The effect of thermal-cycling on the bond strength of a two-step and single-step dentin bonding agent

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SUMMARY

Dentin bonding agents display marked improvement in their mechanical properties as new systems are introduced commercially. The old dentin bonding systems had three application steps; acid –etching, application of primer, and finally the application of the adhesive system. With the newer generations of dentin bonding systems the numbers of application steps have decreased. This improvement resulted in the production of the two-step and single step dentin bonding agents. There are several factors that can influence the bond strength of these adhesive systems. The thermal changes that occur inside the oral cavity as a result of food consumption can create stresses that affect the bond strength of these two-step and single-step dentin adhesive systems. The aim of this study is to investigate if there is any difference in the bond strength between the two-step and the single-step dentin adhesive systems and then to assess the effect of repeated thermal changes on this bond strength following repeated thermal changes. **Aim and objectives:** The aim and objective of this study was to determine the bond strength of a two-step and a single-step dentin bonding agent and to determine the effect of repeated thermal-cycling from 5^0C and 55^0C on the bond strength values of these dentin bonding agents. **Materials and methods:** 100 extracted human molar teeth were used in this study. The roots of the teeth were sectioned at the crown-root junction. The teeth were embedded in cold-cure acrylic resin so that the buccal surface of the teeth projected slightly above the acrylic resin. The buccal surfaces of the teeth were cut off exposing a flat dentin surface. The flat surfaces were polished with 1200 grit silicon finishing paper. The teeth will be randomly divided into two groups (n=50). In which Prime and Bond NT (two-step dentin bonding agent) was used to bond the composite mold to the flat dentin surface in the first group and Xeno V (single-step dentin bonding agent) was used to bond the composite mold to the flat dentin surface in the second group. In each group the teeth were subdivided randomly into another two groups in which (n1=25), in the first subgroup the samples were stored in water at 37^0C for 24 hours before measuring the shear bond strength value and the group was labeled as Prime and Bond NT or Xeno V without thermal-cycling. In the second subgroup the samples were thermal-cycled between 5^0C and 55^0C for 500 cycles before the shear bond strength was measured. **Results:** the shear bond strength test revealed that the bond strength values between Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent) were not statistically different ($p>0.05$). The shear bond strength values of Prime and Bond NT (two-step dentin bonding agent) before and after thermal-cycling were not statistically different ($p>0.05$). The shear bond strength values of Xeno V (single-step dentin bonding agent) before and after thermal-cycling were not statistically different ($p>0.05$). **Conclusion:** Within the limitation of this study there is no difference in the shear bond strength values to dentin between the two-step dentin bonding agent (Prime and Bond NT) and the single-step dentin bonding agent (Xeno V).Thermo-cycling between 5^0C and 55^0C had no effect on the bond strength values of these dentin bonding agents.

DECLARATION

I hereby declare that *the effect of thermal-cycling on the bond strength of a two-step and single-step dentin bonding agent* is my own work, that it has not been submitted before for any degree or examination in any university, and that all the sources I have used or quoted have been indicated and acknowledged by complete references.

Amir Isam Omer

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Signed: ....

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DEDICATION

To my Mother who always supported me day by day
And provided me with all I needed to
reach this level of education.

To my father for his continuous encouragement
And support

To my lovely wife
Who stood by me and support me
Every day and night to finish this project
(I love you).

Last but not least, My grandmother, brother, and sister
For all your love and support

TABLE OF CONTENTS

LISTS OF FIGURES

LISTS OF TABLES

Chapter 1
Introduction

Dentin bonding agents display marked improvement in their mechanical properties as new systems are introduced commercially. The older generation dentin bonding systems had three application steps; acid –etching, application of primer, and finally the application of the adhesive system. With the newer generations of dentin bonding systems the number of application steps has decreased. This improvement has resulted in the introduction of the two-step and the single-step dentin bonding agents. There are several factors that have contributed to the bond strength of these adhesive systems. The thermal changes that occur inside the oral cavity as a result of food consumption also create stresses that can affect the bond strength of all these dentin adhesive systems. The objective of this study was to investigate if there was any difference in the bond strength of the two-step and the single-step dentin adhesive systems following repeated thermal changes.

1.1 Definition of terms:

For the purpose of this study, the following terms are defined as follows:

- **Dentin bonding agent:** resinous material that is applied to the tooth in a thin layer in order to bond a restorative material to the tooth surface micromechanically through the formation of a hybrid layer.

- **Hybrid layer:** an intermediate layer of resin, collagen, and dentin produced by the etching of dentin followed by the resin infiltration into the conditioned dentin.

- **Composite resin:** a highly cross-linked polymeric material reinforced by a dispersion of amorphous particles such as silica, glass, crystalline, or organic resin filler particles and/or short fibers bonded to the matrix by a coupling agent

- **Bond strength test:** tests used to evaluate the force required to break the adhesive bond between the tooth structure and the restorative material.

- **Shear stress:** is the ratio of force to the original cross-sectional area parallel to the direction of the force applied to the test specimen.

- **Shear strength:** is the maximum shear stress at the point of fracture of the bonded surface and/or the specimen.

- **Thermal-cycling:** is an *in vitro* process in which the restorative material and the tooth structure are subjected to repeated thermal changes ranging between 5^0–55^0C for the purpose of this study

- **Mann-Whitney U test:** a non-parametric significance method of testing for equality of population medians among groups.

Chapter 2
Literature review

2.1. Introduction:

The ideal restorative material should have the same physical properties as tooth structure. It should also adhere chemically to the tooth surface, require minimal instrumentation, and be aesthetically acceptable (Davis *et al*, 1992). In the past restorative materials were retained mechanically by the creation of a geometrical shape inside the tooth structure. This procedure has the disadvantage of removal of unnecessarily healthy tooth structure with a subsequent increase in the level of trauma and a resultant weakening in the remaining tooth structure (DiRenzo *et al*, 1995).

In the 1950s Dr. Oskar Hagger developed a resinous material that adhered to tooth structure. The introduction of the acid-etching technique by Michael Buonocore in 1950s improved the resin-enamel bond (Tay *et al*, 2004). These improvements in the adhesive systems resulted in the development of a micromechanical retentive system for the bonding resin to the tooth structure (Bouillaguet *et al*, 2001).

The adhesive systems continued to develop rapidly in the last ten years. This rapid development was largely due to two main factors; the first being the increasing demand for tooth-colored restorative materials by patients with a subsequent enhancement in the adhesive technology used with this type of restorative materials. The other reason is the introduction of minimally invasive operative procedures to the profession which were based on only replacing the diseased tooth tissue by direct bonding of the restorative material to the remaining tooth sound structure (Van Meerbeek *et al*, 2003).

As a result of this improvement in the adhesive systems, the clinical application techniques also showed an improvement represented by a decrease in the number of clinical application steps from the original three-steps to two-steps, and more recently even a one-step application adhesive system for dentin bonding.

The oral cavity is constantly subjected to thermal changes that may affect the strength of these dentin bonding agents (Asaka *et al*, 2007). These temperature changes, that can cause hydrolysis of the bonded interface component, could also result in repeated contraction and expansion of the dentin bonding agents. This dimensional change can result in the creation of stresses on the bonded interface (De Munk *et al*, 2005) with a significant reduction in the bond strength of a variety of dental materials (Soh and Selwynb, 1992).

Bond strength tests have been used to evaluate the strength of the adhesive joint. The bond strength can either be tested under tensile or shear load and sometimes in combination with cyclic fatigue or thermal stress (Eliades, 1994).

Asaka *et al (*2007) investigated the influence of thermal cycling on the bond strength of a single-step, self-etch dentin adhesive system, while Miyazaki *et al* (1998) investigated the influence of thermal cycling on the bond strength of a two-step dentin adhesive system using a self-etching primer system and a self-priming adhesive system. However there are limited studies that have compared the effect of thermal changes on the bond strength of the two-step and the-single step dentin bonding systems under the same conditions.

2.2. Dentin bonding agents:

2.2.1. Basic principle of adhesion:

Adhesion is the process by which two substrates are joined together through formation of an adhesive joint between them. In dentistry these substrates are usually the adhesive and the dental substrate which can be enamel, dentin, or cementum. Dental adhesives have been defined as resin monomer solutions that join a restorative material to a dental substrate following polymerization of the restorative material (Perdigão, 2007).

The adhesive bond can be mechanical, physical, chemical, or a combination of these bonds. Mechanical adhesion is the simplest method of adhesion and it results from the presence of micro irregularities on the surface of the adherend and the ability of the adhesive to penetrate into these micro-irregularities or undercuts such as in the case of pits and fissures or in the presence of etched enamel. Physical adhesion is when two surfaces are brought into intimate contact and there are secondary forces of attraction arising through a dipole-dipole interaction between the polar molecules. This is a weak, rapid and reversible type of bond and can easily be overcome by thermal energy and as such it is not suitable as the only bond for permanent bonding. The third type of bond is the chemical bond in which the molecules of the two substrates dissociate after adsorption onto the surface and the constituents of each material bond separately to the other by covalent or ionic forces, resulting in the formation of a strong adhesive bond (Van Noort, 2002 (a)).

2.2.2. History of adhesion:

The adhesive system developed by Oskar Hagger in the early 1950s for bonding his resinous material to the hard tooth structures was based on the chemical curing of glycerophosphoric acid dimethacrylate with sulphinic acid. The system was produced as a commercial product called Sevriton (Tay *et al*, 2004).

Buonocore's experiments in 1955 on enamel surfaces are still today regarded as the great landmarks in adhesive dentistry. He used 85% phosphoric acid for 30 seconds to achieve acid decalcification of the enamel surface. His experiments showed that acid etching increased the surface area of the enamel exposing the organic framework of the enamel and thereby increasing the potential for adhesion (Buonocore, 1955).

The adhesive systems have continued to improve since then showing an improvement in their bond strength and a decrease in the number of clinical steps involved in the adhesion process (Burrow and Tyas, 2003).

2.2.3. Mechanism of adhesion:

The basic mechanism of adhesion is regarded as an exchange process of the inorganic tooth material by resin monomer in which the resin monomer becomes micro-mechanically interlocked upon setting in the created micro-porosities within the tooth structure (Van Meerbeek *et al*, 2001). This type of adhesion is micro-mechanical in nature. Recently chemical adhesion has been possible through an interaction of specific monomers and the calcium in the hydroxyapatite of tooth structure (Yoshida *et al*, 2004).

2.2.3.1. Adhesion to enamel:

Enamel is the strongest and hardest human tissue (He and Swain, 2008). It consists of 85% hydroxyapatite crystals, 12% water, and 3% organic matrix by volume. Microscopically the enamel consists of enamel rods 5µm in diameter that extend from the dentin-enamel junction to approximately 6-12µm below the tooth surface. Each rod consists of bundles of hydroxyapatite crystals, and each crystal is 50nm in diameter and 100µm long. The hydroxyapatite crystal is covered by an organic layer (Bechtle *et al*, 2010). There are hydroxyapatite crystals between the rods that are arranged parallel to each other to form the interred region, and they have a particular inclination to the adjacent enamel rod axis (He and Swain, 2008). The water and the organic matrix which is mainly protein accumulate between the rod and the interred region, giving rise to the rod sheath. The accumulation in this area is assumed to be due to the lower hydroxyapatite density in this area (Bechtle *et al*, 2010).

The first step in adhesion is the creation of a rough or porous surface to enhance the bonding capacity. This surface is created by the application of a 30 to 40% phosphoric acid to both enamel and dentin (Van Meerbeek *et al*, 2005). Considering adhesion to enamel, the acid etching technique causes demineralization of the inorganic part exposing the organic framework of the enamel (McLean, 2000). This procedure increases the surface roughness and raises the surface energy of the enamel surface thus facilitating adhesion (Van Noort, 2002 (b)).

2.2.3.2. Adhesion to dentin:

Compared to enamel, dentin has a complex hydrated structure (Marshall *et al*, 1997). In 1996 Pashley described the dentin as a porous, biologic composite made up of apatite crystal filler particles in a collagen matrix (Perdigão, 2010). It consists of 69% polycrystalline apatite, 13% water, and 18% proteins that are mainly type I collagen by weight (DiRenzo *et al*, 1995). By volume the dentin consists of 50% minerals in the form of carbonate rich, calcium deficient apatite; 30% organic material in the form of collagen type I in addition to other non collagenous proteins, and 20% fluid similar to plasma (Marshall *et al*, 1997).

Structurally dentin consists of a network of dentinal tubules. Each tubule has a diameter of 1 - 3µm. The average density of the dentinal tubules is about 30,000 tubules/mm^2. The dental pulp gives rise to elongated cell bodies that radiate throughout the entire dentin encased by the dentinal tubule. Surrounding the dentinal tubule is the peri-tubular dentin while the intertubular dentin lies between the tubules. Both the peri- and intertubular dentin form the circumpulpal dentin which results in the greater surface area. Mineral crystals of calcium hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$] are deposited between the peri- and intertubular dentin. These apatite crystals are associated with a network of collagen fibers. A characteristic feature of the peritubular dentin is the lack of a fibrous structure and the presence of a highly mineralized sheath surrounding the tubules (DiRenzo *et al*, 1995).

Adhesion to dentin is more complicated because it is more hydrated as a result of its intrinsic wetness through the dentinal tubules (Marshall *et al*, 1997). The use of phosphoric acid as an etching media was found to have a severe pulpal reaction in the early dentin bonding studies (Retief *et al*, 1974). As a result; it was accepted at that time to avoid any contact of the phosphoric acid with the dentin, but Davis *et al* (1992) showed that the use of 37% phosphoric acid was effective in dentin etching and did not cause post-operative sensitivity provided it was only used for a very short period (10 seconds). The phosphoric acid caused demineralization of the inorganic matrix over a depth of 3 to 5µm thereby exposing collagen fibrils depleted of hydroxyapatite (Van Meerbeek *et al*, 2005).

The micro-mechanical adhesion resulted from the mechanical interlocking of the exposed collagen fibrils to the resin polymer i.e. the exposed collagen fibrils acted as a micro-retentive network for the micro-mechanical interlocking. The main drawback of the acid etch technique of dentin was that the collagen fibrils collapsed and shrunk following drying of the acid-etched dentin, thereby jeopardizing the monomer infiltration into the dentin with a resultant diminishing of the bond strength (Gwinnett, 1994).

The next step was the application of an adhesion promoting monomer “primer” to the dentin surface in order to overcome the shrinkage of the collagen fibrils that followed the acid etching and drying steps. The primer consisted of a mixture of monomers that possessed hydrophilic properties. An example of these monomers is HEMA (Hydroxy Ethyl Methacrylate) which has a low molecular weight and hydrophilic properties. The primer prepares the dentin surface for the adhesive resin penetration into the dentinal tubules by providing sufficient wetting of the exposed collagen fibrils and removing the remaining water from the dentinal tubules (Van Meerbeek *et al*, 2005).

Following this preparation of the dentin surface for adhesive resin penetration, the formation of a hybrid layer resulted. Its formation was considered as the basic mechanism for resin-dentin bonding. It results from the intertwining of the resin into the demineralized dentin collagen fibril network (Yang *et al*, 2005). The hybrid layer consisted of resin that infiltrated into the collagen network, residual hydroxyapatite, and traces of water (Perdigão, 2007). The resin filled up the interfibriller spaces between the collagen fibrils and formed resin tags upon curing thus forming a hybrid layer that provided the bases of micromechanical retention to dentin (Van Meerbeek *et al*, 1993).

Considering adhesion to dentin two problems could occur. The first involves the collapse and shrinkage of the collagen fibrils that follows acid etching; this would prevent proper infiltration of the adhesive resin. The second is that; etching of the dentin surface removes the minerals from the surface leaving the collagen fibrils suspended in water. However a certain amount of water is needed to prevent collagen fibril shrinkage. To overcome these two problems two different techniques were suggested depending on the composition of the primer in the adhesive system (Van Meerbeek *et al*, 2005).

The first technique involves air-drying of the dentin following acid etching and applying a water-based primer which re-expands the collagen fibrils; this technique is known as dry-bonding. The second technique involves leaving the dentin surface slightly wet and the use of an acetone based primer to remove the water during the infiltration of the resin; this technique is known as wet-bonding (Pashley *et al*, 2007).

2.2.4. Classification of dentin bonding agents:

There are different classification systems for dentin bonding agents. Most dental companies classify dentin bonding systems into six generations according to the chronology of their release to the dental market (Murray *et al*, 2003).

The first generation of dentin bonding agents were developed during the period 1950 to 1970. This generation of dentin bonding agents were based on the use of an adhesive containing polymerized compound with polar groups that had an affinity to bond to the tooth structure (Bayne, 2002, Burke and McCaughey, 1995). In 1955 Buonocore used cavity primer that containd glycerophosphoric acid dimethacrylate that interacted with the calcium ions of the dentin structure. This link between the phosphate group and the calcium ion was easily hydrolyzed resulting in weak bond strength values (Stangel *et al*, 2007).

In Masuhara *et al* (1962), cited by Bayne (2002), used tri-n-butyl-borane to facilitate adhesion to dentin collagen. The main drawback of this system was the weak bond strength that resulted from poor surface wetting of the dentin surface. Bowen improved the surface wetting of the dentin by introducing the surface active N-phenylglycine and glycidymethacrylate (NPG-GMA) in 1965 (Burke and McCaughey, 1995). All of these materials had weak bond strength values between the dentin and the composite resin and were termed the first generation dentin bonding systems (Bayne, 2002).

The second generation dentin bonding agents were introduced in the early 1970s (Bayne, 2002). This generation used Bis-GMA as a replacement of the methacrylate. The basis of the adhesion was the phosphate-calcium bond type. The bond strength values were greater than the first generation but nowhere near those achieved with enamel bonding. Other materials in this group used a 2-hydroxyethyl methacrylate (HEMA) and phenyl phosphate ester which resulted in slightly better bond strength values (Burke and McCaughey, 1995).

The third generation dentin bonding agents were developed in the late 1970s (Bayne, 2002) by using a solution that modified the dentin surface before the application of the resin. This generation produced a mechanical basis of adhesion by using hydrophilic and hydrophobic groups that penetrated the dentin tissue and polymerized in that position creating a hybrid layer that was neither resin nor tooth structure but an integrated mixture of the two (Burke and McCaughey, 1995). This was in contrast to just the chemical bond utilized by the first two generations of dentin bonding agents.

The fourth generation dentin bonding system was introduced in the 1990s (Combe *et al*, 1999) or mid to late 1980s (Bayne, 2002). These systems used phosphoric acid for etching both the enamel and the dentin followed by the application of a primer and a bonding agent (Combe *et al*, 1999). The use of phosphoric acid removed the smear layer (Bayne, 2002) and resulted in an improvement in the bond strength values with a subsequent improvement in the resistance of the restorative material to displacement and marginal leakage (Combe *et al*, 1999).

Also in the 1990s the fifth generation dentin bonding agents were introduced (Bayne, 2002). There were based on a reduction in the number of clinical application steps by combining the primer with the adhesive in one bottle (Combe *et al*, 1999). This system used a hydrophilic agent for both enamel and dentin giving the benefit of bonding to moist dentin (Bayne, 2002).

The sixth generation of dentin bonding agents were developed in the late 1990s and had the etchant and the primer combined in one step and hence the name self-etching primers. While in the seventh generation all the clinical application steps were combined in a single step (Bayne, 2002) and hence the name one-step bonding agents.

In recent years, the eighth generation dental adhesive system was introduced as a self-adhering composite resin (Vertise Flow, Kerr, Orange, CA, USA) in which the bonding agent is combined with a flowable composite, thus eliminating the step of application of the adhesive system. Vichi *et al*, 2010 investigated the clinical behaviour of Vertise Flow the self-adhesive composite resin in small sized class I restorations for a 6 month period. They assessed the post-operative sensitivity, marginal discoloration, marginal integrity, secondary caries, maintenance of interproximal contact, and fracture at baseline. They concluded that; Vertise Flow (self-adhesive composite resin) had a successful clinical outcome when used to restore small class I cavities (Vichi *et al*, 2010).

The other classification system of dentin bonding agents is a little confusing. In the Buonocore memorial lecture in 2003 Van Meerbeek *et al*, named three categories of dentin bonding systems according to their adhesive strategy. These were classified as the etching and rinse adhesives, the self-etch adhesives, and the glass ionomer adhesives (Van Meerbeek *et al*, 2003, Van Meerbeek *et al*, 2005). Another name for the etching and rinse adhesive is the total-etching technique. The system based on the etch-rinse and the self-etch techniques are bonded to the dentin through micromechanical retention as a result of the formation of the hybrid layer. While the glass ionomer adhesives bond to tooth structure through micromechanical and chemical retention after the formation of the hybrid layer and the ion exchange that occur between the glass ionomer and the tooth structure (Van Meerbeek, 2005).

Another classification system is based on the number of the clinical application steps involved in the process of bonding namely etching, priming, and bonding to tooth structure and are therefore categorized into three groups; the three-step, the two-step, and the single-step bonding systems (Van Meerbeek *et al*, 2003).

The three-step adhesive system consists of etching, priming, and the application of the adhesive in which each agent is applied separately (Tay *et al*, 2004). The three-step adhesive system uses phosphoric acid for etching both the enamel and the dentin and hence the name total-etch. The acid etching produces microporosities so that the resin binds micromechanically to the tooth structure through resin tag formation. An adequate seal of the pulp or an adequate remaining dentin thickness is necessary for this in process order to prevent the adverse effect of the acid on the pulp during the acid etching phase (Naughton and Latta, 2005). The application of the dentin primer to the demineralized dentin enhances the monomer diffusion and facilitates the formation of the hybrid layer after the application of the bonding agent

(Hayakawa *et al*, 1998). The system also contains hydrophilic monomers that enhance the wettability of the hydrophobic resin thereby enhancing the bond (Miyazaki *et al*, 1998).

In comparison the two-step dentin bonding systems have eliminated an application step from the three-step systems and can be further subdivided into self-priming adhesives and self-etching adhesives combining the chemical components. The self-priming adhesives combine the priming and the application of the adhesive into a single step while keeping the etching step separated. The self-etching adhesives on the other hand use an acidic monomer in a high concentration in the primer so that a self-etching primer is created i.e. enabling the system to combine the etching and the priming in one step (Tay *et al*, 2004). An example of the acidic monomer is the phosphate ester or carboxylic acid, and hydroxyethyl methacrylate (HEMA) (Hayakawa *et al*, 1998). The acidic monomer is ionized by the addition of water to the adhesive system. This ionization allows the acid to cause demineralization of the tooth structure. Unlike the total-etching adhesives the self-adhesive system has the advantage of better resin infiltration into the hybrid layer (Tay *et al*, 2004).

The single-step adhesive system consists of etching, priming, and the application of the adhesive all being combined in one step (Tay *et al*, 2004). The demineralization, conditioning and infiltration of the adhesive into the enamel and dentin occur simultaneously without the rinsing that normally follows the etching procedure. This results in an alteration of the smear layer without its removal (Naughton and Latta, 2005). The primer substance is either HEMA (hydroxyethyl methacrylate) or PENTA (di-pentaerythritol penta-acrylate monophosphate) together with a low viscosity resin. Ethanol or acetone is added as a solvent so that it acts as a water chaser and facilitates the exchange of the water for the monomer inside the collagen web (Cardoso *et al*, 1998).

The bond strength values of the three step dentin bonding agents are high, but these values can be affected by some factors such as excessive etching of the dentin in which case weak bonds will be produced as a result of the incomplete resin infiltration into the base of the collagen fibers (Paul *et al*, 1999). In addition to that the collagen fibers also shrink during the air drying procedure following acid etching resulting in poor resin infiltration between these fibers and thus reducing the bond strength. Furthermore the moisture contamination after etching can affect the bond strength when using the three step adhesive system (Bouillaguet *et al*, 2001). The later products in the generation of the three-step dentin bonding systems were able to produce bond strength values varying from 9 to 18 MPa (Naughton and Latta, 2005).

The difference between the shear bond strength of a two-step dentin bonding agent and a single-step dentin bonding agent was investigated by Hegde and Bhandary in 2008, and they concluded that the two-step dentin bonding agent produced higher bond strength values to dentin when compared to that of the single-step dentin bonding agent (Hegde and Bhandary, 2008).

Miyazaki *et al* investigated the shear bond strength of the two-step dentin bonding systems following thermal cycling and found that there was no significant difference in the bond strength in the self-etching primer sub-system, while there was a significant decrease in the bond strength in the self-priming sub-system (Miyazaki *et al*, 1998).

Other studies suggested that the combination of the etching and the priming in a single step as in the self etching adhesive system reduced the effectiveness of the hybrid layer (Bouillaguet *et al*, 2001); however the effect of the combination on the bond strength was unclear (Sano *et al*, 1999).

Bouillaguet *et al* investigated the microtensile bond strength of composite resin to dentin using a one step adhesive system and found that it ranged between 13.8 and 18.9 MPa with most of the failures occurring at the top of the hybrid layer when using scanning electron microscopy (SEM) to view the fractured sites (Bouillaguet *et al*, 2001).

2.3 Bond strength

2.3.1 Bond strength tests:

Bond strength tests are used for the determination of the strength of the bond between the restorative material and the tooth structure (Cheng *et al*, 1999).

There are several bond strength test methods that have evolved as a result of the continuous development and improvements in the adhesive systems that are introduced into the dental market on a regular basis. As a result of this improvement in the materials, the adhesion testing methods have also improved over time. However this improvement in testing methods has resulted in a lack of standardization between these test methods. The lack of standardization has resulted in the influence of numerous variables that could affect the bond strength values. These variables include substrate type and preparation, etching, priming, bonding, storage and testing methods (Pashley *et al*, 1995). Although the bond strength tests are used to evaluate the adhesive strength of a restorative material, there is little correlation between these tests and *in vivo* findings (Rasmussen, 1996).

Bond strength values are determined by tensile or shear bond strength tests. The bond strength value is obtained by dividing the load at failure by the cross sectional area of the bonded surface (Cheng *et al*, 1999). This is referred to as the nominal or average bond strength value (Placido *et al*, 2007). In a tensile test the material is stretched in a uni-axial direction in a tensile tester. While in a shear bond strength test pressure is applied to the material until it fractures. The shear bond strength test has the advantage of ease of specimen preparation and is a relatively simple test procedure. However cohesive failure in the substrate is observed with the newer adhesive systems resulting in the conclusion that this test is unsuitable to determine the true bond strength with especially materials with high bond strength values (Placido *et al*, 2007). Della Bona and Van Noort explained that this cohesive failure was due to the fact that the stress was concentrated in the substrate rather than in the adhesive which resulted in a premature failure prior to the failure at the interface itself (Della Bona and Van Noort, 1995).

Van Noort *et al*, 1989 compared the nominal bond strength value between the tensile and the shear bond strength tests and found that it varied according to the specimen geometry, loading configuration and material properties. The bond strength value was found to be more sensitive in the shear tests than in the tensile tests. Based on this study the tensile bond strength test is preferred to the shear bond strength test (Van Noort *et al*, 1989).

In 1994 the microtensile bond strength test was introduced. It uses a very small surface area of the specimen ($1mm^2$) (Scherrer *et al*, 2010). However this test is very sensitive and time consuming, but it allows a uniform distribution of stress due to the small surface area of the specimen and allows the use of multiple specimens from a single tooth (Goracci *et al*, 2004). Pashley *et al* listed several advantages of the microtensile bond strength test including; the exclusive ability to test adhesive bond failure of a material if the bonded surface area is $1mm^2$, high interfacial bond strength values that could be easily measured, it also permits the measurement of regional bond strength values, it also allows for means and variances to be calculated for a single tooth, it also permits testing of bonds made to irregular surfaces, permits testing of very small areas, and facilitates SEM examination of the failed bonded surfaces as the surface area in only $1mm^2$. They also listed several disadvantages of the microtensile bond strength test namely that it technically demanding, difficult to measure the bond strength values if it is less than 5MPa, requires special equipment to prepare the specimens , and samples are so small that they can be hydrated very rapidly (Pashley *et al*, 1995).

Recently other methods were advocated by McDonough *et al* (2002) to measure the bond strength under shear load. One such method called the micro-shear or micro-bond strength test uses specimens with a small dimension and allows testing the bond strength in a small area. In this way the test permits regional mapping or depth profiling of different substrates. It also allows the use of different specimens from the same tooth as in the microtensile bond strength test with the main difference being the lack of a sectioning procedure in the micro-shear bond strength test (McDonough *et* al, 2002).

2.3.2 Thermal effect on bond strength:

The oral cavity is constantly subjected to thermal change as a result of the intake of food and fluid at varying temperatures. This temperature change can cause dimensional changes of the tooth structure as well as the restorative material. This temperature change is expressed as the coefficient of thermal expansion (Sidhu *et al*, 2004). The coefficient of thermal expansion of the restorative material should be similar to or near to that of the coefficient of thermal expansion of the tooth; otherwise a gap will be formed between the restorative material and the tooth structure (Nelsen *et al*, 1952). Furthermore loosening and debonding of the restorative material can also occur (Yamaguchi *et al*, 1989). The coefficient of thermal expansion of the tooth structure has been found to be between 11 – $14x10^{-6}$ ^{0}C (Sidhu *et al*, 2004) while that of the restorative material such as a composite resin is in the range of 20 to 80 x 10^{6}/^{0}C (Versluis *et al*, 1996).

Rossomando and Wendt defined the thermal-cycling as an *in vitro* process of subjecting a restoration and a tooth to temperature extremes that resemble to those found in the oral cavity (Rossomando and Wendt, 1995). The different thermal changes that the dentition is subjected to during food consumption can be simulated in *in vitro* studies by thermal-cycling of the specimens to temperatures ranging between 5 and 55^{0}C (Soh and Selwynb, 1992). The thermal-cycling regimen in most studies is comprised of 500 cycles in water between 5^{o}C and 55^{o}C as indicated by the ISO TR 11450 standard established in 1994 (De Munck *et al*. 2005). However 10,000 *in vitro* cycles correspond to one year of *in vivo* functioning as concluded from the work of Gale and Darvell in 1999.

In adhesive dentistry more so than in other aspects of dentistry the influence of the intra-oral thermal changes can be substantial as it affects the restorative material and/or the adhesive system. The composite resin restorative material adheres micromechanically to the dentin after formation of the hybrid layer by the dentin bonding systems (Yang Lee *et al*, 2001). During visible light polymerization of the composite resin the temperature of the composite resin increases as a result of the polymerization reaction within the composite resin and due to the heat output from the dental light curing unit (Stewardson *et al*, 2004). According to Strang *et al* the most significant source of heat during the polymerization of a light activated restorative material is from the light activation unit and not from within the material itself (Strang *et al,* 1988). To reduce the amount of the rise in temperature during visible light polymerization of composite resin Masutani *et al* recommended an incremental placement and polymerization of the composite resin together with the use of a moderate intensity light for the polymerization process (Masutani *et al*, 1988). Goodis *et al*

(1990), Hannig and Bott (1999), and Porko and Hietala (2001) investigated the effect of thermal changes during the polymerization of the composite resin on the pulp. They concluded that all the visible light-curing units tested caused a measurable increase in the pulpal temperature within the range of pulpal physiology. Moreover the type of the light curing unit also influenced the amount of the rise in temperature that resulted during the curing of the composite resin in that the newer model lights caused a lower increase in pulpal temperature. On the other hand, the cooling effect of the rinsing water following acid etching could also cause a rapid decrease in the pulpal temperature, but the overall effect of thermal changes during polymerization on the bond strength of dentin bonding agents is not completely resolved.

During consumption of hot foods heat is generated that can reach a maximum of 47^0C during consumption (Musanje and Darvell, 2004). This increase in temperature generates heat on the composite resin that affects the expansion of the material as well as the dentin bonding agent due to the difference in the coefficient of thermal expansion resulting in stresses that are generated that are sufficient to cause interfacial debonding between the different components involved in the bond. The amount of temperature needed for the debonding gives an indication of the bond strength of the adhesives (Yang Lee *et al*, 2001).

The thermal changes experienced in the mouth induce stresses in the bonded interface between the restorative material and the tooth structure; compromising the adhesive material and resulting in reduced bond strength values (Asaka *et al*, 2007). Another effect of the repeated thermal changes is the acceleration of the hydrolysis of the bonded interface component by the fluids resulting in debonding of the restoration (De Munk *et al*, 2005).

The effect of thermal-cycling on the shear bond strength of the single-step dentin bonding agent was investigated by many researchers including Asaka *et al* 2007 who concluded that the thermal cycling generated different thermal conductivities that resulted in mechanical stresses on the bonding agent. They also concluded that the size of the bonded composite resin influenced the durability and the strength of the adhesive system implying that in large composite fillings a portion of the bonded interface may be protected from the thermal changes (Asaka *et al*, 2007).

Naughton and Latta (2005) also investigated the effect of thermal-cycling on the shear bond strength of a self-etching single step adhesive system like Xeno III and concluded that thermal-cycling did not affect the shear bond strength value of this material (Naughton and Latta, 2005). However this finding differed from the findings reported by El Araby and Talic in 2007 (El Araby and Talic, 2007)

On the other hand the effect of thermal-cycling on the two-step dentin bonding agent was also investigated by a number of researchers including, Miyazaki *et al* (1998) who investigated the influence of thermal-cycling on the dentin bond strength of a two-step bonding system using a self-etching primer system and a self-priming adhesive system and found that the dentin bond strength values decreased as the number of thermal cycling episodes increased with a significant decrease in the bond strength values of the self-priming systems with only a small decrease in the bond strength of the self-etching primer systems (Miyazaki *et al*, 1998). This result was similar to that reported by Alaa and Talic in 2007 (El Araby and Talic, 2007). However Santos *et al* (2005) showed that thermal-cycling did not affect the shear bond strength value of the two-step dentin bonding agent like Prime and Bond NT (Santos *et al*, 2005).

2.4 Failure of adhesive bond caused by thermal-cycling:

The stresses that are created by repeated contraction and expansion due to thermal changes may lead to crack formation along the bonded interface. Once this crack is formed, it continues to increase as a result of the movement of the oral fluids into and out of the gap (Gale and Darvell, 1999).

Furthermore water within the gaps causes hydrolysis of the resin by the breakdown of the covalent bonds between the polymers. The degree of resin hydrolysis is also related to the amount of water sorption by the hybrid layer. Studies have shown that the hydrophilic acidic resin systems that are used with the self-etch adhesives have a higher water sorption ability when compared to the hydrophobic resins which have a low water sorption capacity. Other than resin hydrolysis by water, water sorption also causes a significant decrease in the modulus of elasticity of the resin with a subsequent decrease in the bond strength (Breschi *et al*, 2008).

Moreover this water sorption also results in a loss of the resin from the interfibrillar spaces, resulting in a disorganization of the collagen fibrils with a subsequent degradation of the collagen fibers in the hybrid layer, thereby further weakening the physical properties of the bonded resin-dentin interface (Hashimoto *et al*, 2003).

2.5 Composite resin:

Composite resin is one of the direct esthetic restorative materials that is used daily in dental practice. It is defined as a multiphase material that exhibits the properties of both phases and results in a new enhanced material (Roberson *et al*, 2002). According to Rawls and Esquivel-Upshaw, (2003) a dental composite is defined as a highly cross-linked polymeric material reinforced by a dispersion of amorphous silica, glass, crystalline, or organic resin filler particles and/or short fibers bonded to the matrix by a coupling agent.

2.5.1 Historical background:

The first tooth-colored filling material was introduced in the 1870s as a silicate cement which was based on an alumino-fluro-silicate glass as the “dispersed phase” and phosphoric acid (Puckeet, *et al*, 2007). In the 1940s the first polymeric tooth-colored composite was introduced to minimize the disadvantages of the acrylic resin and the silicate cements (Hervás-Garcia *et al*, 2006). This new material consisted of a poly (methylmethacrylate) powder, methyl-methacrylate monomer, benzoyl peroxide, and n,n-dimethylparatoluidine. However this material had a number of disadvantages including poor color stability, high polymerization shrinkage, lack of bonding to tooth structure, and a large coefficient of thermal expansion (Roberson *et al*, 2002). In 1962 Bowen attempted to improve the physical properties of acrylic resin by developing the Bis-GMA compound (Bowen, 1963). Since that time the dental composite resins have undergone continuous improvement in their mechanical and esthetic properties.

2.5.2 Chemical composition of composite resin:

Chemically, composite resins consist of two phases an organic matrix phase and an inorganic dispersed phase. The two phases are combined by a surface interfacial phase (Hervás-García *et al*, 2006).

2.5.2.1 Organic matrix phase:

The organic matrix phase represents 40% to 50% of the composite by volume. It consists of polymers and remnants from monomers, organic compounds, polymerization inhibitors, chemical initiators, accelerators, and ultraviolet and visible photoactivators (Craig, 1981).

The major component of the organic matrix phase is the Bisphenol-A glycidylmethacrylate (Bis-GMA) or Bowen's resin (table 2.1) which is a high molecular weight monomer that has high viscosity (Bowen, 1958). Other composites also use additional high molecular weight monomers based on a urethane dimethacrylate (UDMA) in conjunction with Bis-GMA and diluents (Puckeet, *et al*, 2007). Recently the use of a liquid crystalline monomer and a ring opening resin such as oxiranes, spiro ortho ester, spiro ortho carbonates, and silorane as a resin matrix have been introduced to decrease the polymerization shrinkage in the composite resin (Weinmann. 2005).

Table 2.1: Characteristics of composite resins based on Puckeet *et al*, 2007;

Composite classification	Weight %	Volume %	Volume shrinkage %	Average particle size (µm)
Hybrid	74-87	57-72	1.6-4.7	0.2-3.0
Macrohybrid	72-87	58-71	2.0-3.4	0.4-0.9 (macro)
Nanohybrid	____	____	____	0.015-0.05 (nano)
Microfills	35-80	20-59	2-3	0.04-0.75
Flowable	40-60	30-55	4-8	0.6-1.0
Compomers	59-77	43-61	2.6-3.4	0.7-0.8

To overcome the high viscosity of the resin matrix, an organic compound such as bisphenol A dimethacrylate (Bis-DMA), ethylene glycol dimethacrylate (EGDMA), triethylene glycol dimethacrylate (TEGDMA), methyl methacrylate (MMA), or urethane dimethacrylate (UDMA) is added to the resin matrix (Puckeet *et al*, 2007).

The organic matrix of the light-cured composites also contain photoinitiators such as alpha diketone (camphoroquinone) and co-initiators like amines that when exposed to light in the proper wave length produce free radicals that initiate the polymerization reaction (Benjamin, 2003). Whereas in the chemically-cured composite resins the chemical initiators consist of benzoyl peroxide that is used in combination with an aromatic tertiary amine (n,n-dihydroxyethyl-p-toluidine) and an acceleration system (dimethylaminoethyl methacrylate or DMAEM, ethyl-4-dimethylaminobenzoate or EDMAB, or N,N-cyanoethyl-methylaniline or CEMA) which initiates the polymerization reaction (Koblitz *et al*, 1977).

To maximize the shelf life of the composite resin before curing a stabilizer or an inhibitor such as hydroquinone monomethyl ether is added to the resin matrix. In addition to that an ultra-violet light absorber such as 2-hydroxy-4-methoxybenzophenone is also added to the resin matrix (Hervás-García *et al*, 2006).

2.5.2.2 Inorganic dispersed phase:

The filler content varies from one product to another but usually represents 55% by volume and 78% by weight of the composite resin (Dennison and Craig, 1972). The filler particles are added to the resin matrix in order to improve the physical and mechanical properties of the material as the filler particles reduce the coefficient of thermal expansion, reduce the polymerization shrinkage, provide radio-opacity, and improve the handling and aesthetic properties of the material (Labella *et al*, 1999). The main filler particles found in dental composite resins include silicon dioxide, boron silicate and lithium aluminium silicates (Xu, 1999).

Lutz and Philips, (1983) classified the inorganic filler particles according to the size of the particles into three types namely; traditional macrofillers, microfillers (pyrogenic silica), and microfiller-based complexes in which the traditional macrofillers have a particle size ranging from 0.1 to 100 μm while the microfillers have an average particle size of 0.04 μm. The microfiller based complexes can be further subdivided according to the manufacturing technique into splintered prepolymerized microfilled complexes, spherical polymer-based microfilled complexes, and agglomerated microfiller complexes. The microfiller based complexes have a particle size ranging from 1 to 200 μm.

2.5.2.3 Interfacial phase:

In order to improve the mechanical properties of the resin composite and to transfer the high stresses from the high strength filler particles to the more delicate resin matrix a good bond must exist between the resin matrix and the inorganic filler particles. This bond is achieved by addition of a coupling agent such as Ƴ-methacryloxpropyl-trimethoxy silane or vinyl triethoxysilane to the filler particles (Puckeet *et al*, 2007).

The mechanism by which the coupling agent binds the resin matrix to the inorganic filler particles is thought to be due to hydrolysis of the methoxy groups with bound surface water on the inorganic filler particles or with the silanol group of the filler (Craig, 1981).

2.5.3 Classification of dental composites:

There have been a number of classification systems for composite resins. One of the most common used classification systems is based on the size of the filler particles.

In 1983 Lutz and Philips classified composite resins according to the type of the filler particles they contained into the traditional composite resin, the hybrid composite resin, the homogeneous microfilled composite resin, and the heterogeneous microfilled composite resin. The traditional composite resin is composed of traditional macrofillers, the hybrid composite resin is composed of traditional macrofillers and microfillers, the homogeneous microfilled composite resin is composed of microfiller, while the heterogeneous microfilled composite resin is composed of microfillers and microfiller-based complexes (Lutz and Philips, 1983). This classification is illustrated in figure 2-1

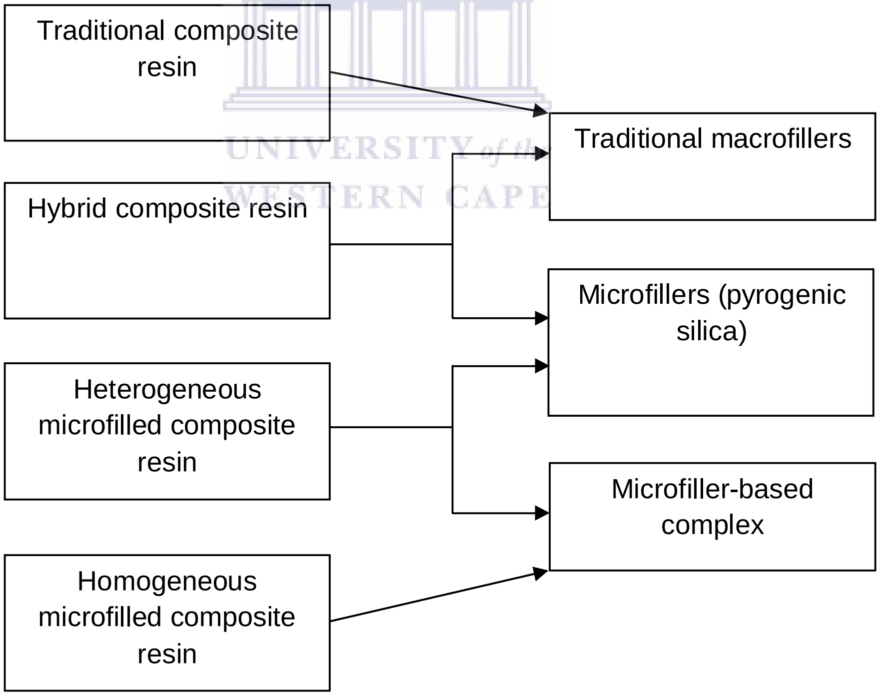


Figure 2.1: Classification of composite resin based on the content of the filler type.

Another classification promoted by Roulet in 1987 was similar to the Lutz and Philips classification with the only difference being in the number of composite resin classes. Roulet classified the composite resins into; traditional composites, hybrid composites, homogenous microfilled composites, and inhomogeneous microfilled composites. The inhomogeneous microfilled composites were further sub-classified into the splintered pre-polymerized particles, spherical polymer-based microfilled complexes, and the agglomerated microfilled complexes (Lang *et al*, 1992).

In 1988 Marshall *et al*, cited by Lang *et al* (1992), classified composite resins according to the amount of filler by volume and weight into; unfilled composite resins, microfilled composite resins, hybrid composite resins for anterior restorations, macrofilled composite resins, midifilled composite resins, and hybrid composite resins for posterior restorations (Lang *et al*, 1992).

The most common classification used is based on the size and distribution of the filler particles. This classification classifies the composite resins into; microfills, hybrid, packable, and compomers. The microfill composite resins are further subclassified into flowable composite resins, and hybrid composite resins. The hybrid composite resins are subdivided into the nano-and microhybrid composite resins (Puckeet *et al*, 2007). The packable composite resins were developed to improve the handling properties of the composite resins. This property was obtained by the addition of fibers to its composition. A summary of the attributes of each type of composite resin is presented in table (2.1).

The other system used for the classification of composite resins is based on its polymerization initiation technique where they are classified into chemical, light and dual-curing composite resins (Willems *et al*, 1992).

2.5.4 Properties of composite resins:

The properties of the composite resins are continuously changing as a result of the continuous development in their composition, the size and distribution of the filler particles, and also due to the different methods of curing of the composite resin (Von Fraunhofer and Curtis, 1989).

2.5.4.1 Polymerization shrinkage:

Composite resins undergo volumetric shrinkage as a result of the polymerization process. This shrinkage results in the loss of marginal integration at the cavosurface margin and can induce destructive stresses in the bonded esthetic restorations which ultimately affects the quality of the composite restoration (Visvanathan *et al*, 2007).

In addition post operative sensitivity, marginal staining, and eventually recurrent caries can result from polymerization shrinkage. The polymerization shrinkage can also lead to cuspal displacement and even crack propagation of the healthy tooth structure as a result of the stresses created in the tooth-restorative interface (Weinmann et al, 2005).

The intensity of the curing light and the time of application will determine the efficiency of polymerization (Strydom, 2002). The use of a high intensity curing unit decreases the curing time but it creates high polymerization stresses at the tooth-restorative interface which can result in microleakage (Silikas *et al*, 2000). As a result the use of a two-step polymerization technique in which the composite resin is cured initially using a low intensity curing unit followed by a high intensity unit, can result in a decrease in the polymerization shrinkage stress as a result of prolonging the composite resin polymerization time (Visvanathan et al, 2007).

In addition to the intensity of the curing unit, polymerization shrinkage can also be affected by the filler content of the composite resin. The polymerization shrinkage is known to decrease as the filler content increases (Herrero et al, 2005).

Polymerization shrinkage tends to be towards the acid-etched enamel more than to the bonded dentin. This is based on the premise that the bonded strength to enamel is greater than that to dentin. As a result of this the composite resin tends to move towards the enamel surface leading to the creation of a microgap between the dentin and the restorative resin which could result in post-operative sensitivity and marginal leakage at the dentin margin (Gamborgi et al, 2007).

Several options have been suggested to overcome this problem. The use of a chemically cured composite resin as a base under the light cured composite resin can be one of the options. The chemically cured composite resin is thought to shrink towards the dentin surface (Kinomoto *et al*, 1999). In addition an incremental pattern of placement of the composite resin will also facilitate the polymerization shrinkage of the composite resin towards the cavity wall (Van Noort. 2002 (b)).

Polymerization shrinkage can also be minimized in deep cavities by curing some of the composite resin outside the cavity and then placing it in the cavity and continuing with composite resin application incorporating the extraorally cured composite resin (Braga et al, 2003).

Recently attempts were made to reduce polymerization shrinkage by changing the nature of the resin matrix to that of a liquid crystalline monomer as the resin matrix, and silorane resin (Weinmann *et al*, 2005).

2.5.4.2 Thermal properties:

The coefficient of thermal expansion of composite resins depends on the inorganic filler content. It was found that the coefficient of thermal expansion decreases as the filler content of the composite resin increases (Chung, 1990). Craig, (1981), showed that the coefficient of thermal expansion of the unfilled composite resin to be in the range between 80 to 90×10^{-6} per ^{0}C, while that of the filled composite resin to be in the range between 46 to 70×10^{-6} per ^{0}C. While the coefficient of thermal expansion of the tooth structure was found to be in the range between 26 to 40×10^{-6} per ^{0}C (Craig, 1981).

This difference in the coefficient of thermal expansion of the tooth structure and the composite resin can result in the formation of a gap between the composite resin and the tooth structure (Asmussen, 1985).

2.5.4.3 Mechanical properties:

Craig, (1981) listed a number of mechanical properties of composite resins that include compressive strength, compressive fatigue limit, diametral tensile strength, yield strength, transverse strength, shear strength, elastic modulus, Pisson’s ratio, modulus of elasticity, fracture toughness, Rockwell hardness, indentation depth, recovery from indentation, and wear. The mechanical properties are some of the factors that determine the longevity of composite resins *in vivo* (Von Fraunhofer and Curtis, 1989). The mechanical properties of the composite resins are expressed in the compressive strength and the tensile strength of the material (Asmussen, 1985).

2.5.4.3.1 Compressive and tensile strength:

Anusavice, (2003) defined compressive strength as “the compressive stress (in a compression test specimen) at the point of fracture” and he also defined tensile strength as “the tensile stress (in a tensile test specimen) at the point of fracture”.

The compressive strength of the microfilled composite resin is usually higher than that of the macrofilled composite resin. However the compressive strength has no clinical significance (Asmussen, 1985).

On the other hand the tensile strength is an indicator of the cohesion of the material (Von Fraunhofer and Curtis, 1989). The tensile strength of composite resin is dependent on the filler particle size and its concentration (Covey *et al*, 1992). Asmussen, (1985) showed that the macrofilled composite resins had a higher tensile strength compared to the microfilled composite resins. In addition to that, the degree of conversion of the resin monomer to polymer during composite resin polymerization had a direct relationship to the tensile strength, implying that a high degree of conversion could result in a higher tensile strength (Covey *et al*, 1992).

2.5.4.5 Fracture toughness and mode of fracture:

Fracture toughness is one of the intrinsic features of a composite resin material that measures the resistance of that material to the spreading of a crack (Kovarik *et al*, 1991). Regarding the failure of a composite resin restorative, fracture within the body of the restoration and at the margin are the major types of failure modes (Roulet, 1988). It has been shown that the filler volume fracture and filler load level of the composite resin has a direct relation with the material strength and fracture toughness of the material (Germain, *et al*, 1985).

Chapter 3
Aims and Objectives

3.1. Aims of the study:

- To compare the bond strength of a two-step and a single-step dentin bonding agent to dentin.
- To compare the effect of thermal-cycling on these bond strength values.

3. 2. Objectives of the study:

- To determine the bond strength of a two-step and a single-step dentin bonding agent to dentin.
- To determine the effect of thermal-cycling between 5^0C and 55^0C on the bond strength of these dentin bonding agents.

3. 3. Null hypothesis:

- There is no significant difference in the bond strength of the two-step and the single-step dentin bonding agents.
- Thermo-cycling between 5^0C and 55^0C has no effect on the bond strength of the two-step and the single-step dentin bonding agents.

Chapter 4
Materials and Methods

4.1 Study design:

This was an in vitro experimental study.

4.2 Sample size:

100 extracted human molar teeth were used in this study**.**

4.3 Inclusion criteria:

Only non-carious and non-restored human molar teeth extracted for orthodontic or prophylactic reasons were used in the study.

4.4 Exclusion criteria:

- Teeth extracted due to dental caries.
- Extracted teeth with restorations or cracks.

4.5 Ethical considerations:

- The teeth collected for this study were extracted for reasons other than the purpose of this study.
- On completion of this study, the teeth were disposed according to the current medical waste disposal practice at the Faculty of Dentistry/ Oral Health Centre, University of the Western Cape.
- The materials used in this study were supplied by the Faculty of Dentistry/Oral Health Center, University of the Western Cape.
- No financial support neither before nor after the completion of this study was received from the company producing the materials used in this study.

4.6 Materials used:

- TPH®3 Spectrum Micro Matrix composite restorative material by Dentsply (Canada):

  Resin matrix: Bis-EMA and triethylene glycol dimethacrylate.

  Photoinitiator: Camphorquinone (CQ).

  Stabilizer.

  Pigments.

  Filler type: barium alumino baro silicate glass and barium fluoro alumino baro silicate glass.

Figure 4.1: TPH®3 Spectrum (micro matrix composite restorative material).

- Xeno V: one component light-cured self-etching dental adhesive by Dentsply, (Canada):

  Bifunctional acrylates.

  Acidic acrylate.

  Functionalized phosphoric acid ester.

  Acrylic acid.

  Water.

  Tertiary butanol.

  Initiator.

  Stabilizer.

Figure 4-2: Xeno V (single-step dentin bonding agent).

- Prime and Bond NT (nano-technology dental adhesive): light-cure self priming dental adhesive by Dentsply (Canada):

  Di-and Trimethacrylate resin.

  Dipentaerythritol penta acrylate monophosphate (PENTA).

  Nanofillers-Amorphous Silicon Dioxide.

  Photoinitiators.

  Stabilizer.

  Cetylamine hydrofluoride.

  Acetone.

Figure 4.3: Prime and Bond NT (two-step dentin bonding agent).

4.7 Procedure:

One hundred non carious freshly extracted molar teeth were stored in saline solution with 0.2% thymol to prevent any bacterial infection during storage at room temperature (Goracci, *et al*, 2004). Samples were prepared as follows. The teeth were cleaned using a sharp knife to remove any calculus or soft tissue debris from the tooth surface. The roots of the teeth were sectioned with a separating disc at the crown-root junction and the teeth were embedded in a chemically-cured acrylic resin so that the buccal surfaces projected just above the acrylic resin (figure 4.4) (Hasegawa, *et al.*1995). A portion of tooth 2.5mm in dimension was cut off from the buccal surface of the teeth using a water-cooled diamond disk-cutter at slow speed (Minitom, Struers, Denmark) (Figure 4.5) to expose a flat dentin surface (figure 4.6) (Miyazaki, *et* al.1998). The flat buccal surface was then finished using a universal polisher with a 1200 grit silicon finishing paper (figure 4.7) (Retief, 1991).

Figure 4.4: Embedded teeth in cold-cure acrylic resin.

Figure 4.5: Water-cooled diamond disk-cutter (Minitom, Struers, Denmark).

Figure 4.6: Exposed flat dentin surface 2.5mm depth from the buccal surface.

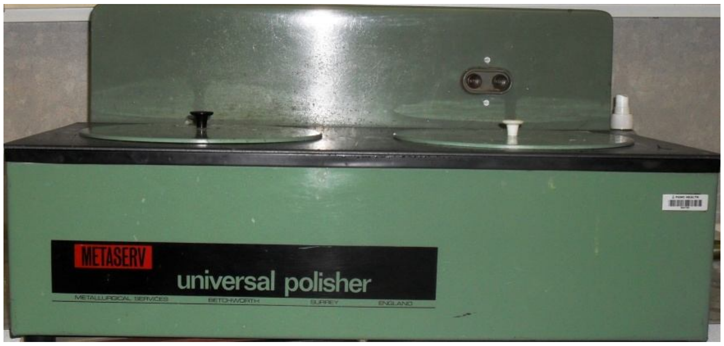


Figure 4.7: Universal polisher with 1200 grit silicon finishing paper.

Each tooth was labeled with a different number and the teeth were randomly divided into two groups (n= 50). In the first group Prime and Bond NT a two-step dentin bonding agent was used to bond the composite mold to the flat dentin surface while in the second group Xeno V a single-step dentin bonding agent was used to bond the composite mold to the flat dentin surface. Random division of the specimens provided each tooth an equal chance to fall into any group.

- **Prime and Bond NT (Two-step dentin bonding agent):**

In the first group, Prime and Bond NT (two step dentin bonding system) was applied to the flat dentin surface as follows:

➢ ***Acid conditioning (total etch technique):***

The flat dentin surface was etched with Caulk 34% a tooth conditioner gel (34% phosphoric acid) for 15 seconds. The etchant gel was removed by rinsing the flat dentin surface with water using a dental syringe for 10 seconds until all the etchant gel was removed. The etched dentin surface was then blotted using a moist cotton pellet without rubbing until there was no pooling of water.

➢ ***Application of prime and bond:***

A single layer of Prime and Bond NT was applied to the flat etched dentin surface using an application tip for 20 seconds in a light brushing motion as recommended by the manufacturer. The Prime and Bond NT was air thinned for 5 seconds and then light-cured for 10 seconds using a halogen light curing unit (Demetron LC, sdsKerr, USA).

➢ ***Application of TPH ®3 Spectrum composite resin:***

A Teflon mold, 2mm high and 4 mm in diameter was used to form and contain the composite restorative material against the tooth surface. TPH®3 Spectrum Composite resin was condensed through the Teflon mold onto the prepared flat dentin surface immediately after the application and curing of the Prime and Bond NT. The TPH®3 Spectrum Composite resin was then light-cured with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 20 seconds following the manufacturer’s instructions (figure 4-9 and 4-10).

The teeth were then subdivided randomly into another two groups of 25 each (n_1=25). Random division provided each tooth an equal chance to fall into any group. The number of the teeth in each group was recorded in an excel spreadsheet.

In the first group the samples were stored in water at 37^{0}C for 24 hours (Holderegger et al, 2008) and the group was labeled as the Prime and Bond NT (two-step) without thermal-cycling group.

In the second group the samples were thermal-cycled between 5^{0}C and 55^{0}C for 500 cycles. Following thermal-cycling the samples were stored in water at 37^{0}C for 24 hours (Holderegger *et al*, 2008) and the group was labeled as the Prime and Bond NT (two-step) with thermal-cycling group.

- **Xeno V (single-step dentin bonding agent):**

In this group, Xeno V (a single-step dentin bonding agent) was applied to the flat dentin surface as follows:

- ***Application of Xeno® V:***

Two layers of Xeno® V (a single-step dentin bonding agent) were applied to the prepared flat dentin surface following the manufacturer's instructions. The flat dentin surface was cleaned with a water spray and air dried lightly without desiccating the dentin using a dental syringe. Xeno® V was applied with a disposable microbrush applicator tip twice onto the dentin surface generously. The two layers of Xeno® V were gently agitated for 20 seconds. The excess solvent was removed gently by drying with clean, dry air from a dental syringe for 5 seconds. The Xeno® V adhesive system was then light-cured for 20 seconds using a halogen light curing unit (Demetron LC, sdsKerr, USA).

- ***Application of TPH ®3 Spectrum composite resin:***

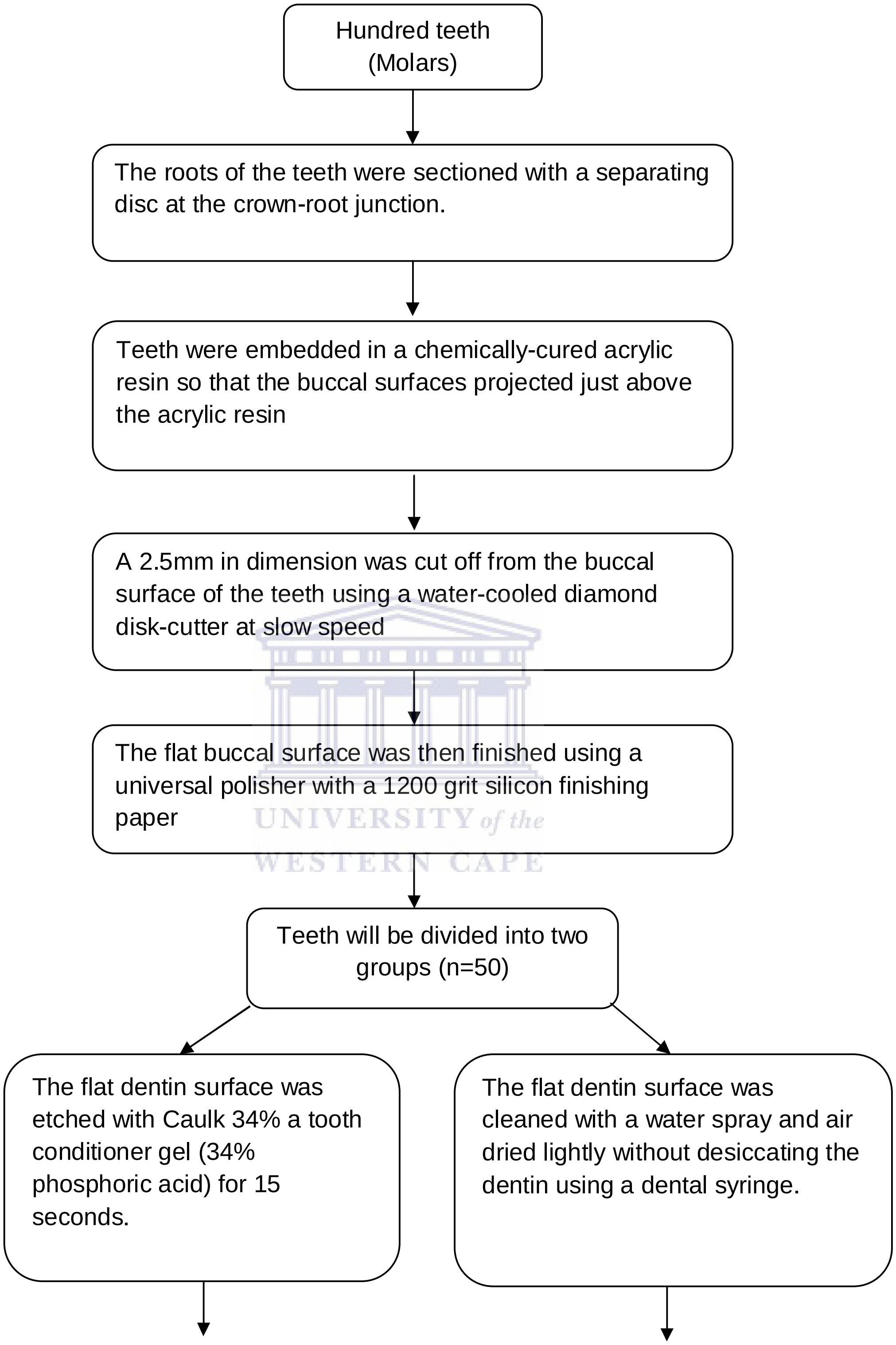
A Teflon mold, 2mm high and 4 mm in diameter was used to form and contain the composite restorative material against the tooth surface. TPH®3 Spectrum Composite resin was condensed through the Teflon mold onto the prepared flat dentin surface immediately after the application and curing of the Xeno® V. The

TPH®3 Spectrum Composite resin was then light-cured with a halogen light curing unit (Demetron LC, sdsKerr, USA) for 20 seconds following the manufacturer’s instructions (figure 4-9 and 4-10).

The teeth were then subdivided randomly into another two groups of 25 each (n1=25). Random division provided each tooth an equal chance to fall into any group. The number of the teeth in each group was recorded in an excel spreadsheet.

In the first group the samples were stored in water at 37^0C for 24 hours (Holderegger et al, 2008) and the group was labeled as the Xeno® V (single-step) without thermal-cycling group.

In the second group the samples were thermal-cycled between 5^0C and 55^0C for 500 times. Following thermal-cycling the samples were stored in water at 37^0C for 24 hours (Holderegger et al, 2008) and the group was labeled as the Xeno® V (single-step) with thermal-cycling group. The method of the study is graphically illustrated in figure 4-8.



Hundred teeth (Molars)

The roots of the teeth were sectioned with a separating disc at the crown-root junction.

Teeth were embedded in a chemically-cured acrylic resin so that the buccal surfaces projected just above the acrylic resin

A 2.5mm in dimension was cut off from the buccal surface of the teeth using a water-cooled diamond disk-cutter at slow speed

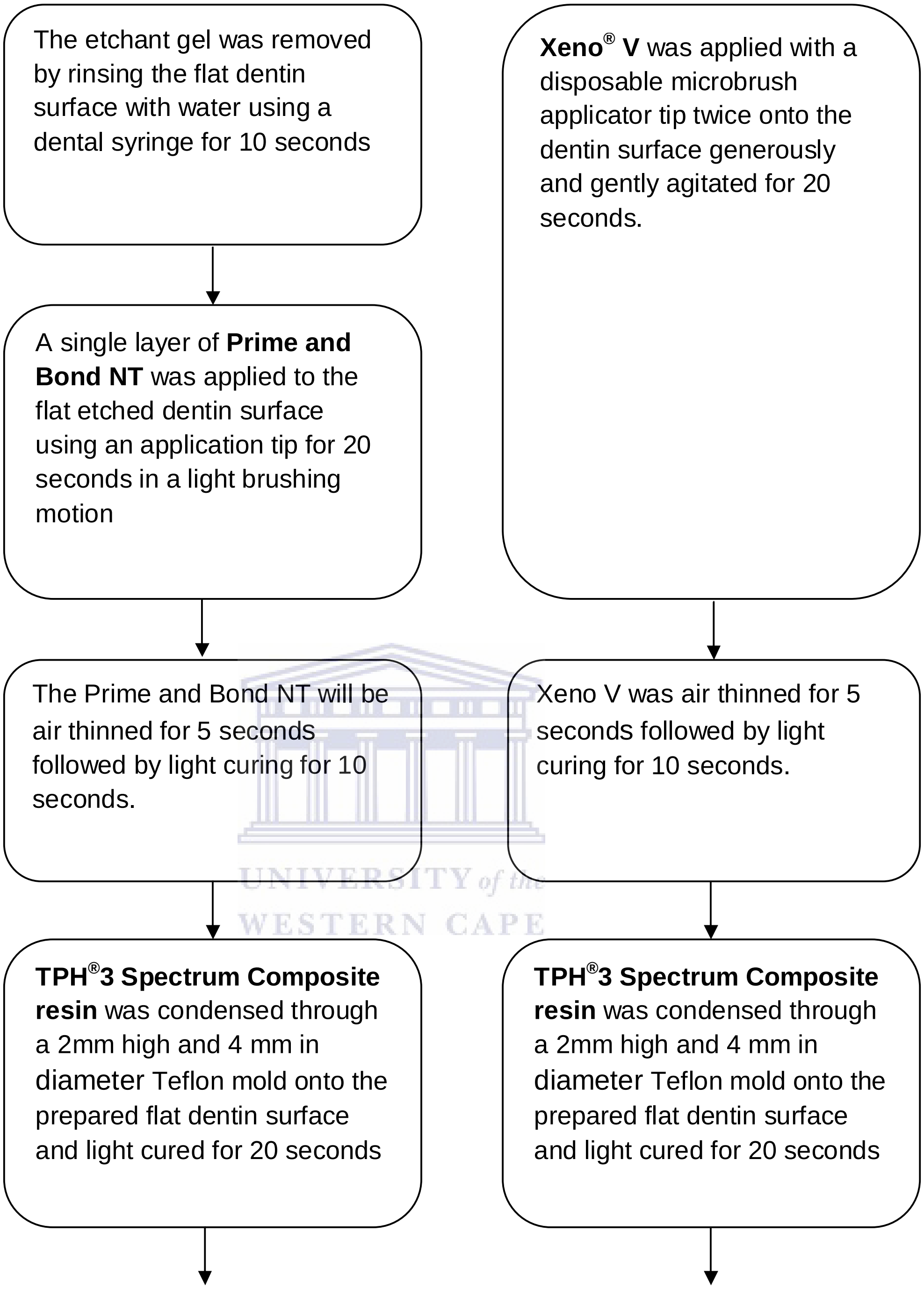
The flat buccal surface was then finished using a universal polisher with a 1200 grit silicon finishing paper

Teeth will be divided into two groups (n=50)

The flat dentin surface was etched with Caulk 34% a tooth conditioner gel (34% phosphoric acid) for 15 seconds.

The flat dentin surface was cleaned with a water spray and air dried lightly without desiccating the dentin using a dental syringe.

Figure 4.8: Flow chart depicting the study.



The etchant gel was removed by rinsing the flat dentin surface with water using a dental syringe for 10 seconds

A single layer of **Prime and Bond NT** was applied to the flat etched dentin surface using an application tip for 20 seconds in a light brushing motion

The Prime and Bond NT will be air thinned for 5 seconds followed by light curing for 10 seconds.

TPH$^{®}$3 Spectrum Composite resin was condensed through a 2mm high and 4 mm in diameter Teflon mold onto the prepared flat dentin surface and light cured for 20 seconds

Xeno$^{®}$ V was applied with a disposable microbrush applicator tip twice onto the dentin surface generously and gently agitated for 20 seconds.

Xeno V was air thinned for 5 seconds followed by light curing for 10 seconds.

TPH$^{®}$3 Spectrum Composite resin was condensed through a 2mm high and 4 mm in diameter Teflon mold onto the prepared flat dentin surface and light cured for 20 seconds

Figure 4.8 (continued): Flow chart depicting the study.

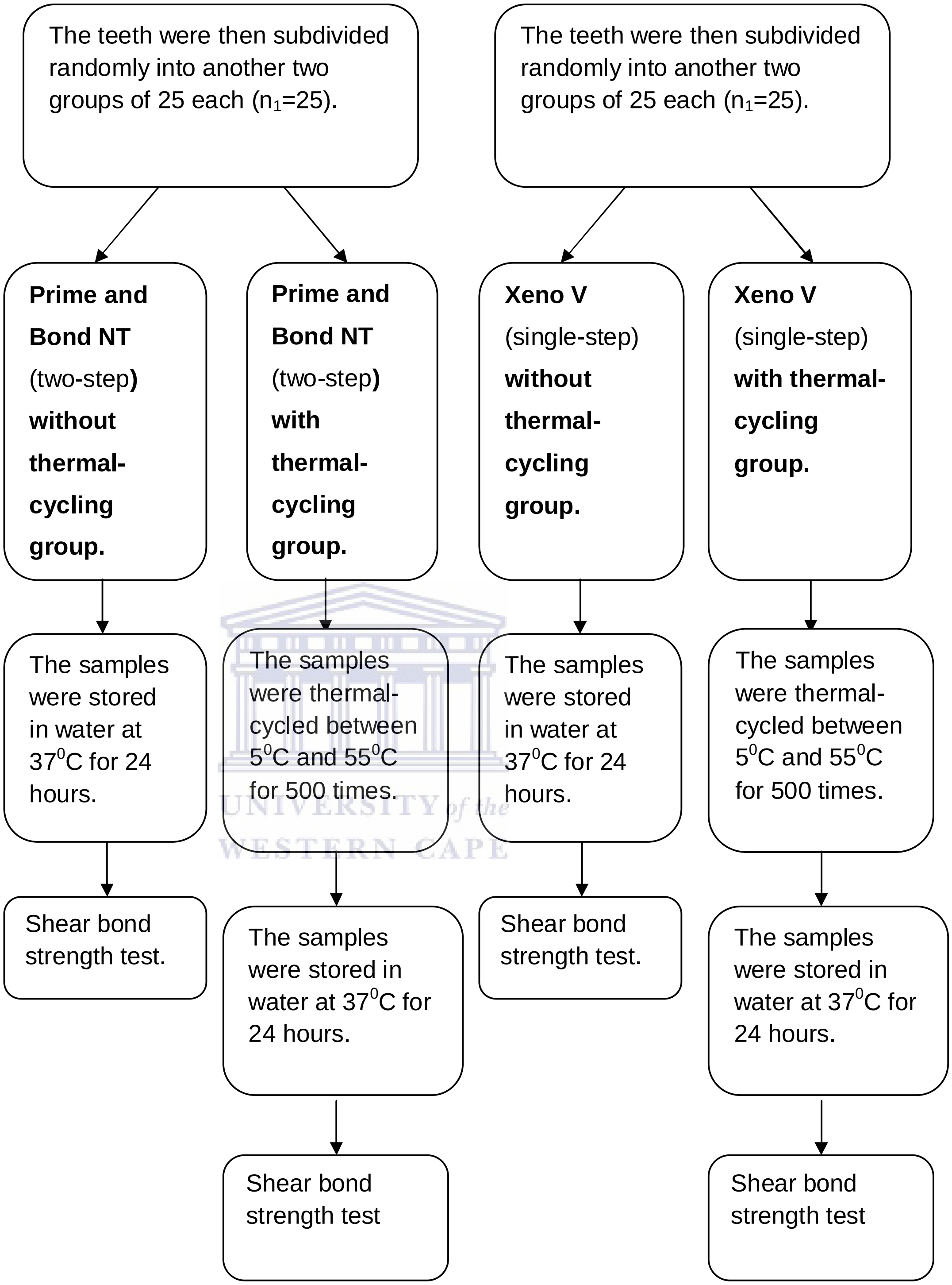


Figure 4.8 (continued): Flow chart depicting the study.

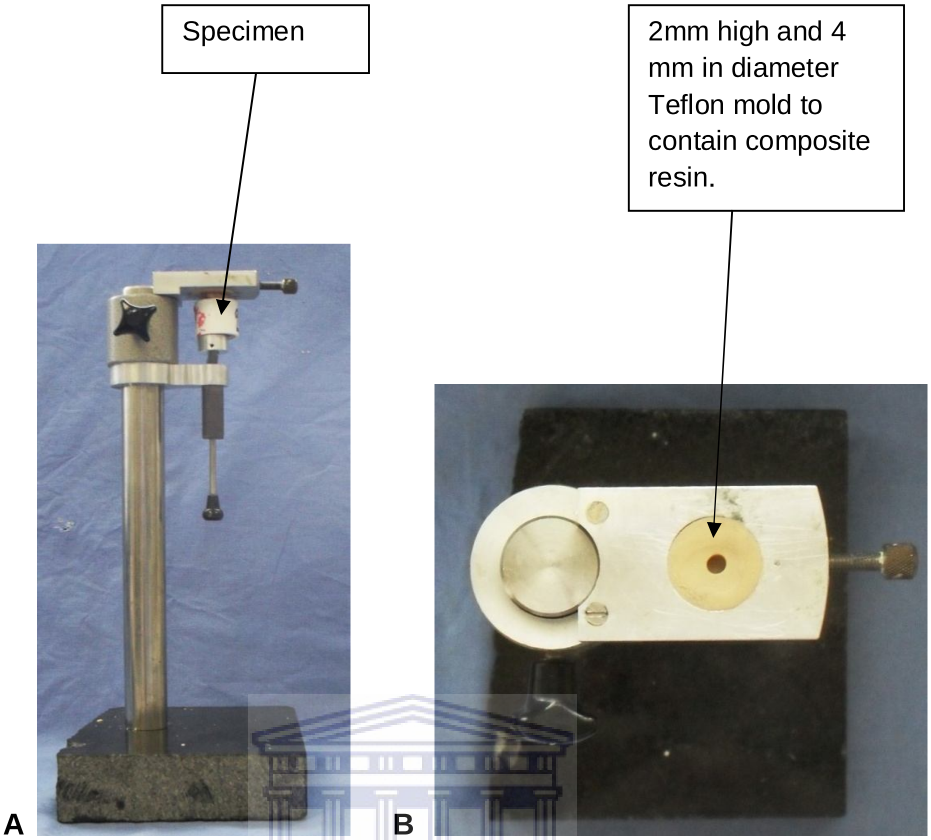


Figure 4.9: (A) Assembly of specimen and Teflon mold containing composite resin; (B) Magnified view of the Teflon mold to retain composite resin.

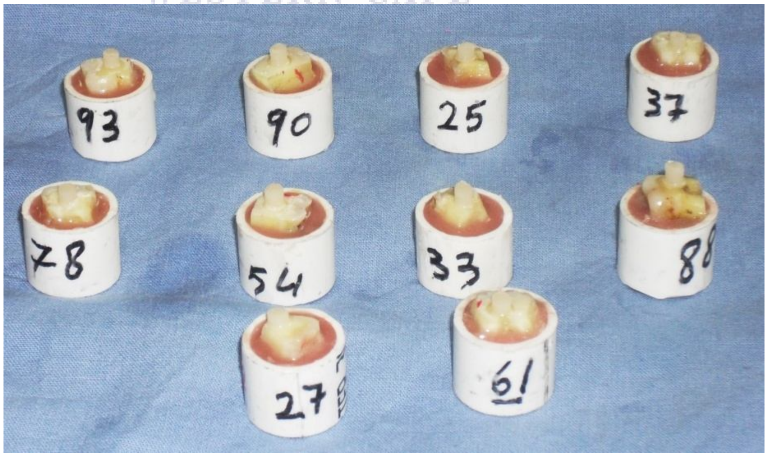


Figure 4.10: Composite mold bonded to prepared specimens after releasing from assembly.

All samples were arranged haphazardly before measuring the shear bond strength so that the examiner did not know which sample belonged to which group. The shear bond strength values were measured for each sample from each group using an Instron (Zwick 1644) testing machine. The machine was adjusted so that the force was applied to the base of the composite-tooth interface at a crosshead speed of 0.5 mm/minute with a knife-edged rod 0.5mm thick (figure 4.11). The shear bond strength values were calculated and expressed in megapascals (MPa). Any value less than 2 MPa was not recorded by the testing machine and the sample was discarded.

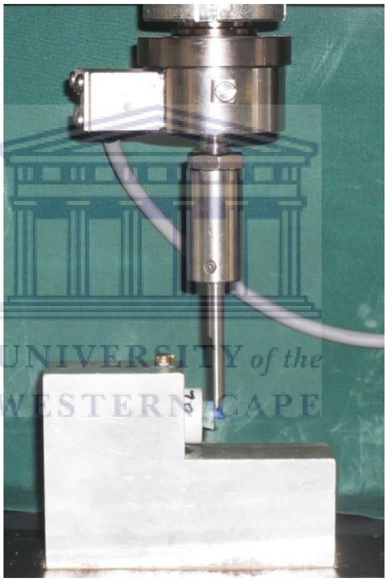


Figure 4.11: Shear load applied to the composite-tooth interface with Instron (Zwick 1644).

Chapter 5
Data analysis

All results for each group were transfered to an Excel spreadsheet (Microsoft Corporation, USA) for further analysis (Appendix 1,). The data of each group was tested for their distribution as regards normality. All groups showed an abnormal distribution of results. A Mann-Whitney U test (non-parametric test for independent values) was then used to determine a statistically significant difference, if any, between the bond strength values of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent) without thermal-cycling, between Prime and Bond NT (two-step dentin bonding agent) before and after thermal-cycling, between Xeno V (single-step dentin bonding agent) before and after thermal-cycling, and between Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single step-dentin bonding agent) after thermal-cycling. These variables are summarized in table 5.1. Only P-values less than 0.05 were regarded as statistically significant differences. All statistical analysis were carried out using SPSS 14.0 for windows (SPSS©, Inc. Chicago, IL, USA) and Microsoft Excel 2007 (Microsoft Corporation, USA).

Table 5.1: Summery of variables being compared in the study.

Prime and Bond NT (two-step) without thermal-cycling.	Vs	Xeno V (single-step) without thermal-cycling.
Prime and Bond NT (two-step) Without thermal-cycling.	Vs	Prime and Bond NT (two-step) with thermal-cycling.
Xeno V (single-step) without thermal-cycling.	Vs	Xeno V (single-step) with thermal-cycling.
Prime and Bond NT (two-step) with thermal-cycling.	Vs	Xeno V (single-step) with thermal-cycling.

Chapter 6
Results

The raw data of the study for all bond strength values appear in (Appendix 1, 2, 3, and 4). All values were expressed as a median (Interquartile range).

6.1 Bond strength values of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent):

Following removal of any bond strength value less than 2 MPa, the median and interquartile range of the remaining 24 samples in the Prime and Bond NT without thermal-cycling group was compared to the median of the samples of the Xeno V without thermal-cycling using a Mann-Whitney U-test (non-parametric test for independent samples) to determine if there is any statistically significant difference between the two groups (table 6.1). A P-value of 0.810 indicates that; there is no statistically significant difference in the bond strength of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent).

Table 6.1: Comparison between the bond strength values of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent) without thermal-cycling (Values presented as median (Interquartile range):

	Percentile 25	Percentile 75	Median	Interquartile range	P-value
Prime and Bond NT (two-step) without thermal-cycling	5	7	6.14	3.08	0.810
Xeno V (single-step) without thermal-cycling	3.72	9.12	6.13	6.06	

The distribution of the bond strength values of each group is graphically illustrated in figure 6.1. It is evident from figure 6.1 and appendix 1 and 2 that the bond strength values of Prime and Bond NT without thermal-cycling ranged from 2.29 to 10.43 MPa with only one value less than 2 MPa. On the other hand the bond strength values of Xeno V without thermal-cycling ranged from 2.33 to 17.44 MPa with no value less than 2 MPa.

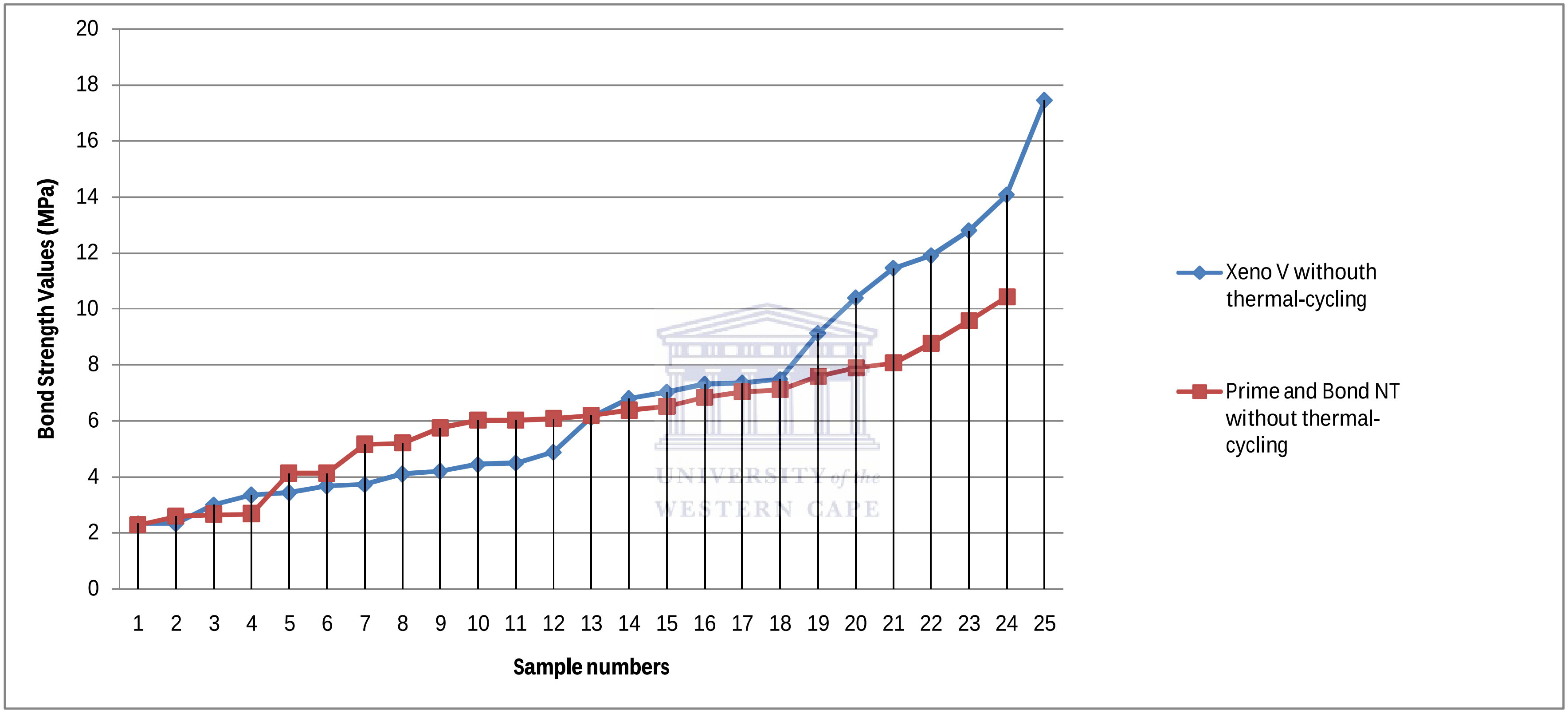


Figure 6.1: Distribution of bond strength values of Prime and Bond NT two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent).

6.2 Effect of thermal-cycling on the bond strength values of Prime and Bond NT (two-step dentin bonding agent):

Following removal of any bond strength value less than 2 MPa, the median and interquartile range of the remaining 24 samples in the Prime and Bond NT without thermal-cycling group was recorded and compared to the median of the 22 samples in the Prime and Bond NT with thermal-cycling group using a Mann-Whitney U test (non-parametric test for independent samples) to determine if there is any statistically significant difference between the two groups table 6.2. A P-value of 0.692 indicates that; there is no statistically significant difference in the bond strength of Prime and Bond NT before and after thermal-cycling between 5^0C and 55^0C for 500 times.

Table 6.2: Comparison between the bond strength values of Prime and Bond NT (two-step dentin bonding agent) with and without thermal-cycling (Values presented as median (Interquartile range):

	Percentile 25	Percentile 75	Median	Interquartile range	P-value
Prime and Bond NT (two-step) without thermal-cycling	5	7	6.14	3.08	0.692
Prime and Bond NT (two-step) with thermal-cycling	4	7	5.60	3.13	

The distributions of the bond strength values for each group are graphically illustrated in figure 6.2. It is evident from figure 6.2 and appendix 1 and 3 that the bond strength values of Prime and Bond NT without thermal-cycling ranged from 2.29 to 10.43 MPa with only one value less than 2 MPa. On the other hand the bond strength values of Prime and Bond NT with thermal-cycling ranged from 2.35 to 14.98 MPa with three values less than 2 MPa.

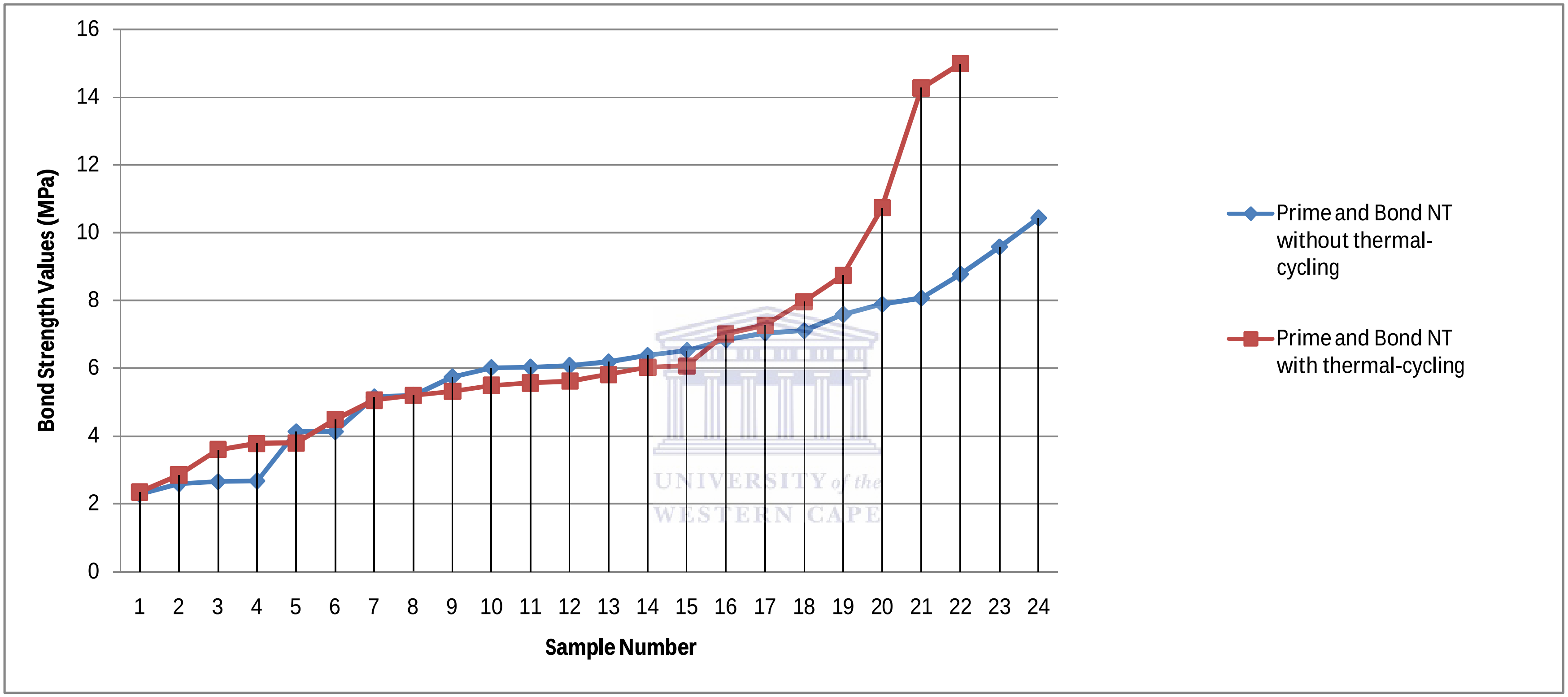


Figure 6.2: Distribution of bond strength values of Prime and Bond NT (two-step dentin bonding agent) with and without thermal-cycling.

6.3 Effect of thermal-cycling on the bond strength value of Xeno V (single-step dentin bonding agent):

All the bond strength values for the two groups using the single-step dentin bonding agent were above 2 MPa, so no sample was removed. The median and interqurtile range of the Xeno V without thermal-cycling group was compared to the Xeno V with thermal-cycling group using a Mann-Whitney U test (non-parametric test for independent samples) to determine if there was any statistically significant difference between the two groups table 6.3. The P-value was 0.861 indicating that there was no statistically significant difference in the bond strength of Xeno V before and after thermal-cycling between 5^0C and 55^0C for 500 times.

Table 6.3: Comparison between the bond strength values of Xeno V (single-step dentin bonding agent) with and without thermal-cycling (Values are presented as median (Interquartile range):

	Percentile 25	Percentile 75	Median	Interquartile range	P-value
Xeno V (single-step) without thermal-cycling	3.72	9.12	6.13	6.06	0.861
Xeno V (single-step) with thermal-cycling	4.01	7.11	5.83	3.28	

The distribution of the bond strength values of the two groups is graphically illustrated in figure 6.3. It is evident from figure 6.3 and appendix 2 and 4 that the bond strength values of Xeno V without thermal-cycling ranged from 2.33 to 17.44 MPa with no value less than 2 MPa. On the other hand the bond strength values of Xeno V after thermal-cycling ranged from 2.25 to 16.14 MPa with no value less than 2 MPa.

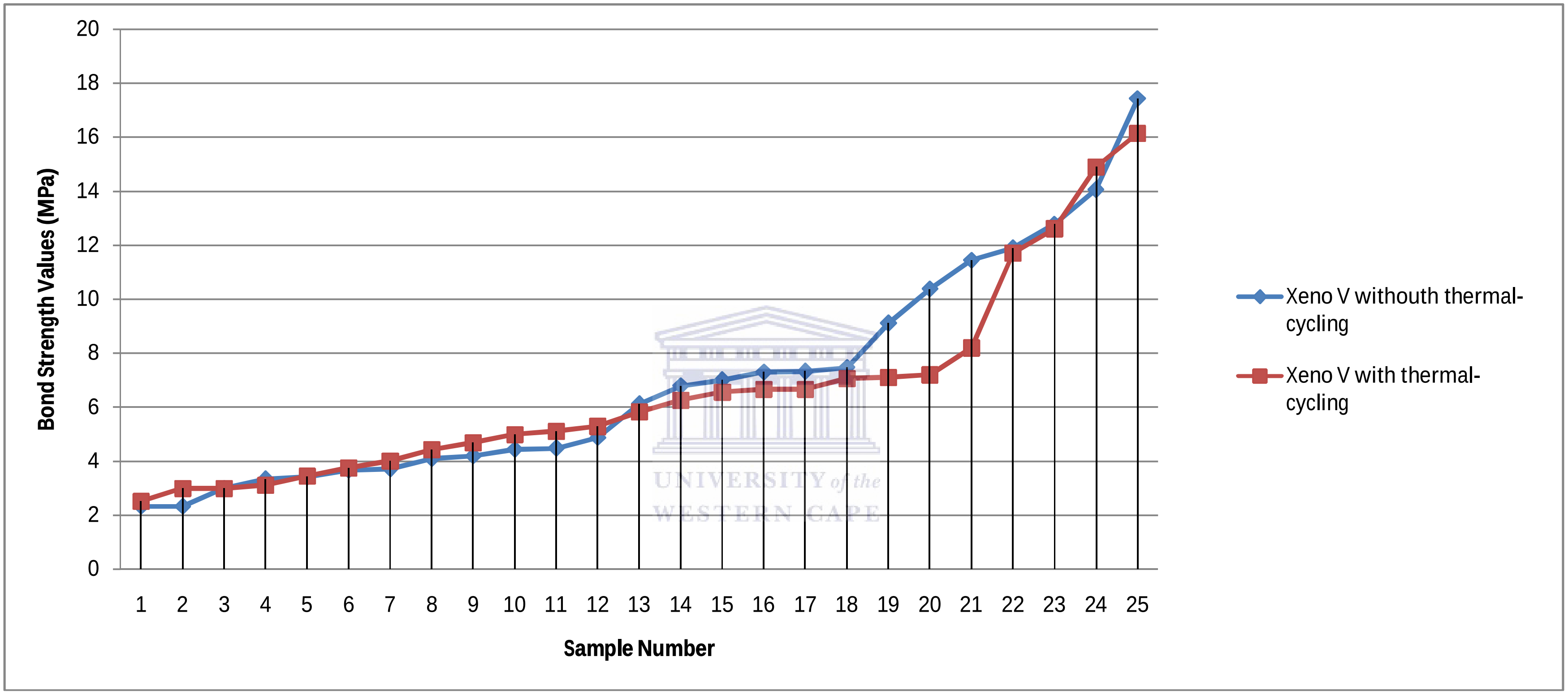


Figure 6.3: Distribution of bond strength values of Xeno V (single-step dentin bonding agent) with and without thermal-cycling.

6.4. Effect of thermal-cycling on bond strength value of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent):

Following removal of any bond strength value less than 2 MPa, the median and interquartile range of the remaining 22 samples of the Prime and Bond NT with thermal-cycling group was recorded and compared to the median of all samples of the Xeno V with thermal-cycling group using a Mann-Whitney U test (non-parametric test for independent samples) to determine if there was any statistically significant difference between the two groups table 6.4. The P-value was 0.932 indicating that there was no statistically significant difference in the bond strength of Prime and Bond NT with thermal-cycling and Xeno V with thermal-cycling.

Table 6.4: Comparison between the bond strength values of Prime and Bond NT (two-step dentin bonding agent) with thermal-cycling and Xeno V (single-step dentin bonding agent) with thermal-cycling (Values are presented as median (Interquartile range):

	Percentile 25	Percentile 75	Median	Interquartile range	P-value
Prime and Bond NT (two-step) with thermal-cycling	4	7	5.60	3.13	0.932
Xeno V (single-step) with thermal-cycling	4.01	7.11	5.83	3.28	

The distribution of the bond strength values of the two groups is graphically illustrated in figure 6.4. It is evident from figure 6.4 and appendix 3 and 4 that the bond strength values of Prime and Bond NT with thermal-cycling ranged from 2.35 to 14.98 MPa with three values less than 2 MPa. On the other hand the bond strength values of Xeno V with thermal-cycling ranged from 2.25 to 16.14 MPa with no value less than 2 MPa.

All the results are summarized in table 6.5

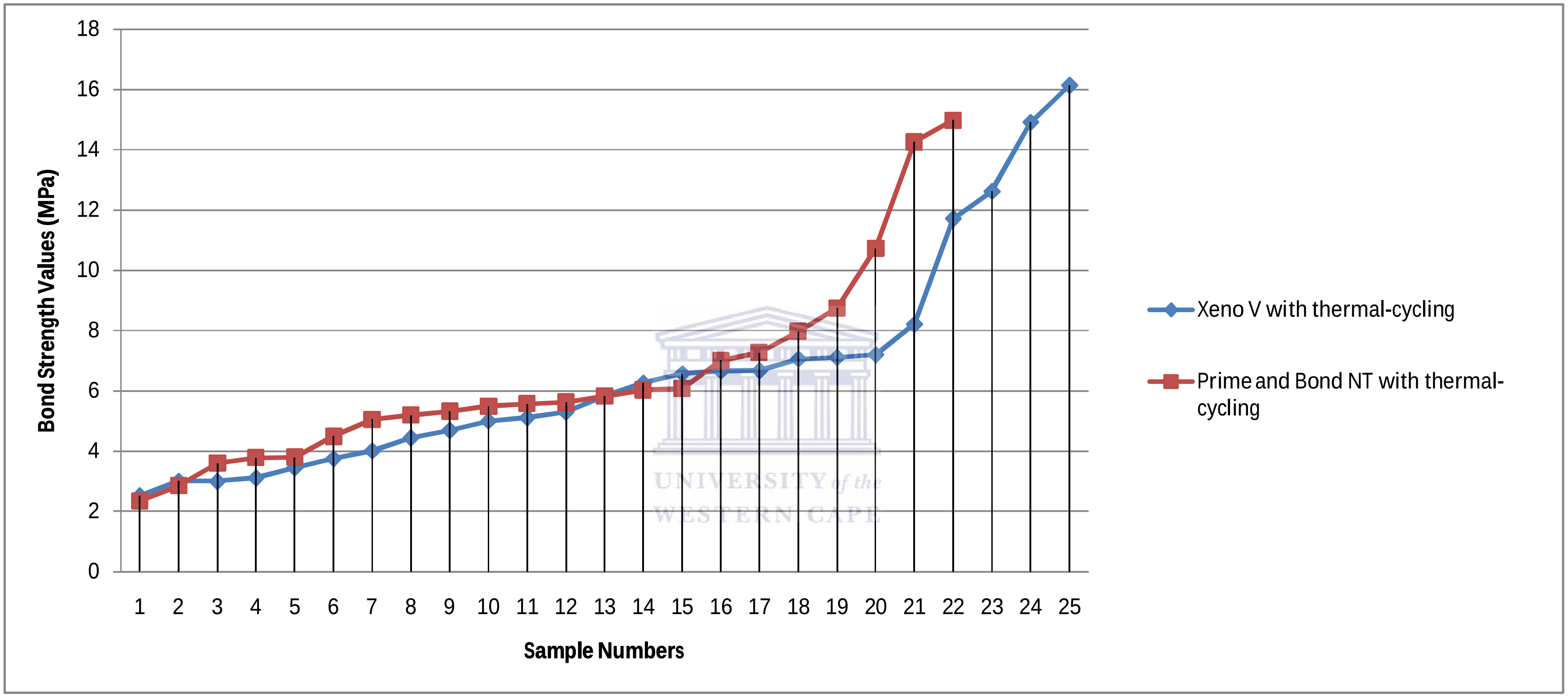


Figure 6.4: Distribution of bond strength values of Prime and Bond NT (two-step dentin bonding agent) with thermal-cycling and Xeno V (single-step dentin bonding agent) with thermal-cycling.

Table 6.5: Summery of the results.

	Table number	P-value
Bond strength values of Prime and Bond NT (two-step) without thermal-cycling Vs Bond strength values of Xeno V (single-step) without thermal-cycling	6-1	0.810
Bond strength values of Prime and Bond NT (two-step) without thermal-cycling Vs Bond strength values of Prime and Bond NT (two-step) with thermal-cycling	6-1	0.692
Bond strength values of Xeno V (single-step) without thermal-cycling Vs Bond strength values of Xeno V (single-step) with therml-cycling	6-3	0.861
Bond strength values of Prime and Bond NT (two-step) with thermal-cycling Vs Bond strength values of Xeno V (single-step) with thermal-cycling	6-4	0.932

Chapter 7
Discussion

7.1 Introduction:

In vitro bond strength tests were introduced to evaluate the clinical performance of different adhesive systems currently used (Retief, 1991). There is a great diversity in the techniques used for these types of tests (Van Meerbeek *et al*, 2003). The bond strength can be evaluated either by tensile bond strength test, shear bond strength test (Retief, 1991), microtensile bond strength test (Van Meerbeek *et al*, 2003), and most recently microshear bond strength test (Placido *et al*, 2007). However the restorative material and the teeth are subjected to a complex type of forces intraorally which is difficult to be simulated in *in vitro* studies.

In addition thermal-cycling is one of the *in vitro* methods that are used for the evaluation of the bond strength values of the adhesive systems following repeated thermal change (De Munk *et al*, 2005).

In the present study the bond strength values of a two-step dentin bonding agent and a single-step dentin bonding agent were evaluated. The effect of thermal-cycling on these bond strength values was also evaluated. A shear bond strength test was used to measure the bond strength values of these adhesive systems due to its simplicity and good predictive result.

7.2 Discussion of the methodology:

Extracted human teeth have the potential for multiple uncontrollable variations when used for *in vitro* studies. In the present study, the samples were standardized as much as possible, regarding the tooth type, the depth of exposed dentin, preparation technique of the dentin surface, and the diameter of the bonded area.

The selected teeth were human molar teeth due to their large surface area. The buccal surfaces were used rather than the occlusal surface so that sufficient distance from the pulp and the pulp horn can be obtained. A 2.5 mm section was cut off from the buccal surface so that an even depth of exposed dentin was obtained for the entire sample. The finishing procedure for the dentin surfaces were the same for all the samples in that they were all finished using a universal polisher with a 1200 grit silicon finishing paper. A Teflon mold of 4 mm diameter was used to control the diameter of the composite resin-adhesive-dentin complex bonded area in all the samples. The Teflon mold that was used to maintain the composite resin during condensation was of a 2 mm thickness. This thickness was selected to ensure that complete curing for composite resin was obtained. All the samples were stored in the same storage media for an equal time.

The number of thermal-cycles was selected according to the ISO TR 11450 standard (1994).

7.3 Discussion of the results:

7.3.1 Bond strength values of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agnet):

One of the aims of this study was to test and compare the bond strength values of a two-step dentin bonding agent and a single-step dentin bonding agent. The adhesive systems used in this study were Prime and Bond NT (two-step) and Xeno V (single-step).

Figure 6-1 and appendix1 and 2 reflect the shear bond strength values of each adhesive system. The bond strength values are represented as mega pascal (MPa). It is clear from figure 6-1 and appendix 1 and 2 that the bond strength values of Prime and Bond NT (two-step) were ranged from 2.29 to 10.43 with only one value that was less than 2 MPa. On the other hand the bond strength values of Xeno V (single-step) ranged from 2.33 to 17.44. The median of the Prime and Bond NT (two-step) without thermal-cycling group was 6.14 and the median of the Xeno V (single-step) without thermal-cycling group was 6.13. It is clear that the range between the two medians was very close. The P-value was

0.810. Statistical analysis showed no statistically significant difference in the bond strength values between Prime and Bond NT (two-step) and Xeno V (single-step).

This means that Prime and Bond NT (two-step) and Xeno V (single-step) used in this study had the same bonding performance to dentin, although they are different in their chemical composition and in the number of steps involved in the bonding process. Prime and Bond NT (two-step) uses 34% phosphoric acid for dentin etching, while Xeno V (single-step) uses a weak acid such as acrylic acid.

The results of this study differed from those obtained by Hegde and Bhandary (2008). They showed that Prime and Bond NT (two-step) had a higher shear bond strength value compared to a single step dentin bonding agent (Xeno III) they used. However Xeno V may be an improvement on the Xeno III used in this study and this may explain the difference in the results obtained.

The difference in composition between the Xeno III and Xeno V is largely in the chemical composition. Xeno III consisted of two liquids in which the first liquid contained 2-hydroxyethyl methacrylate (HEMA), purified water, ethanolurethane dimethacrylate resin, butylated hydroxy toluene (BHT), and highly dispersed silicon dioxide, while the second liquid contained phosphoric acid modified polymethacrylate resin, mono fluoro phosphazene modified methacrylate resin, urethane dimethacrylate resin, butylated hydroxyl toluene (BHT), and camphorquinone, ethyl-4-dimethylaminobenzoate. On the other hand Xeno V (single-step) consists of one liquid that contains bifunctional acrylic, acidic acrylate, functionalized phosphoric acid ester, acrylic acid, water, tertiary butanol, initiator, and stabilizer.

El Araby and Talic (2007) also showed different results from the results of the present study. They showed that Xeno III acheived high bond strength values to dentin when compared to the bond strength of Prime and Bond NT used in the same study.

7.3.2 Effect of thermal-cycling on the bond strength values of Prime and Bond NT (two-step dentin bonding agent) and Xeno V (single-step dentin bonding agent):

The other aim of this study was to evaluate the effect of thermal-cycling on the bond strength values of Prime and Bond NT (two-step) and Xeno V (single-step) dentin bonding agents.

- **Prime and Bond NT (two-step):**

Appendix 1 and 3 and figure 6-2 represent the shear bond strength values and their distribution for the Prime and Bond NT group (without thermal-cycling and with thermal-cycling groups). The highest and lowest bond strength values for Prime and Bond NT without thermal-cycling group were 10.43 and 2.29 respectively with only one value less than 2 MPa, while for Prime and Bond NT with thermal-cycling group the highest and lowest bond strength values were 14.98 and 2.35 respectively with 3 bond strength values less than 2 MPa. The median was 6.14 for the Prime and Bond NT without thermal-cycling group and 5.60 for Prime and Bond NT with thermal-cycling group. The P-value was 0.692. Statistical analysis showed no statistically significant difference in the shear bond strength value of Prime and Bond NT with and without thermal-cycling.

The results of this study indicated that, the shear bond strength value of Prime and Bond NT did not changed after the specimen were subjected to thermal-cycling.

This result was similar to other results obtained by Santos *et al*, (2005). However El Araby and Talic, (2007) showed that the bond strength value of Prime and Bond NT decreased after thermal-cycling. Moreover Miyazaki *et al*, (1998) also showed a significant decrease in the bond strength value of another two-step dentin bonding agent following thermal-cycling.

- **Xeno V (single-step):**

Appendix 2 and 4 and figure 6-3 represent the shear bond strength values and their distribution for the Xeno V groups (without thermal-cycling and with thermal-cycling groups). The highest and lowest bond strength values for Xeno V without thermal-cycling group were 17.44 and 2.33 respectively with no value less than 2 MPa, while for Xeno V with thermal-cycling group the highest and lowest bond strength values were 16.14 and 2.52 respectively with no value less than 2 MPa. The median was 6.13 for the Xeno V without thermal-cycling group and 5.83 for Xeno V with thermal-cycling group. The P-value was 0.861. Statistical analysis showed no statistically significant difference in the shear bond strength value of Xeno V with and without thermal-cycling.

In the present study there was no statistically significant difference in the shear bond strength value of Xeno V with and without thermal-cycling indicated that thermal-cycling had no influence in the bond strength value of Xeno V.

Naughton and Latta, (2005) also showed similar results to the results of the present study although they used other single-step dentin bonding agents (Clearfil SE Bond, Optibond Solo Plus, Xeno III, Tyrian SPE, and Adper Prompt L-Pop). However El Araby and Talic, (2007) showed that the bond strength value of other single-step dentin bonding agents (Xeno III (XE3) and Prompt L-Pop) decreased after thermal-cycling.

7.4 Mode of Failure:

In trying to understand the failure pattern of the bonded interface it was decided to study the fractured surface. Because examination of the mode of failure of the adhesive bond was not one of the aims of this study, only four samples were selected from each group. The selected samples were the two samples that exhibited the highest and the lowest shear bond strength values. The mode of bond failure was investigated under a conventional microscope with 300 times magnification by two investigators.

➢ *Prime and Bond NT (two-step):*

The modes of bond failure for the Prime and Bond NT with and without thermal-cycling groups were as follows

Table 7.1: Description for the mode of failure of selected samples from the Prime and Bond NT groups (with and without thermal-cycling):

	Prime and Bond NT without thermal-cycling	**Prime and Bond NT with thermal-cycling**
Sample 1: (high value)	• 95% from the total bonded area break between the dentin and the adhesive system. • 5% from the total bonded area break between the composite and the adhesive system.	• 55% from the total bonded area break between the dentin and the adhesive system. • 45% from the total bonded area break through the composite.
Sample 2: (high value)	• 90% from the total bonded area break between the dentin and the adhesive system. • 10% from the total bonded area break between the composite and the adhesive system.	• 45% from the total bonded area break between the dentin and the adhesive system. • 20% from the total bonded area break between the composite and the adhesive system. • 35% from the total bonded area break through the composite.
Sample 3: (low value)	• 90% from the total bonded area from the total bonded area break between dentin and adhesive system. • 10% from the total bonded area break between the composite and the adhesive system.	• 75% from the total bonded area break between the dentin and the adhesive system. • 25% from the total bonded area break between the composite and the adhesive system.
Sample 4: (low value)	• 75% from the total bonded area break between the dentin and the adhesive system. • 25% from the total bonded area break between the composite and the adhesive system.	• 75% from the total bonded area break between the dentin and the adhesive system. • 25% from the total bonded area break between the composite and the adhesive system.

It can be suggested from table 7.1 that most of the bond failure in both Prime and Bond NT groups (with and without thermal-cycling) was between the dentin and the adhesive system. An example of this mode of failure is shown in figure 7.1. However this result cannot be conclusive because of the small number of the selected samples from each group subjected to this examination.

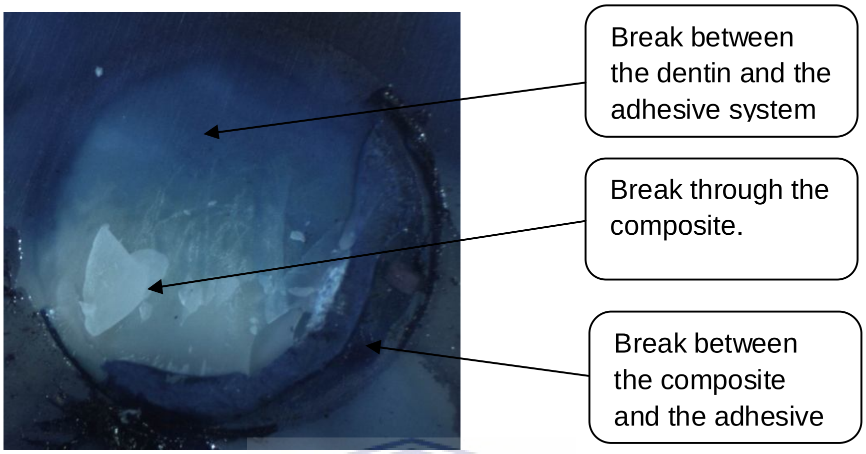


Figure 7.1: Mode of failure of Prime and Bond NT (two-step) shows 45% break between the dentin and the adhesive system, 20% break between the composite and the adhesive system, and 35% break through the composite.

- ***Xeno V (single-step):***

The modes of bond failure for the two Xeno V groups (with and without thermal-cycling) were as follows:

Table 7.2: Description for the mode of failure of selected samples from the Xeno V groups (with and without thermal-cycling):

	Xeno V without thermal-cycling	**Xeno V with thermal-cycling**
Sample 1: (high value)	• 60% break through the dentin. • 30% break through the composite. • 10% break between the composite and the adhesive system.	• 80% break between the composite and the adhesive system. • 20% break between the dentin and the adhesive system.
Sample 2: (high value)	• 90% break through the dentin. • 10% break through the composite.	• 80% break between the composite and the adhesive system. • 20% break between the dentin and the adhesive system.
Sample 3: (low value)	• 60% break between dentin and adhesive system. • 40% break between the composite and the adhesive system.	• 100% break between the dentin and the adhesive system.
Sample 4: (low value)	• 100% break between the composite and the adhesive system.	• 100% break between the dentin and the adhesive system.

It can be suggested from table 7.2 that Xeno V (single-step) can produce different patterns of bond failure. An example of this mode of failure is shown in figure 7.2, and 7.3. However this result cannot be conclusive because of the small number of the selected samples from each group subjected to this investigation, further investigation is needed in this area to explore the mode of failure of the bonded interface.

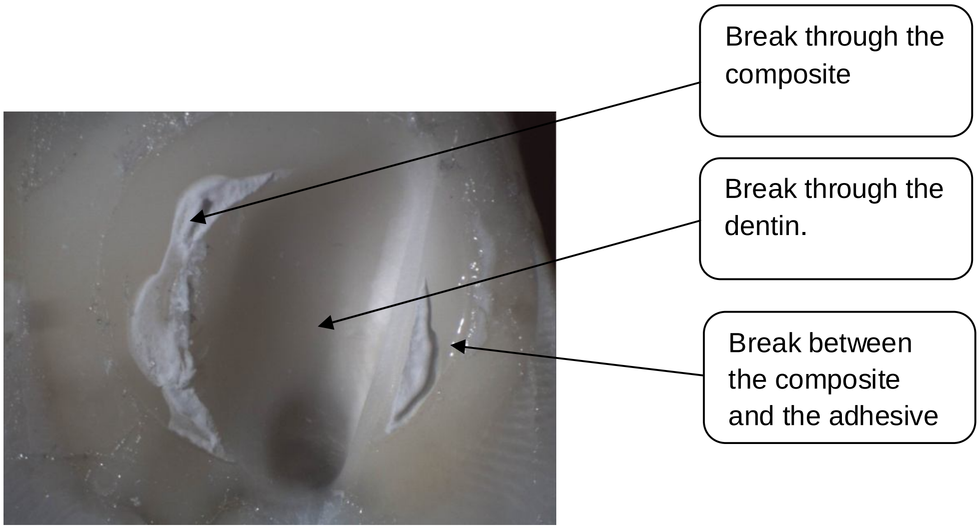


Figure 7.2: Mode of failure of Xeno V (single-step) shows 60% break through the dentin, 30% break through the composite, and10% break between the composite and the adhesive system.

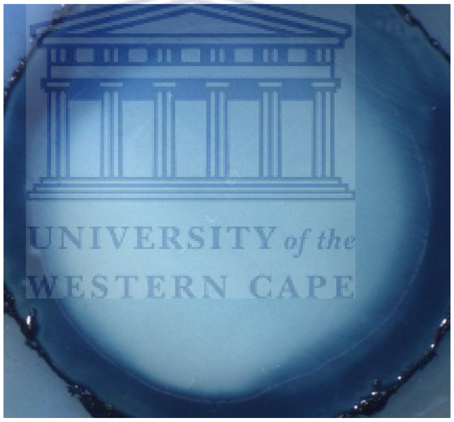


Figure 7.3: Mode of failure of Xeno V (single-step) shows 100% break between the composite and the adhesive system.

Chapter 8
Limitations of the study

With respect to this study, limitations that could have affected the outcome of the study are as follow:

- Only two materials were used to evaluate the bond strength of the two-step dentin bonding agents and the single-step dentin bonding agents.
- 500 thermal-cycles were used in this study, this could affect the bond strength values following thermal-cycling.
- The age of the selected teeth was not considered in this study.
- Results were based on a sample size of 25 specimens per group and may be differed if the samples were increased.

Chapter 9
Conclusion and recommendations

9.1 Conclusion:

The result of this study supports the null hypothesis that is there is no significant difference in the bond strength between the two-step and the single-step dentin bonding agents, and thermo-cycling for 500 cycles between 5^0C and 55^0C has no effect on the bond strength of these dentin bonding agents.

When comparing the results from this study with the results from other studies that used different materials, it can be suggested that the bond strength values with and without thermal-cycling can be variable among the different products used.

9.2 Recommendations:

1. Based on the results of this study both the two-step and the single-step dentin bonding agents could be as effective in bonding to dentin.
2. Also based on the results of this study the thermal changes in the mouth will equally affect both the two-step and the single-step dentin bonding agents.

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Appendixes:

- **Appendix 1:** Bond strength values of Prime and Bond NT (a two-step dentin bonding agent) without thermal-cycling:

Sample number	Prime and Bond NT without thermal-cycling group
1	2.29
2	2.6
3	2.66
4	2.68
5	4.13
6	4.13
7	5.16
8	5.2
9	5.75
10	6.02
11	6.03
12	6.08
13	6.19
14	6.38
15	6.53
16	6.84
17	7.04
18	7.11
19	7.59
20	7.89
21	8.07
22	8.77
23	9.58
24	10.43
25	<2

- **Appendix 2:** Bond strength values of Xeno V (a single-step dentin bonding agent) without thermal-cycling:

Sample number	**Xeno V withouth thermal-cycling group**
1	2.33
2	2.33
3	3
4	3.35
5	3.43
6	3.67
7	3.72
8	4.1
9	4.2
10	4.44
11	4.49
12	4.88
13	6.13
14	6.8
15	7.02
16	7.31
17	7.34
18	7.48
19	9.12
20	10.39
21	11.45
22	11.9
23	12.79
24	14.07
25	17.44

- **Appendix 3:** Bond strength values of Prime and Bond NT with thermal-cycling group:

Sample number	Prime and Bond NT with thermal-cycling
1	2.35
2	2.85
3	3.6
4	3.78
5	3.8
6	4.49
7	5.06
8	5.2
9	5.32
10	5.49
11	5.57
12	5.62
13	5.82
14	6.03
15	6.07
16	7.01
17	7.27
18	7.97
19	8.75
20	10.73
21	14.27
22	14.98
23	<2
24	<2
25	<2

- **Appendix 4:** Bond strength values of Xeno V with thermal-cycling group:

Sample number	Xeno V with thermal-cycling group
1	2.52
2	3
3	3
4	3.11
5	3.45
6	3.75
7	4.01
8	4.44
9	4.69
10	5
11	5.11
12	5.3
13	5.83
14	6.26
15	6.57
16	6.66
17	6.67
18	7.06
19	7.11
20	7.2
21	8.21
22	11.72
23	12.62
24	14.91
25	16.14