

This figure is corresponding to the Scatter plot (*see Figure: 14.*). From this follows that Tb XT-E-NT (Transbond XT and e-Max and not Thermocycled) yielded the maximum SBS, but after being Thermocycled it dropped to the second lowest position of SBS N (a fall of six positions). Tb XT-Z-NT, R-X U-Z-NT and R-X U-E-NT dropped two positions, five positions and one position respectively.

R-X U-Z-NT, Tb XT-Z-NT, Tb XT-E-NT three of the not Thermocycled treatments are in the top positions, showing the adverse effects of Thermocycling. The treatment R-X U-Z-T (20.7) had the lowest SBS.

4.3. Shear bond strength comparisons

The results after debonding were sorted into maximum and minimum values and the means and medians were calculated (*see Tables: 6-16.*).

Table: 17. expresses the mean Shear bond strength (SBS) of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa) and their respective standard deviations (S.D).

Figures: 10 and 11. are line graphs showing the mean and median shear bond strengths (SBS) values in Newtons (N) of each adhesive/crown combination. Figure: 12. are superimposed line graphs comparing the mean and median shear bond strength (SBS) values of each adhesive/crown combination in Mega Pascals (MPa). Figure: 13. are superimposed line graphs comparing the mean shear bond strength (SBS) values in Mega Pascals (MPa) of the thermocycled and non-thermocycled adhesive/crown combinations.

For a graphical display of the shear bond strength (SBS) values in Newtons (N) for each of the 8 adhesive/crown combinations Stem-and-Leaf diagrams were constructed. Table: 18. displays a flat distribution of shear bond strength (SBS) values for both the R-X U/E-T and the Tb XT/E- T combinations. Table: 19. displays a narrow dispersion of shear bond strength (SBS) values for the R-X U/E-NT combination and the Tb XT/E-NT combination contained two outliners (104 N and 172 N).

Table: 20. displays a distribution of shear bond strength (SBS) values which is skewed towards the larger values for the R-X U/Z-T combination and a wide dispersion of shear bond strength (SBS) values for the TbXT/Z-T combination. Table: 21. displays a wide dispersion of shear bond strength (SBS) values for the R-X U/Z-NT combination and a narrow dispersion of shear bond strength (SBS) values for the TbXT/Z-NT combination.

Table: 22. represents the medians of the shear bond strength (SBS) values of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa). Table: 23. represents the Means of the shear bond strength (SBS) values of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa). The order in these two tables is exactly the same because the two units are linearly related.

Figure: 14. shows a scatter plot of the Medians and Means of the shear bond strength (SBS) values in Newtons (N) of the 8 adhesive/crown combinations (*see Table: 24.*) and the symmetry of the statistical distribution of the shear bond strength (SBS) values in Newtons (N) is confirmed.

4.3.1. Rely X™ Unicem 2 self-adhesive resin

Comparative shear bond strengths of the 2 all ceramic non-thermocycled crowns

The results after debonding were compared. The mean shear bond strength for this adhesive bonded to the all ceramic non-thermocycled crowns ranged from a low of 5.1 MPa (45.5 Newtons) when brackets were bonded to the eMax crowns to a high of 5.8 MPa (51.9 Newtons) when brackets were bonded to the porcelain veneered zirconia crowns. The standard deviation in the shear bond strength values displayed in the 2 groups was small even though the range between the maximum and minimum values was large.

Rely X Unicem 2 self-adhesive resin/non-thermocycled eMax crown combination

The Rely-X Unicem 2 self-adhesive resin displayed the sixth highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled eMax crowns (Transbond™ XT adhesive resin cement displayed the 5 highest shear bond strength values). The shear bond strength values for this combination ranged from a minimum of 2.5 MPa (22.2 Newtons) to a maximum of 7.3 MPa (65.3 Newtons) with a mean value of 5.1 MPa (45.5 Newtons) *(see Table: 12.)*. This combination displayed a standard deviation of 1.43 *(see Table: 17.).*

Rely-X Unicem 2 self-adhesive resin/non-thermocycled porcelain veneered zirconia crown combination

The Rely-X Unicem 2 self-adhesive resin displayed the highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled porcelain veneered zirconia crowns. The shear bond strength values for this combination ranged from a minimum of 0 MPa (the bracket debonded without registering a value on the shearing machine) to a maximum of 9 MPa (81 Newtons) with a mean value of 5.8 MPa (51.9 Newtons) (*see Table: 13*.). This combination displayed a standard deviation of 2 *(see Table: 17.).*

4.3.2. Transbond XT adhesive resin

Comparative shear bond strengths of the 2 all ceramic non-thermocycled crowns

The results after debonding were compared. The mean shear bond strength for this adhesive bonded to the all ceramic non-thermocycled crowns ranged from a low of 6.4 MPa (57.3 Newtons) when brackets were bonded to the porcelain veneered zirconia crowns to a high of 8.1 MPa (72.7 Newtons) when brackets were bonded to the eMax crowns.

Transbond XT adhesive resin/non-thermocycled eMax crown combination

The Transbond XT adhesive resin displayed the highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled eMax crowns. The shear bond strength values for this combination ranged from a low of 4.8 MPa (43.4 Newtons) to a maximum of 19.1 MPa (172.2 Newtons) with a mean value of 8.1 MPa (72.7 Newtons) *(see Table: 15.).* This combination displayed a standard deviation of 4.33 *(see Table: 17.).*

Transbond XT adhesive resin/non-thermocycled porcelain veneered zirconia crown combination

The Transbond XT adhesive resin displayed the second highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled porcelain veneered zirconia crowns. The shear bond strength values for this combination ranged from a low of 4.6 MPa (41.2 Newtons) to a maximum of 8 MPa (72.1 Newtons) with a mean value of 6.4 MPa (57.3 Newtons) *(see Table: 16.).* This combination displayed a standard deviation of 1.07 *(see Table: 17.).*

The side by side Box-and-Whisker plots of the shear bond strengths *(see Figure: 15.)* show wide and overlapping dispersions of the treatment combinations which consequently lessen the probability of significant differences between the treatment combinations. According to the Kruskal-Wallis test *(see Tables: 25 and 26.)* ($p < 0.05$), and the Bonferroni Test the non-thermocycled crown/adhesive resin combination do not differ significantly.

4.4. The Adhesive Remnant index (ARI) And Porcelain Fracture Index (PFI) results:

Analysis of the ARI and the PFI:

Group 1	Adhesive Remnant Index (ARI)					Porcelain Fracture Index (PFI)				
	0	1	2	3	Average	0	1	2	3	Average
R-X U/E-T	4	4	2	0	0.8	10	---	---	---	0
R-X U/Z-T	10	0	---	----	0	10	---	---	---	0
Group 2										
Tb XT/E-T	10	---	---	---	0	10	---	---	---	0
Tb XT/Z-T	10	---	---	---	0	9	---	---	1	0.3
Group 3										
R-X U/E-NT	1	2	2	5	2.1	10	---	---	---	0
R-X U/Z-NT	10	0	---	---	0	10	---	---	---	0
Group 4										
Tb XT/E-NT	5	1	0	4	1.3	10	---	---	---	0
Tb XT/Z-NT	3	1	1	5	1.8	10	---	---	---	0

Table: 27: Adhesive Remnant Index (ARI) and Porcelain Fracture Index (PFI) (sorted)-Groups 1,2,3,4.

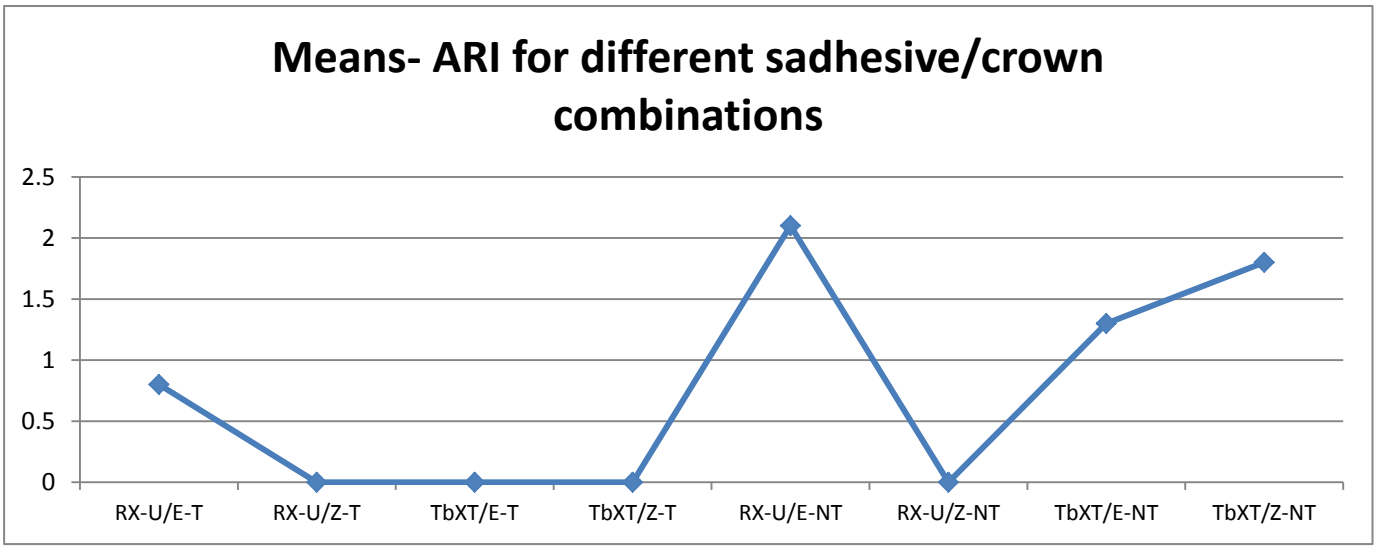

Figure: 16: Line graph showing the Means of the Adhesive Remnant Index (ARI) for the different adhesive/crown combinations.

For the next two measurements: ARI-score and PFI-score the values consists discrete integers. For the ARI-score the following summary table provides a list of similarities and differences.

Group	R-X U-Z-T	Tb XT-E-T	Tb XT-Z-T	R-X U-Z-NT	R-X U-E-T	Tb XT-E-NT	Tb XT-Z-NT	R-X U-E-NT
Mean	0	0	0	0	0.8	1.3	1.8	2.1

Table: 28. Mean ARI-scores for the eight adhesive/crown combinations.

For the last measurement, PFI only one observation was different from zero, therefore all eight medians were equal to zero.

4.4. Conclusions

Despite the small sample sizes and the overlapping dispersions, the study gives an indication of a trend, in the Shear Bond Strengths (SBS). The two units (Newtons and Mpa) differ only for a linear transformation of nine (9), therefore the statistical outcomes hold for both units. The detrimental influence of Thermocycling was observed in the measured shear bond strengths.

Chapter 5

Discussion

Discussion

Introduction

Optimal bracket adhesion to the bonding surface of porcelain crowns is always of concern to orthodontists because the forces applied during treatment should not result in bond failure. Glazed porcelain is not an appropriate surface for resin penetration and orthodontic bonding due to the physical properties of glazed surfaces and the chemical properties of bonding resins (Smith *et al* 1988). Recommended surface treatment methods can be time consuming or even harmful to soft tissues. Hydroflouric acid (HFA) etching is an effective surface treatment for porcelain-composite bonding (Kocadereli *et al* 2001). However, the risk of soft tissue burns and toxic effects of HFA requires extreme care during intraoral application, causing many orthodontists to be hesitant in its use (Zachrisson *et al* 1996, Lamour *et al* 2006).

In the present study, due to the potential toxicity of HFA, the ceramic surfaces were treated with 37% phosphoric acid and a silane coupling agent. Etching of porcelain surfaces with phosphoric acid alone does not provide adequate shear bond strength, capable of resisting the forces applied during orthodontic treatment (Guimaraes *et al* 2012). Anecdotal evidence suggests brackets bonded with silane coupling agents and phosphoric acid or hydrofluoric acid has sufficient bond strength for orthodontic treatment (Nebbe and Stein 1996, Schmage *et al* 2003, Ajlouni *et al* 2005, Lamour *et al* 2006, Abu Alhaija and Al-Wahadani 2007). Phosphoric acid does not etch porcelain, and it does not produce physical or topographical changes in the porcelain surface. Instead, phosphoric acid has the effect of neutralising the alkalinity of the adsorbed water layer, which is present on all porcelain restorations in the oral cavity. This enhances the chemical activity of the silane coupling agents which are subsequently applied (Wolf *et al* 1993, Samruajbenjakul and Kukiattrakoon 2009, Purmal *et al* 2013). Silane coupling agents have been reported to enhance bond strength to porcelain surfaces (Wood *et al* 1986, Kao 1988,Winchester and Orth 1991,Newman 1994, Bourke and Rock 1999,

Kocadereli *et al* 2001) . The silane reacts with the silica within the porcelain and the organic groups of the bonding resin, thus forming a bridge between the two materials (Newman 1994).

Commercially available porcelains are usually similar in chemical formula but have distinct differences in constituents in particle size, and crystalline structure. Therefore, different results are expected regarding bonding to different types of porcelain. In the present study, 40 IPS eMax and 40 porcelain-veneered zirconia crowns were fabricated off a single die and were divided into 4 groups containing 20 crowns each (10 IPS eMax crowns and 10 porcelain-veneered zirconia crowns). The IPS eMax crown and the porcelain-veneered zirconia crowns were chosen because currently they are the most commonly used crowns to restore teeth in the anterior region (Fradaeni 2012). A minimum of 10 specimens is recommended to perform shear bond strength testing (Fox *et al* 1994). However, a sample size greater than 10 specimens per group is recommended for bond strength testing of natural teeth where variations in tooth shape exist (Eliades and Brantley 2000). The maxillary anterior teeth are the teeth most frequently restored with porcelain restorations (Fradaeni 2012). Therefore, in this present study, the lateral incisor tooth form was selected to allow clinical simulation. Some studies (Nebbe and Stein 1996, Schmage *et al* 2003, Purmal *et al* 2013), have used porcelain tabs with flattened surfaces, while some have used porcelain discs (Guimaraes *et al* 2012) and others porcelain denture teeth (Lamour *et al* 2006).

The maximum bond strength which may be achieved to porcelain is not usually required for orthodontic purposes. The ideal bond should be sufficiently strong to endure a course of orthodontic treatment, yet be sufficiently weak at debond to permit restoration of the original porcelain surface.

There are a few scientifically-based recommendations in the literature for minimum orthodontic bracket shear bond strength. Reynolds (1975) recommended a tensile force of 60kg/cm^2 to 80kg/cm^2, while Newman (1994) stated that 14kg/cm^2 was the maximum that should be applied by an orthodontic appliance. Whitlock *et al* (1994) based upon the works of Reynolds (1975), also suggested that 6-8 MPa was adequate for orthodontic attachments and this was used in the present study. The Adhesive Remnant Index and the Porcelain Fracture Index was also examined to establish which regime produced adequate strength for orthodontic bracket attachment to all-ceramic crowns, with least porcelain surface damage following bracket removal.

The overall time required to place an appliance is an important factor in the cost of the treatment (Ajlouni *et al* 2005). Newer, self-adhesive cements have the potential to further simplify the bonding process, that is, by reducing the bonding of orthodontic brackets to a one-step procedure, and thereby reduce chair time and increase cost effectiveness, resulting in increased convenience and reduced costs for the patient (Hayakawa *et al* 1992). Reducing the steps during the bonding process will also reduce the risks of saliva contamination and the effects of humidity which could both have an adverse effect on the bond strength of the cement.

Although there are innumerable protocols for bonding orthodontic brackets to porcelain, there is still no scientific consensus about which of the techniques would be the ideal standard protocol for the purpose of overcoming the two points of contrast mentioned above (Herion *et al* 2010).

Increasing demands of adults for orthodontic treatment and the variation of the results in efficient methods of bonding to ceramics require more investigations. Hence, the purpose of the present study was to test and compare the shear bond strength and the resultant failure pattern of 2 types of resin cements (a self-adhesive, dual cured resin cement and a 2-step bonding, light cured resin cement) to etched and silane treated ceramic crowns.

Additionally, a further aim of this study was to substitute the etching using hydrofluoric acid which is noxious and potentially harmful. Instead, etching with 37% ortho-phosphoric acid and silane coupling application as a pre-treatment conditioning procedure of the ceramic crown surfaces before bonding was used.

Furthermore, examining the effect of thermocycling (ie. some the ceramic specimens were thermocycled to simulate the oral environment prior to bonding of the orthodontic bracket to the ceramic crown) on the shear bond strengths, which many studies have not included, was also documented.

The first objective of this study was to compare the shear bond strengths of the dual-cured, self-adhesive resin cement (RelyX™ Unicem 2) and the light-cured, 2 step bonding resin cement (Transbond™ XT light cure adhesive primer and Transbond™ XT adhesive resin cement) (3M, Unitek) to the pre-treated (35% ortho-phosphoric acid and silane coupling agent application) IPS eMmax and porcelain veneered zirconia crowns.

The results of the non-thermocycled groups (Group 3 and Group 4) show the highest mean shear bond strength (SBS) of 8.1 MPa (72.7 Newtons) was for the Transbond XT/eMax crown combination, second highest shear bond strength of 6.4 MPa (57.3 N) for the Transbond XT/ porcelain veneered zirconia crown combination, third highest shear bond strength of 5.8 MPa (51.9 N) for the RelyX Unicem 2/ porcelain veneered zirconia crown combination and the lowest mean shear bond strength of 5.1 MPa (45.5 Newtons) was for the RelyX Unicem 2/eMax crown combination *(see Tables: 12 and 13. and Tables:15 and 16.).* Guimaraes *et al.'s* (2012) study on shear bond strength (SBS) of Transbond XT bonded to feldspathic porcelain discs conditioned with 37% phosphoric acid and silane application showed a mean SBS value of 7.32 MPa and concluded the shear bond strength (SBS) to be ideal for orthodontic bonding. Larmour *et al.'s* (2006) study on

shear bond strength (SBS) of Transbond^{TM} XT bonded to porcelain denture teeth conditioned with 37% phosphoric acid and a silane coupling agent showed a mean shear bond strength (SBS) value of 7.9 MPa. However, it must be borne in mind that these studies have used feldspathic porcelain discs and porcelain denture teeth respectively, which may behave differently then when bonding to porcelain crowns. The Mean shear bond strengths (SBS) of the 4 non-thermocycled adhesive/crown combinations in the present study (ie. 8.1 MPa for Transbond^{TM} XT/IPS eMax crown, 6.4 MPa for the Transbond^{TM} XT/porcelain veneered zirconia crown, 5.8 MPa for the RelyX^{TM} Unicem 2/ porcelain veneered zirconia crown and 5.1 MPa for the RelyX^{TM} Unicem 2/ IPS eMax crown combinations) are in agreement with the current literature and even though the Mean SBS of 5.8 MPa for the RelyX^{TM} Unicem 2/ porcelain veneered crown and 5.1 MPa for the RelyX^{TM} Unicem 2/ IPS eMax crown combination are lower than the ideal rupture force of 5.9 MPa (Guimaraes 2012), in this study, there is no statistically significant difference between the SBS of RelyX^{TM} Unicem 2 dual-cured, self-adhesive resin cement and Transbond^{TM} XT light-cured, 2-step adhesive resin cement (which is a commonly used orthodontic adhesive resin cement) to IPS eMax and porcelain veneered zirconia crowns, and should therefore still be clinically acceptable. Moreover, cohesive fractures may be seen on the ceramic surface, if the bond strength results between the ceramic and the composite resin are greater than 13 MPa (Thurmond *et al* 1994). In our present study, the bond strength values in all 4 groups did not exceed this value.

As this is the first shear bond strength study on IPS eMax and porcelain-veneered zirconia crowns conditioned with 35% phosphoric acid and a silane coupling agent in the literature, there are no values to compare the results with.

Shear bond strength values will be compared with results from bonding orthodontic brackets to ceramic crowns conditioned with Hydrofluoric acid (HFA) and a silane coupling agent. Jivanescu and Bratu (2014) compared RelyX^{TM} Unicem self-adhesive resin to a light cured bonding system on porcelain-fused to metal crowns which were conditioned with 10% HFA, a primer and an adhesive. No statistically significant difference was found between the RelyX^{TM} Unicem resin (SBS-5.18MPa) and the light cured bonding system. They concluded that both materials may be recommended for bonding orthodontic brackets to ceramic surfaces. In this study, the shear bond strength of the RelyX^{TM} Unicem 2 dual-cured, self adhesive resin cement/ IPS eMax crown combination was 5.1 MPa and 5.8 MPa for the RelyX^{TM} Unicem 2 dual-cured, self-adhesive resin cement/ porcelain veneered zirconia crown combination.

In Group 3 and Group 4, no statistically significant differences were found in the shear bond strengths of metal brackets bonded with the RelyX^{TM} Unicem 2 dual-cured, self-adhesive resin cement and metal brackets bonded with the Transbond^{TM} XT light-cured, 2-step bonding orthodontic adhesive cement to IPS eMax and porcelain-veneered zirconia crowns which were treated with 35% phosphoric acid and a silane coupling agent. This is in agreement with a study by Bilgic *et al* (2013) who had treated the porcelain surfaces with 9.6% HFA and a silane primer. This is also in agreement with a study by Elham *et al* (2007). However, Turk *et al* (2006) reported that lithium disilicate had a higher shear bond strength (SBS) than feldspathic porcelain restorations. Moreover, Abu Alhaija and Al-Wahadani (2007) observed significant differences between feldspathic and lithium disilicate ceramic restorations (IPS empress 2), with higher mean shear bond strength (SBS) reported in the feldspathic porcelain group. This may also be due to the structural differences between IPS empress 2 crown (earlier version of IPS eMax crown) and the IPS eMax crown. Ahluwalia *et al's* (2013) study which used a 9.6% HFA etch and silane primer found the IPS eMax crowns to have the greatest shear bond strength. The ceramo-metal and ceramo-zirconia crowns had comparable shear

bond strengths. This may be due to the differences in the processing methods and the molecular structure of the all-ceramic restorations.

5.1. Shear bond strengths (SBS) comparisons: non-thermocycled groups vs thermocycled groups *(see Tables: 6-16.)*

The third objective of this study was to compare the effects of thermocycling on the shear bond strengths of the tested groups. The results of the thermocycled groups (Group 1 and Group 2) show the Transbond^{TM} XT/non-thermocycled eMax crown combination yielded the highest mean shear bond strength of 8.1 MPa (72.7 Newtons) but dropped to a mean shear bond strength of 5.1 MPa (46.1 Newtons) (36.4% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The Transbond^{TM} XT/non-thermocycled porcelain veneerd zirconia crown combination yielded the second highest mean shear bond strength of 6.4 MPa (57.3 Newtons) and dropped to a mean shear bond strength of 5.1 MPa (45.8 Newtons) (19.3% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The RelyX^{TM} Unicem 2/non-thermocycled porcelain veneered zirconia crown combination yielded the third highest mean shear bond strength of 5.8 MPa (51.9 Newtons) but dropped significantly to a mean shear bond strength of 3.2 MPa (29.1 Newtons) (a significant 43.8% drop in shear bond strength) when the crowns were thermocycled prior to bonding *(see Table: 23.)*. Lastly, the RelyX^{TM} Unicem 2/non-thermocycled eMax crown combination yielded the fourth highest mean shear bond strength of 5.1MPa (45.5 Newtons) but dropped to a mean shear bond strength of 4.9 MPa (44.5 Newtons) (a drop in shear bond strength of only 3%) when the crowns were thermocyled prior to bonding. Relaxing the significance level (p-value) somewhat demonstrates the adverse effect of thermocycling on the shear bond strength of the adhesive/crown combinations *(see Tables: 23-26.).*

In this study, a statistically significant difference was found between the shear bond strengths of the non-thermocycled and thermocycled groups. As this is the first study on the influence of thermocycling prior to bonding on shear bond strength in the literature, there are no values to compare the results with. However, the adverse influence of thermocycling can be seen on the measured shear bond strength values.

It should be emphasized that the difference between in vitro versus in vivo bond strengths needs to be considered carefully, especially when bonding brackets to other restorative materials. Andreasen and Stieg (1988) indicated that the shear and tensile bond strengths of in vivo incisor and premolar enamel were significantly less than those of in vitro incisor and premolar enamel. They suggested that part of the in vivo increase in the rate of deterioration may be because of the mechanical and masticatory stresses placed on the bonds in the oral environment. They listed other factors, which may be of importance, including the moisture within the living tooth, flexing of the enamel during mastication, moisture contamination during bonding, as well as the thermal fluctuation in the oral cavity and the constant bathing of saliva. However, as this is a first in-vitro study on the influence of thermocycling prior to bonding of orthodontic brackets, there is no explanation in the literature as to why bonding of orthodontic brackets using adhesive resin cements to porcelain, which is an inert material, would be adversely affected by thermocycling prior to bonding. This may be an interesting topic for future research in order to gain a better understanding of the bonding orthodontic brackets to porcelain crowns.

Andreasen and Stieg (1988) calculated that there was a decrease of approximately 17% to 22% in tensile strengths and 48% to 52% in shear strengths in vivo when compared with the in vitro bond strengths. They suggested that if this percent of in vivo decline is evident when bonding to porcelain surfaces, stronger bond strengths would be required for efficient bonding of orthodontic brackets in the actual patient. In this study, even though the shear bond strengths of the adhesive/crown combinations were reduced statistically significantly when the porcelain crowns were thermocycled

prior to bonding, according to the literature (Andreason and Stieg 1988), may be something that is expected. In this study, a decrease of 36.4% in shear bond strengths for the Transbond^{TM} XT/ thermocycled eMax crown combination, a decrease of 19.3% in shear bond strengths for the Transbond^{TM} XT/ thermocycled porcelain veneered zirconia crown combination, a decrease of 43.8% in shear bond strengths for the RelyX^{TM} Unicem 2/ thermocycled porcelain veneered zirconia crown combination, and a decrease of 3% in shear bond strengths for the RelyX^{TM} Unicem 2/ thermocycled eMax crown combination, are significant, but according to the literature (Andreason and Stieg 1988), may still be clinically acceptable.

The number of thermal cycle is another point of dispute between different researchers. It has been 100, 150, 200 and 500 times in previous researches (Smith *et al* 1988, Newman 1994). We applied the biggest number in our study.

5.2. Adhesive Remnant Index comparisons (non-thermocycled crowns- group 3 and group 4)

The second objective of this study was to compare the resultant failure pattern of the tested groups.

Description of each category of the adhesive remnant index *(see Table: 1.).*

RelyX^{TM} Unicem 2 adhesive/non-thermocycled eMax crown combination *(see Table: 27.)*

- 5 specimens debonded at the adhesive/bracket interface (ARI 3)
- 2 specimens had more than 50% of the adhesive on the ceramic surface (ARI 2)
- 2 specimens had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 1 specimen had all the adhesive removed with the bracket (ARI 0)

- mean score of 2.1

RelyX™ Unicem 2 adhesive/non-thermocycled porcelain veneered zirconia crown combination *(see Table: 27.)*

- All 10 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 0

Transbond™ XT adhesive/non-thermocycled eMax crown combination *(see Table: 27.)*

- 4 specimens debonded at the adhesive/bracket interface (ARI 3)
- 1 specimen had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 5 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 1.3

Transbond™ XT adhesive/non-thermocycled porcelain veneered zirconia crown combination *(see Table:27.)*

- 5 specimens debonded at the adhesive/bracket interface (ARI 3)
- 1 specimen had more than 50% of the adhesive on the ceramic surface (ARI 2)
- 1 specimen had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 3 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 1.8

The ARI results for the non-thermocycled crown/adhesive combinations display a mean score of 2.1 for the RelyX™ Unicem 2/non-thermocycled eMax crown combination, a mean of 0 for the RelyX™ Unicem 2/non-thermocycled porcelain veneered zirconia crown combination, a mean of 1.3 for the Transbod™ XT/ non-thermocycled eMax crown combination and a mean of 1.8 for the Transbond™ XT/non-thermocycled porcelain veneered zirconia crown combination *(see Table: 27. and Figure: 16.).* Study of the mean ARI values for the non-thermocycled crown/adhesive combinations shows that brackets bonded with RelyX™ Unicem 2/non-thermocycled porcelain veneered zirconia crowns failed entirely at the ceramic/adhesive interface and for all the other non-thermocycled ceramic/adhesive combinations most of the failures of the bond (70%) occurred at the bracket/adhesive interface and cohesive fractures within the composite resin. No cohesive fractures of the porcelain crowns were noted. The present findings indicate that there was no significant difference in the debonding patterns of the four non-thermocycled ceramic/adhesive combinations. This finding is different to the study by Bishara *et al* (2000) who tested RelyX™ Unicem with Transbond™ XT and their findings indicated that the brackets bonded with RelyX™ Unicem failed at the enamel/adhesive interface, whereas brackets bonded using Transbond™ XT typically, failed at the bracket/adhesive interface.

5.3. Adhesive Remnant Index (ARI): comparison of non-thermocycyled and thermocycled groups

In this present study a similar trend to the shear bond strength was noted when ARI scores were examined. The non-thermocycled all ceramic crown/adhesive combinations showed mean ARI values of between 1.3 and 2.1 indicating cohesive fractures within the composite resin and efficient bonding of the adhesive material to the porcelain. However, the thermocycled all ceramic crown/adhesive treatment combinations showed mean ARI values of between 0 and 0.8 indicating a

bond failure between adhesive and porcelain and highlighting the adverse influence of thermocycling on bond strength of the adhesive resin cement *(see Table: 27. and Figure: 16.).* Bracket failure at each of the two interfaces has its own advantages and disadvantages. As an example, bracket failure at the bracket/adhesive interface is advantageous because it leaves the porcelain surface intact; however, considerable chair time is needed to remove the residual adhesive with the added possibility of damaging the porcelain during the cleaning process (Bishara *et al* 2000). On the other hand, when brackets fail at the porcelain/adhesive interface, less residual adhesive remains, therefore making the cleaning of the porcelain surface so much easier (Bishara *et al* 1998).

5.4. Porcelain Fracture Index (PFI)

The fourth objective of this study was to compare the surface integrity of the IPS eMax and porcelain veneered zirconia crowns after debonding for each of the groups tested using the Porcelain Fracture Index (PFI). Description of each category of the porcealain fracture index (*see Table 2.*).

All specimens were scored 0 but one specimen from the Transbond^{TM} XT adhesive/thermocycled porcelain veneered zirconia crown combination was scored 3 due to delamination of the veneered porcelain from the underlying zirconia core. This may possibly be due to the poor bonding of the veneered porcelain to the underlying zirconia core. In the present study, optical microsope examination revealed no damage to the porcelain surfaces of 98.75% of the all ceramic crowns after debonding (*see Table: 28.*). A previous study (Thurmond *et al* 1994) showed that if the bond strength between the porcelain and the adhesive is greater than 13 MPa, the porcelain is fractured. In this study all 4 groups obtained shear bond strength values less than 13 MPa.

5.5. Ethical Consideration

This is a full laboratory study and no human tissue was used.

5.6. Conflict of interest

No conflict of interest was declared.

5.7. Limitations

This study has some limitations that may preclude the extrapolation of the results: a small sample size was used; the use of one type of orthodontic bracket and it is an in vitro study, which tested only resistance to shear forces, under constant load. Thermocycling studies also have limits. Thermocycling in water poorly represents the dynamic environment of the oral cavity (Mair and Padipatvuthikul 2009). There is also important variability in the methods used to evaluate bond strength within the orthodontic literature, partially due to the lack of standardization protocols. As a result, it is difficult to draw any meaningful conclusion when comparing studies.

5.8. Future Research

Future research avenues can be orientated towards alternative debonding methods. Debonding should be explored using manual debonding, electrothermal debonding devices and lasers (Tocchio *et a*l 1993, Azzeh and Feldon 2003, Bishara and Trulove 1990). Studies comparing machine debonding and manual debonding can be interesting.

Chapter 6

Conclusion

Conclusion

Within the limitations of this study, it can be concluded that:

1.There was no significant difference in the shear bond strengths of metal orthodontic brackets bonded with RelyX^{TM} Unicem self-adhesive resin cement and metal orthodontic brackets bonded with Transbond^{TM} XT adhesive cement to non-thermocycled IPS eMax and porcelain-veneered zirconia crowns which were conditioned with 35 % phosphoric acid and a silane coupling agent.

2. Conditioning the porcelain surface with 35% phosphoric acid and a silane coupling agent would be safer to use than Hydrofluoric acid and should make it less risky for clinicians to clean the adhesive on the porcelain surface after debonding.

3. The negative influence of thermocycling prior to bonding can be seen on shear bond strength values.

4. Most of the bond failures for the non-thermocycled crown/adhesive combinations occurred at the bracket/adhesive interface and cohesive fractures within the composite resin and most of the bond failures for the thermocycled crown/adhesive combinations occurred at the adhesive/porcelain interface. No cohesive fractures of the porcelain crowns were noted.

References

References

Abu Alhaija ES, Abu AlReesh IA, AlWahadni AM. 2010. Factors affecting the shear bond strength of metal and ceramic brackets bonded to different ceramic surfaces. Eur J Orthod, 32: 274-280.

Abu Alhaija ES, Al-Wahadani AMS. 2007. Shear bond strength of orthodontic brackets bonded to different ceramic surfaces. Eur J Orthod, 29: 386-389.

Aidam M, Hayakawa T, Mizukawa K. 1995. Adhesion of composite to porcelain with various surface conditions. J Prosthetic Dent, 73: 464-470.

Ajlouni R, Bishara SE, Oonsombat C, Soliman M, Laffoon J. 2005. The effects of porcelain surface conditioning on bonding orthodontic brackets. Angle Orthod, 75: 858-864.

Alhuwalia KS, Gupta A, Gaurav I, Jain N, *et al*. 2013. Shear bond strength of self-ligating orthodontic brackets on different types of porcelain crowns. J Orofacial Sc, 5(2): 101-104.

Alexander RG. 1986. The Alexander Discipline: Contemporary concepts and philosophies. Ormco Corp. 119-133.

Andreason GF and Stieg MA. 1988. Bonding and debonding brackets to porcelain and gold. Am J Orthod Dentofacial Orthop. 93: 341-345.

Anusavice KJ. 2003. Phillips' Science of Dental Materials. 11th edition. Elsevier Health Sciences.

Artun J and Bergland S. 1984. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod, 85: 333-340.

Azzeh E, Feldon PJ. 2003. Laser debonding of ceramic brackets: a comprehensive review. Am J Orthod Dentofacial Orthop, 123: 79-83.

Banks P, MacFarlane TV. 2007. Bonded versus banded first molar attachments: a randomised controlled clinical trial. J Orthod, 34: 128-136.

Barry GR. 1995. A clinical investigation of the effects of omission of pumice prophylaxis on band and bond failure. Br J Orthod, 22: 245-248.

Bilgic F, Alkis H, Gungor AY, *et al*. 2013. Shear bond strength of ceramic brackets bonde to three different porcelain surfaces. Eur J Orthod, 1: 17-20.

Bishara SE, Trulove TS. 1990. Comparisons of different debonding techniques for ceramic brackets: an in vitro study. Part II. Findings and clinical implications. Am J Orthod Dentofacial Orthop, 98: 263-273.

Bishara SE, Fehr DE, Jakobsen JR. 1993. A comparative study of different ceramic brackets, enamel conditioners and adhesives. Am J Orthod Dentofacial Orthop, 104: 170-190.

Bishara SE, Fonesca JM, Fehr DE, Boyer DB. 1994. Debonding forces applied to ceramic brackets simulating clinical conditions. Angle Othodontist, 64: 277-282.

Bishara SE, Fonesca JM, Boyer DB. 1995. The use of debonding pliers in the removal of ceramic brackets. Am J Orthod Dentofacial Orthop, 108: 242-248.

Bishara SE, Olsen M, Von Wald I. 1997. Comparisons of shear bond strength of precoated and uncoated brackets. Am J Orthod Dentofacial Orthop, 112: 617-621.

Bishara SE, Gordon VV, VonWald L, Olson ME. 1998. Effect of an acidic primer on shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop, 114: 243-247.

Bishara SE, Olsen ME, Von Wald L, Jakobsen JR. 1999. Comparison of the debonding characteristics of two innovative ceramic bracket designs. Am J Orthod Dentofacial Othop, 116: 86-92.

Bishara SE, VonWald L, Laffoon JF, Jacobsen JR. 2000. Effect of altering the type of enamel conditioner on the shear bond strength of a resin-reinforced glass ionomer adhesive. Am J Orthod Dentofacial Orthop, 118: 288-294.

Blatz MB, Sadan A and Kern M. 2003. Resin ceramic bonding: A review of the literature. J Prosthet Dent, 89: 268-274.

Bourke BM, Rock WP. 1999. Factors affecting the shear bond strength of Orthodontic brackets to porcelain. Br J Orthod, 26: 285-290.

Britton JC, McInnes P, Weinberg R, Ledoux WR, Relief DH. 1998. Shear bond strength of ceramic orthodontic brackets to enamel. Am J Ortho Dentofacial Orthop, 114: 243-247.

Clarke SA, Gordon PH, Mc Cabe JF. 2003. An ex vivo investigation to compare orthodontic bonding using a 4-META based adhesive or a composite adhesive to acid etched and sandblasted enamel. J Orthod, 30: 53-58.

Conrad HJ, Scong WJ and Pesun IJ. 2007. Current ceramic materials and systems with clinical recommendations: A systemic review. J Prosthet Dent, 98: 389-404.

Daub J, Berzins DW, Linn BJ and Bradley TG. 2006. Bond strength of Direct and Indirect bonded brackets after thermocycling. Angle Orthodontist,76: 295-300.

De Munck J, *et al*. 2004. Bonding of an auto-adhesive luting material to enamel and dentine. Dent Mater, 20: 963-971.

Elham S. Abu Alhaija J and AL-Wahadni AM. 2007.Shear bond strength of orthodontic brackets to different ceramic surfaces. Eur J Orthod, 29: 386-389.

Eliades T. 2006. Orthodontic materials research and applications: Part 1. Current status and projected future developments in bonding and adhesives. Am J Orthod Dentofacial Orthop, 130: 445-451.

Eliades T and Brantley WA. 2000. The inappropriateness of conventional orthodontic bond strength assessment protocols. Er J Orthod, 22: 13-23.

Faltermeier A, *et al*. 2012. The influence of surface conditioning of ceramic restorations before metal bracket bonding. Mat Science and Applications, 3: 1-5.

Farquhar RB. 1986. Direct bonding comparing a polyacrylic acid and a phosphoric acid technique. Am J Orthod Dentofac Orthop, 90: 187-194.

Fernandez L, Canut JA. 1999. In vitro comparison of the retention capacity of aesthetic brackets. Eur J Orthod, 21: 71-77.

Fox NA, McCabe JB and Buckley JG. 1994. A critique of bond strength testing in orthodontics. Br. J. Orthod, 21: 33-43.

Fradaeni M. November 2012. Presentation: Aesthetic challenges on natural dentition. SADA Dental Congress, Cape Town, South Africa.

Garcia-Godoy F, Perez R, Hubbard GW. 1991. Effect of Prophylaxis Pastes on Shear Bond Strength. J Clin Ortho, 25: 371-373.

Gillis J and Redlich M. 1998. The effect of different porcelain conditioning techniques on shear bond strength of stainless steel brackets. Am J Orthod Dentofac Orthop, 114: 387-392.

Guimaraes MB, Lenz HF, Bueno RS, Blaya MBG, Hirakata LM. 2012. Orthodontic bonding to porcelain surfaces: In vitro shear bond strength. Rev Odonto Cienc, 27: 47-51.

Hayakawa T, Horie K, Aida M, Kanaya H, Koboyashi T and Murata Y. 1992. The influence of surface conditions and silane agents on the bond of resin to dental porcelain. Den Mat, 8: 238-240.

Hecht R, *et al*. Longevity data of the first self-adhesive universal resin cement. *http://iadr.confex .com.*

Heravi F, Moazzami SM, Dehghani M. 2010. Effects of different surface preparations on shear bond strength of orthodontic brackets to porcelain. J Calif Dent Assoc, 38: 794-799.

Herion DT, Ferracane JL, Covel DA. 2010. Porcelain surface interactions and refinishing after use of two orthodontic bonding materials. Angle Orthod, 80: 167-174.

Hirce JD. 1980. The effect of topical fluoride after acid etching on the bond strength of directly bonded orthodontic brackets. Am J Orthod, 78: 444-452.

Hudson APG. 2007. Thesis: Comparative bond strengths of stainless steel orthodontic brackets bonded with different bonding agents. University of the Western Cape. 7-20.

Jivanescu A and Bratu CD. 2014. Tensile bond strength evaluation of two adhesive cements used for bonding orthodontic metal brackets to porcelain-fused-to-metal crowns. Mat Plastice, 51: 465-467.

Kalange JT. 2007. Indirect Bonding: A Comprehensive Review of the Literature. Semin Orthod, 13: 7.

Kao EC, Bolz KC and Johnson WM. 1988. Direct bonding of orthodontic brackets to porcelain veneer laminates. Am J Orthod Dentofacial Orthop, 94: 458-468.

Karan S, Buyukyilmaz T, Toroglu MS. 2007. Orthodontic bonding to several ceramic surfaces: are there acceptable alternatives to conventional methods? Am J Orthod Dentofacial Orthop, 132: 144-147.

Kauppi MR, Combe EC. 2003. Polymerization of orthodontic adhesives using modern high-intensity visible curing lights. Am J Orthod Dentofacial Orthop, 124: 316-322.

Kelly JR, Benetti P. 2011. Ceramic materials in dentistry: historical evolution and current practice. Aust Dent J, 56 Suppl 1: 84-96.

Klocke A, Shi J, Kahl-Nieke B, Bismayer U. 2003. Bond strength with custom base indirect bonding techniques. Angle Orthod, 73: 176-180.

Klocke A, Kahl-Nieke B. 2005. In vitro bond strength values: Meaningful or confusing? World J Orthod, Supplement 6: 114.

Kocadereli I, Canay Sand Akea K. 2001. Tensile bond strength of ceramic orthodontic orthodontic brackets bonded to porcelain surfaces. Am J Orthod Dentofacial Orthop, 119: 617-620.

Kumbuloglu O, *et al*. 2004. A study of the physical and chemical properties of four resin composite luting cements. Int J Prostho, 17: 357-363.

Kumbuloglu O, *et al*. 2005. Shear bond strengths of composite resin cements to lithium disilicate ceramics. J Oral Rehabil, 32: 128-133.

Lamour CJ, Bateman G and Stirrups DR. 2006. An investigation into the bonding of orthodontic attachments to porcelain. Eur J Orthod, 28(1): 74-77.

MacColl GA, Rossouw PE, Titley KC, Yamin C. 1998. The relationship between bond strength and orthodontic bracket base surface area with conventional and microetched foil-mesh bases. Am J Orthod Dentofac Orthop, 113: 276-281.

Mair L, Padipatvuthikul P. 2009. Variables related to materials and preparing for bond strength testing irrespective of the test protocol. Dent Mater, 26: 17-23.

Matasa CG. 2003. In search of a better bond: State of the Art. The Orthod Material Insider, 15: 1.

Matasa CG. 2005. Resin based composites: Today. The Orthod Materials Insider, 17: 1-3.

Mattos AM, Capelli JM. 2006. Porcelain surface evaluation after debonding of orthodontic brackets. Rev Dent Press Orthodon Orthopedi Facial, 11: 151-158.

Nebbe B, Stein E. 1996. Orthodontic brackets bonded to glazed and deglazed porcelain surfaces. Am J Orthod Dentofacial Orthop, 09: 431-436.

Nelson E. 1989. Effects of APF etching time on resin bonded porcelain. J Dent Research, 68: 271 *(abstract).*

Newman GV. 1965. Epoxy adhesives for orthodontic attachments: progress reports. Am J Orthod, 51: 901-912.

Newman GV. 1994. Epoxy adhesives for orthodontic attachments: Progress report. Am J Orthod Dentofacial Orthop, 106: 358-364.

O'Brien KD, Watts DC, Read MJF. 1991. Light-cured direct bonding- is it necessary to use a primer? Eur J Orthod, 13: 22-26.

Okemwa JR, Kudlick EM, Eichmuller FC. 2002. Shear bond strength of 2 materials: Transbond and an experimental amorphous calcium phosphate. Technical programme abstract IADR, San Diego.

Olsen ME, Bishara SE, Boyer DB, Jakobsen JR. 1996. Effect of varying etching times on the bond strength of ceramic brackets. Am J Orthod Dentofacial Orthop, 109: 403-409.

Olsen ME, Bishara SE, Jacobsen JR. 1997. Evaluation of Scotchbond multipurpose and maleic acid a alternative methods of bonding orthodontic brackets. Am J Orthod Dentofacial Orthop, 111: 496-501.

Perry AC. 1980. Rebonding orthodontic brackets. J Clin Orthod, 14:850-854.

Pickett KL, Sadowsky PL, Jacobsen A, Lacefield W. 2001. Orthodontic in vivo bond strength: comparison with in vitro results. Angle Orthod, 7: 41-44.

Piwowarczyk A, Lauer HC. 2003. Mechanical properties of luting cements after water storage. Oper Dent, 28: 535-542.

Piwowarczyk A, Lauer HC and Sorensen JA. 2004. In vitro shear bond strength of cementing agents to fixed prosthodontic restorative materials. J Prostho Dent, 92: 265-273.

Porseld V, *et al*. Effect of three years' aging on mechanical properties of RelyX Unicem. Available at: *http://iadr.confex.com.*

Powers JM, Kim HB, Turner DS. 1997. Orthodontic adhesives and bond strength testing. Semin Orthod, 3: 147-156.

Proffitt WR, Fields HW. Contemporary Orthodontics. C.V. Mosby Company. 3rd Ed. 287-310.

Purmal K, Alam MK and Sukumaran P. 2013. Shear bond strength of orthodontic buccal tubes to porcelain. Dent Res J, 10: 81-86.

Raigrodski AJ. 2004. Contemporary materials and technologies for all- ceramic fixed partial dentures: a review of the literature. J Prosthet Dent, 92: 557-562.

Raji SH, Birang R, Majdzade F, Ghorbanipour R. 2012. Evaluation of shear bond strength of orthodontic brackets bonded with Er-YAG laser etching. Dent Res J, 9:288-293.

Read MJ, O'Brien KD. 1990. A Clinical trial of an indirect bonding technique with a visible light-cured adhesive. Am J Orthod Dentofacial Orthop, 98: 259-262.

Reynolds IR. 1975. A review of direct orthodontic bonding. Br J Orthod, 2: 171-178.

Rosensteil S, Land MF, Fujimoto J. 2006. Contemporary Fixed Prosthodontics. 4th Edition.St-Louis, Missouri: Mosby Elsevier.

Rossouw PE. 2010. A Historical Overview of the Development of the Acid-Etch Bonding System in Orthodontics. Semin Orthod, 16: 2-23.

Rueggeberg FA. 1991. Substrate for adhesion testing to tooth structure- review of the literature. Dent Mat, 7: 2-10.

Samruajbenjakul B and Kukiattrakoon B. 2009. Shear Bobd Strength of Ceramic Brackets with Different Base designs to Feldspathic Porcelain. Angle Orthod, 79: 571-576.

Schmage P, Nergiz I, Herrman W, Ozcan M. 2003. Influence of various surface-conditioning methods on the bond strength of metal brackets to ceramic surfaces. Am J Orthod Dentofacial Orthop, 123: 540-546.

Sfondrini MF, Cacciafesta V, Scibante A, De Angelis M and Klersy C. 2004. Effect of blood contamination on shear bond strength of brackets bonded with conventional and self-etching primers. Am J Orthod Dentofac Orthop, 125: 357-360.

Sharma-Sayal S, Rossouw PE, Kulkarni GV, Titley KC. 2003. The influence of orthodontic bracket base design on shear bond strength. Am J Orthod Dentofac Orthop, 124: 74-84.

Smith GA, McInnes-Ledoux P, Ledoux WR, Weinberg R. 1988. Orthodontic bonding to porcelain-bond strength and refinishing. Am J Orthod Dentofacial Orthop, 119: 245-252.

Sorel O, Alam R, Chagneau F, Cathelnieau G. 2002. Comparison of bond strength between simple foil mesh and laser-structured base retention brackets. Am J Othod Dentofac Orthop, 122: 260-266.

Swanson T, Dunn WJ, Childers DE, Taloumis LJ. 2004. Shear bond strength of orthodontic brackets bonded with light emitting diode curing units at various polymerization times. Am J Orthod Dentofac Orthop, 12: 337-341.

Thomas RL, De Rijk WG, Evans CA. 1999. Tensile and Shear stresses in orthodontic attachment adhesive layer with 3d finite element analysis. Am J Orthod Dentofac Orthop, 116; 530-532.

Thurmond JW, Barkmeier WW and Wilwerding TM. 1994. Effect of porcelain surface treatments on bond strengths of composite resin bonded to porcelain. J Prosth Dent, 72: 355-359.

Tocchio RM, Williams PT, Mayer FJ and Standing KG. 1993. Laser debonding of ceramic orthodontic brackets. Am J Orthod Detofacial Orthop, 103: 155-162.

Toodehzaeim MH, Kazemi A, Aghili HA, Barzegar K, Fallahtafti T. 2012. Comparison of shear bond strength of orthodontic brackets bonded with halogen and plasma arc light curing. Dent Res J, 9: 321-327.

Turk T, Sarac D, Sarac Y, Elekdag-Turk S. 2006. Effects of surface conditioning on bond strength of metal brackets to all-ceramic surfaces. Eur J Orthod, 28: 450-456.

Van Noort R, Noroozi S, Howard IC, Cardew G. 1989. A critique of bond strength measurements. J Dent, 17: 61-67.

Whitlock BO, Erick JD, Ackerman RJ, Glaros AG and Chappell RP. 1994. Shear strength of ceramic brackets bonded to porcelain. Am J Orthod Dentofacial Orthop, 106: 358-364.

Winchester L and Orth M. 1991. Direct orthodontic bonding to porcelain. Br J Orthod, 18: 299-308.

Wolf DM, Powers JM, and O'Keefe KL. 1993. Bond strength of composite to etched and sandblasted porcelain. Am J Dent, 6: 155-158.

Wood DP, Jordan RE, Way DC, Galil KA. 1986. Bonding to porcelain and gold. Am J Orthod, 89: 194-205.

Zachrisson YO, Zachrisson BU and Buyukyilmas S. 1996. Surface preparation of orthodontic bonding to porcelain. Am J Orthod Dentofacial Orthop, 109: 420-430.