



An in-vitro evaluation of repair protocols applied to composite resin



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AN IN-VITRO EVALUATION OF REPAIR PROTOCOLS APPLIED TO COMPOSITE RESIN

KEYWORDS

Aging

Artificial saliva

Shear bond strength

Repair

Resin composite



ABSTRACT

The shift towards minimally invasive dentistry has meant that dental practitioners are now undertaking procedures that are conservative and preserve as much of the existing tooth structure as possible. Repairing composite is a more conservative way of managing damaged restorations when compared to their replacement. A number of different protocols for repairing composite restorations exist but there is little information as to which is the most effective method.

Aim: The aim of this study was to evaluate the effect the following treatment procedures have on the shear bond strength of repaired composite:

- i. Five different repair protocols,
- ii. Two different types of repair composite materials and
- iii. Aging in artificial saliva prior to repairing.

Materials and methods: Two hundred and forty composite cylinders of 5mm diameter and 5mm height made from Filtek Supreme XTE (3M ESPE, St. Paul, MN, USA) were prepared with the aid of a silicon matrix. They were then divided into two groups: a hundred and twenty of these cylinders were aged in a solution of artificial saliva for 28 days and the remaining samples were left unchanged with no aging. All the aged and non-aged composite cylinders were then randomly allocated to six groups of twenty each corresponding to the repair protocol applied.

The first group from both of the aged and non-aged samples was treated by roughening the top surface with a diamond bur followed by an application of Scotchbond 1XT (3M ESPE, St. Paul, MN, USA). The second group received a surface roughening with a diamond bur, etching with 35% phosphoric acid and application of Scotchbond 1XT. The third group

received an application of Scotchbond Universal (3M ESPE, St. Paul, MN, USA) and the fourth one had a single application of Tetric N-Bond Universal (Ivoclar Vivadent AG, Schaan, Liechtenstein) on its top surface. The fifth group was treated by blasting with COJET Sand (3M ESPE, St. Paul, MN, USA) particles together with an application of Scotchbond Universal. The final group was used as the control where no surface treatment was done.

After the surface treatments, each of the composite samples was repaired by the addition of fresh composite in the shape of cylinders measuring 3mm in diameter and 4mm in height. This was done with the aid of a silicon matrix. Within each treatment sub-group (n=20), 10 cylinders were repaired using either Filtek Supreme XTE or Tetric N-Ceram.

All two hundred and forty repaired samples were then subjected to shear bond strength testing on a Universal testing machine.

Data analysis: The results of the shear bond strength tests expressed in megapascals (MPa) were recorded and analysed for the effect of three different factors under consideration. The effectiveness of the repair protocols, type of composite and aging in artificial saliva were compared using the analysis of variance. Differences within the groups were identified using a post hoc analysis.

Results: The mean highest repair shear bond strength was observed when COJET Sand in conjunction with Scotchbond Universal was used to repair the aged composite blocks.

There were no significant differences in the shear bond strength observed when either Filtek Supreme XTE or Tetric N-Ceram was used as the repair composites.

Aging in artificial saliva led to a mean reduction of 18.08% in the repair bond strength across the six treatment groups.

Conclusions: The application of a surface treatment and intermediate adhesive is crucial in improving bond strength in the composite repair interface. Repair with Filtek Supreme XTE and Tetric N-Ceram was equally effective. Aging in artificial saliva produced significantly reduced bond strength.



DECLARATION

I hereby declare that “*An in-vitro evaluation of repair protocols applied to composite resin*” is my own work, that it has not been submitted before for any degree or examination at any university, and that all the sources I have used or quoted have been indicated and acknowledged by complete references.



Ken W. Irarĩ

14th March 2016



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DEDICATION

This work is dedicated to my wife Carolynne and our daughter, Cīra.



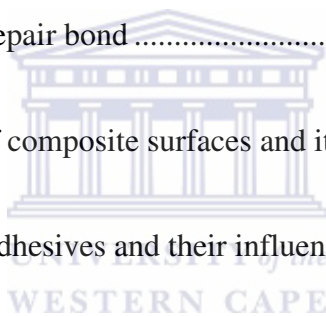
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CHAPTER 1

INTRODUCTION

The repair of old and defective composite restorations is increasingly gaining acceptance among dental practitioners. When compared to replacement of these restorations, repair of composites is less time-consuming, more economical and results in preservation of tooth structure that may have otherwise been lost. These repairs may be considered the treatment of choice for superficial discolouration of existing restorations, marginal defects limited to enamel, cusp fractures adjacent to a sound restoration, restoration of endodontic access cavities (Staxrud and Dahl, 2011), small areas of recurrent caries or when removal of a very large restoration would unnecessarily jeopardize the health of a tooth (Gordan *et al.*, 2012).

There exists a wide variety of approaches to the repair of composite restorations. With the simplest types of repair, fresh composite may be added to the old restoration with only phosphoric acid etching or roughening of the existing surface. More complex protocols may involve the use of adhesives, silanes and sandblasting with fine particles to improve the integrity and durability of the repair interface. However, there is still no strong clinical evidence as to which repair protocol is the most effective (Loomans *et al.*, 2011a).

Regardless of the repair method used, available evidence from laboratory and clinical studies have demonstrated that repair of failing composite restorations may prolong their lifespan while conserving enamel and dentine (Hickel *et al.*, 2013).

In this study, the influence of three different factors on the shear bond strength of repaired composite was evaluated based on a series of experiments that simulated clinically repaired restorations. The use of a solution of artificial saliva inoculated with oral bacteria represented a novel approach to the aging process for the testing of repair protocols for composite.

The first part of the literature review focuses on the composition and structure of current resin composites as well as a detailed examination of the characteristics of old composites and the nature of the repair interface. The second part reviews the reasons for failure of composite restorations and existing methods for repair. The third section provides an overview as to the different methods for evaluation of the repair, and clinical evidence for the effectiveness and durability of these repair procedures.



CHAPTER 2

LITERATURE REVIEW

Composition of resin-based composites

In general, resin composites consist of a polymeric matrix, inorganic fillers, a silane coupling agent and compounds that initiate or modulate the polymerization process. The relative amounts and types of each of these components will vary with each product and its clinical indication (Ferracane, 2011).

Resin-based composites (RBC) may be classified based on filler particle size and distribution (Table 1). The filler particles may be made from borosilicate, fused quartz, aluminium silicate, lithium aluminium silicate, barium, strontium, zirconium and zinc glass (Anusavice *et al.*, 2012). Fillers in the older macrofilled composites ranged between 10-50 μm , while the microfilled materials have 40-50 nm particles. In an attempt to maximize the advantages of these two materials, hybrid composites incorporating both macro- and microfilled particles were introduced (Ferracane, 2011).

More recent composites include the nanofilled materials, with filler particles ranging from 5-100 nm in size and the small particle hybrids which have a combination of nanoscale and microscale filler particles. The size, composition, shape and structure of the filler particles used determine, to a large extent, the mechanical and optical characteristics of the resin composite.

Table 1: Classification of Resin-based composites and indications for use (Anusavice *et al.*, 2012).

Class of composite	Particle size and type	Clinical use
Traditional (large particle)	1-50 μ m glass or silica	High-stress areas
Hybrid (large particle)	1-20 μ m glass or 40-nm silica	High-stress areas requiring improved polishability (Classes I, II, III and IV)
Hybrid (midfilled)	0.1-10 μ m glass or 40-nm silica	High-stress areas requiring improved polishability (Classes III and IV)
Hybrid (minifilled/ SPF)	0.1-2 μ m glass 40-nm silica	Moderate-stress areas requiring optimal polishability (Classes III and IV)
Nanohybrid	0.1-2 μ m glass or resin microparticles \leq 100nm nanoparticles	Moderate-stress areas requiring optimal polishability (Classes III and IV)
Packable hybrid	Midfilled/ minifilled hybrid, but with lower filler fraction	Situations where improved condensability is needed (Classes I and II)
Flowable hybrid	Midfilled hybrid with finer particle size distribution	Situations where improved flow is needed and/or where access is difficult (Class II)
Homogenous microfilled	40nm silica	Low-stress and subgingival areas that require a high luster and polish
Heterogenous microfilled	40nm silica Prepolymerized resin particles containing 40nm silica	Low-stress and subgingival areas where shrinkage is essential
Nanofilled	<100nm silica or zirconia Homogenous independent nanoparticles or nanoclusters	Anterior and noncontact posterior areas

Resin matrix

In many of the current composites, the polymeric matrix consists of a blend of dimethacrylate monomers such as bisphenol-A glycidyl methacrylate (bis-GMA) and urethane dimethacrylate (UDMA) (Anusavice *et al.*, 2012). Owing to its high viscosity, these monomers are usually combined with diluent dimethacrylate monomers such as triethylene glycol dimethacrylate (TEGDMA). In some of the more recent products modified versions of the diluent monomers, such as TEGDMA, may themselves serve as the base monomer (Ferracane, 2011).

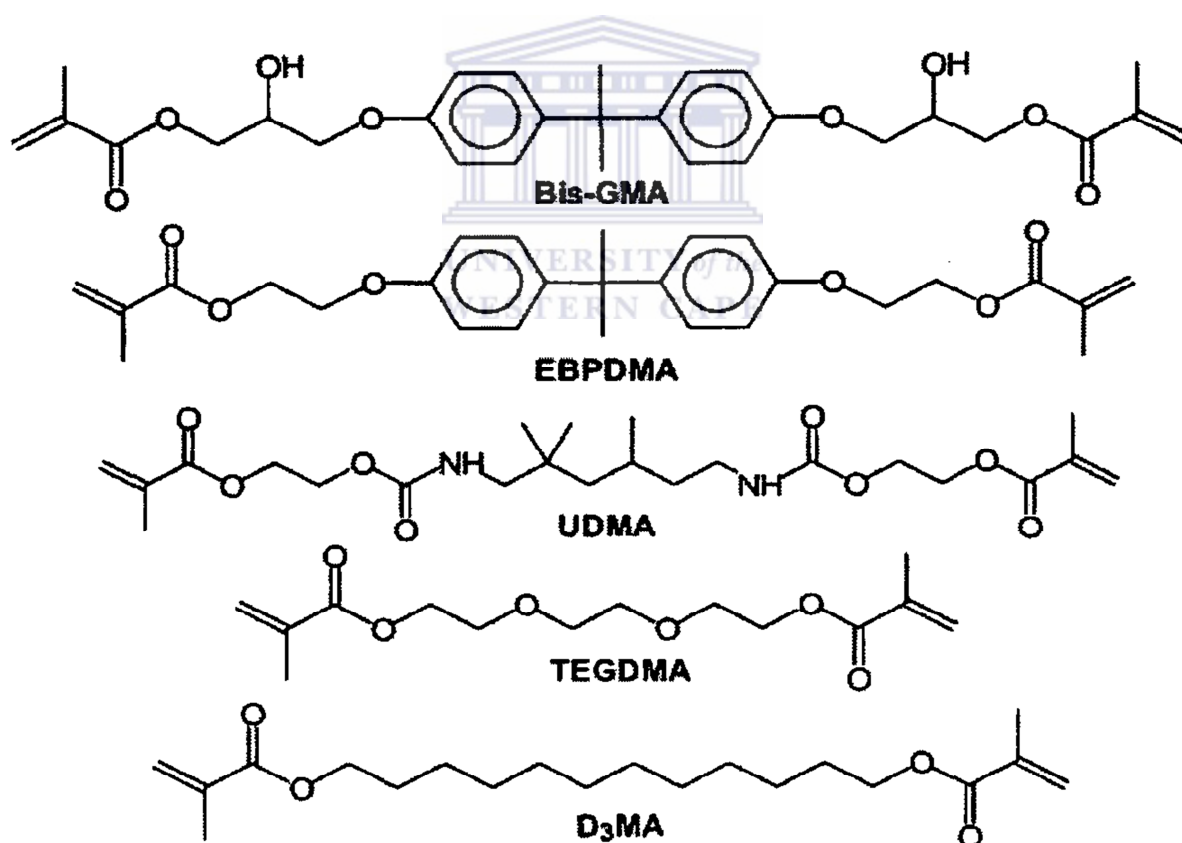


Figure 1: Chemical structure of monomers commonly used in resin-based composites (Anusavice *et al.*, 2012)

The polymerization of these monomers may be initiated by visible light, a chemical reaction or a combination of both mechanisms. For most light- or dual-cured composites, the polymerization is mediated by a photoinitiator such as camphoroquinone accelerated by a tertiary amine (Ilie and Hickel, 2011).

Silane coupling agents

Silanes are hybrid organic-inorganic surface-active compounds that react with and promote adhesion between the inorganic and organic components of dissimilar materials (Matinlinha *et al.*, 2004). In the case of resin composites, the silane coupling agent (SCA) serves to chemically bind the inorganic filler particles to the resin matrix. By doing so, the more flexible resin is able to transfer stresses to the filler particles which are more rigid and stiffer (Anusavice *et al.*, 2012).

The most commonly used silanes are the organosilanes such as γ -methacryloxypropyl trimethoxysilane (MPS), whose structure is shown below (Figure 2).

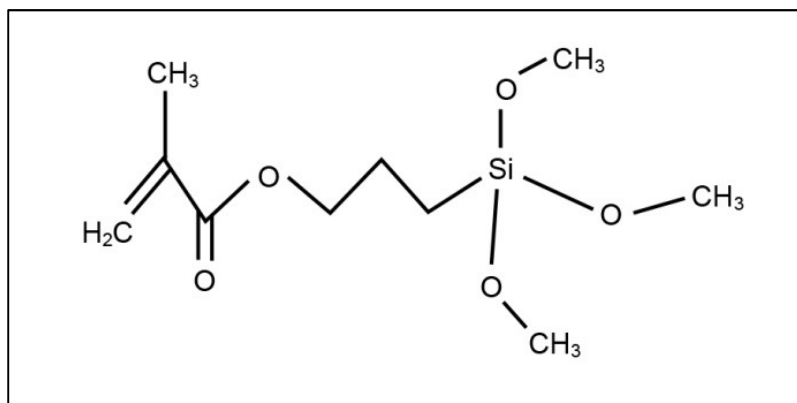


Figure 2: Chemical structure γ -methacryloxypropyl trimethoxysilane (Matinlinha *et al.*, 2004)

In the presence of water, the methoxy groups (-OCH₃) are hydrolysed to silanol (-Si-OH) groups, which then bond to form siloxane (-Si-O-Si-) bonds with other silanols on the surface of the filler. On the other hand, the methacrylated groups bond to the resin after polymerization, thus linking the filler and resin matrix (Figure 3). The bond mediated by the SCA between the resin matrix and filler particles is crucial and has a significant influence on the mechanical properties and durability of the resultant composite.

This allows for improved mechanical and physical properties while protecting the filler-resin interface from hydrolytic degradation.

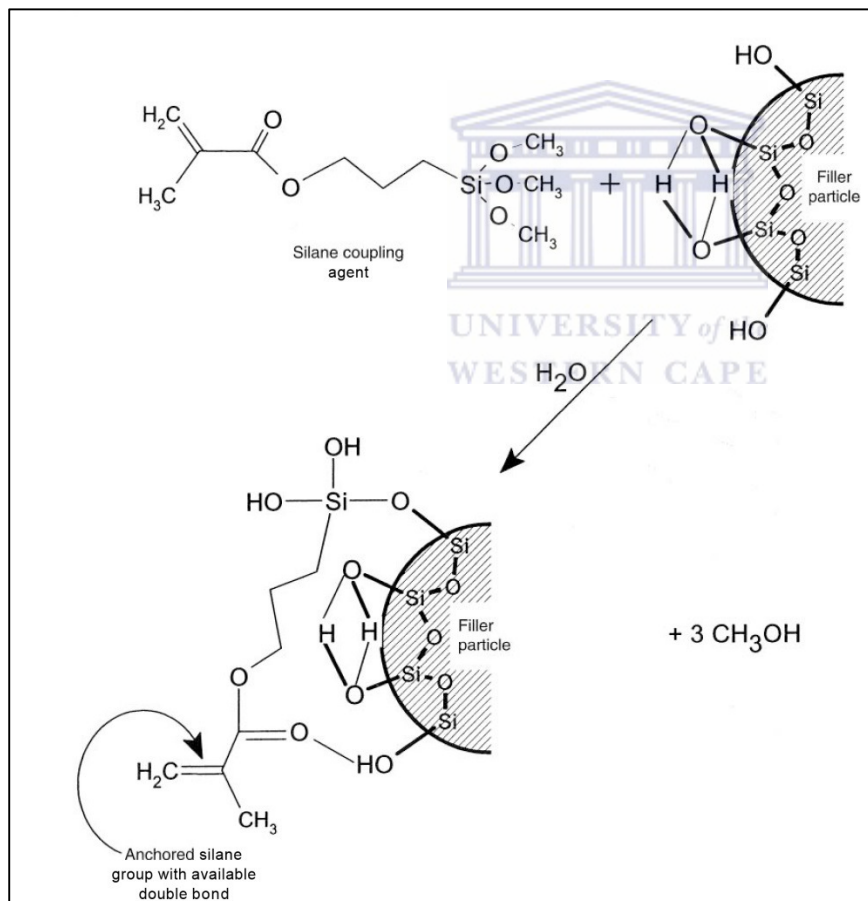


Figure 3: Simplified mechanism showing the action of a silane coupling agent (Anusavice *et al.*, 2012)

Mechanisms of RBC degradation

Following placement of the restoration, resin based composites undergo significant changes intraorally that result in a gradual degradation of the material over a period of time. The extent and rate of deterioration is dependent on the nature of monomer making up the resin matrix, degree of monomer conversion, structure of the filler particles and the integrity of the silane coupling between the filler and resin matrix (Curtis, 2008). In vitro, one consequence of aging in artificial saliva is the degeneration of one or possibly all of the components of the RBC; resin matrix, filler particles, silane and initiators (Asmussen and Peutzfeldt, 2003).

The clinical failure of composite restorations almost always results from a deterioration of the material through a combination of chemically-induced or mechanistic processes. Chemically induced degradation occurs following the action of moisture, salivary enzymes and solvents present in food on the restoration. On the other hand, mechanical degradation may be as a result of micro-cracks developing within the restoration, wear of the surface and static fatigue (Curtis, 2008).

Chemically induced failure

The most common form of degradation takes place within the resin matrix, occurring by hydrolysis in the presence of moisture or due to the action of salivary enzymes. In a process known as chain scission, the highly crosslinked polymer chains are broken down into shorter fragments (Figure 5). This coupled with oxidation of the functional groups within the polymer, lead to eventual inactivation of the resin (Curtis, 2008).

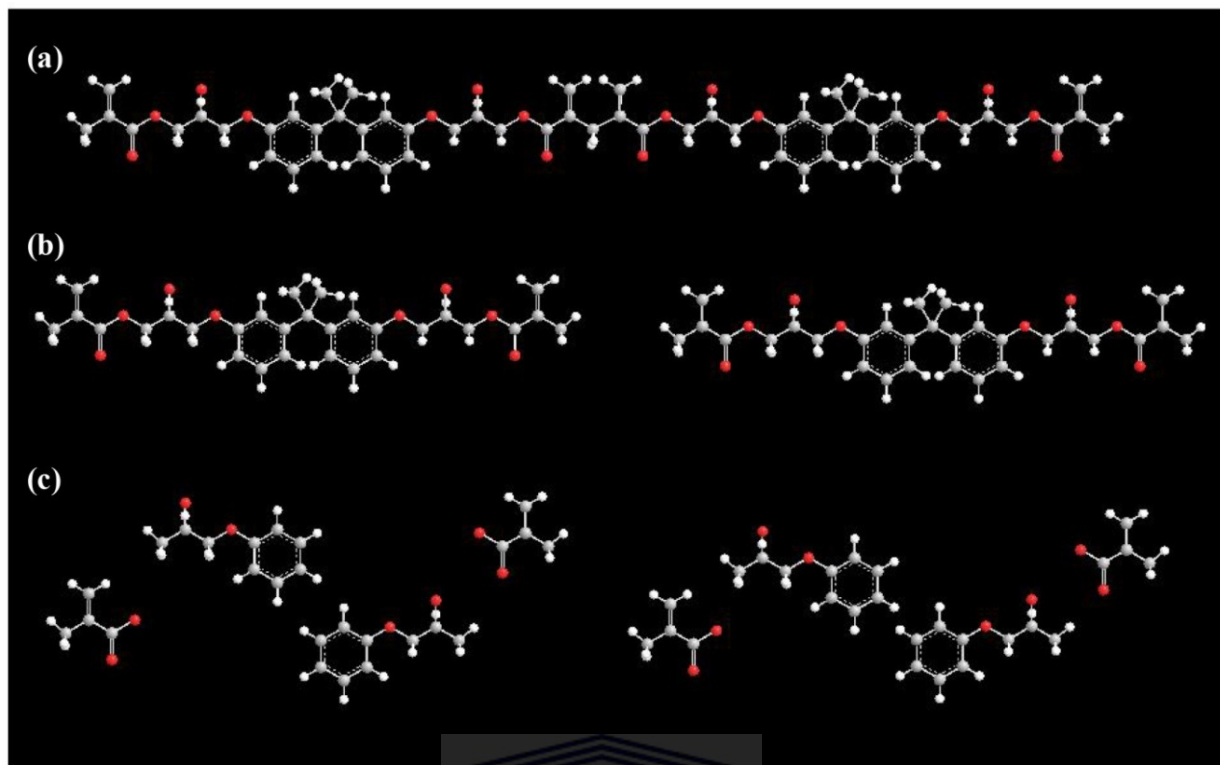
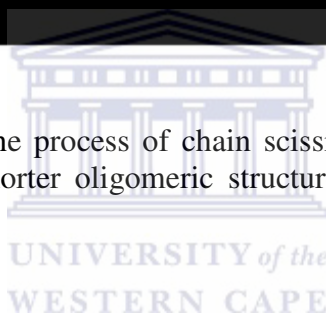


Figure 4: Diagram illustrating the process of chain scission. (a) bis-GMA based polymer. Chain scission has led to (b) shorter oligomeric structures and eventually (c) monomeric chain fragments (Curtis, 2008).



This process is influenced by the degree of cross linkage within the polymer which is in turn dependent on the degree of monomer conversion (DC). A high degree of conversion leads to a highly crosslinked structure with improved mechanical properties while a low DC results in a polymer that is more susceptible to hydrolytic and chemical degradation (Asmussen and Peutzfeldt, 2003).

The presence of moisture is another factor that is known to promote the disintegration of the resin composite. Water sorption may lead to dissociation of the silane-filler bond which lowers the mechanical properties of the material and renders its surface more susceptible to degradation.

In this process, water uptake into the body of the composite causes the material to become plasticized and lose its fracture strength and elastic modulus (Lohbauer *et al.*, 2003). Hydrophilic monomers such as TEGDMA are responsible for the bulk of water sorption; and as such, RBCs with a high content of these monomers have an increased susceptibility to hydrolytic degradation (Musanje *et al.*, 2001).

Salivary enzymes, to a degree, are also responsible for deteriorating of resin composite restorations. A study by Jaffer *et al.* (2002) showed that human saliva esterases catalysed the degradation of BisGMA and TEGDMA within two different brands of resin composite. Methacrylate byproducts of this chemical breakdown were later identified by high-performance liquid chromatography (HPLC), showing that the action of saliva was on the polymer matrix as well as unreacted monomer within the RBC. The mechanism of action of these salivary enzymes on resin composite involves hydrolysis of ester bonds to methacrylic acid and scission of the polymer chains (Yap *et al.*, 2000).

Continual elution of the monomer may result in only a very small amount of residual monomer which in turn is unable to form a chemical bond between the old and fresh composite. Indeed, the effect of the oxygen inhibition on the surface is also greatly reduced since passive polymerization further reduces the amount of unreacted monomer (Curtis, 2008).

Mechanical failure of RBCs

A number of factors may influence the clinical longevity of RBCs namely; the occurrence of fatigue (Garoushi *et al.*, 2007), environmentally assisted crack growth (Söderholm and Roberts, 1990) and wear resistance (Bagheri *et al.*, 2007). This is due to the fact that, intraorally, composite restorations are exposed to a variety of mechanical forces that may induce their failure. During function, the restoration is subject to shearing, compressive and tensile forces.

These cyclic masticatory stresses may lead to the eventual displacement and loss of material from the restoration (Curtis, 2008).

Clinical failure of composite restorations

Failure of resin-based composite restorations may be as a result of aesthetic, functional and biologic reasons. A restoration may be regarded as having failed due to poor aesthetics when one or more of the following is identified: loss of surface gloss, surface and marginal staining, poor colour match and loss of aesthetic anatomical form (Hickel *et al.*, 2010). On the other hand, fracture of the restoration, inadequate marginal adaptation, occlusal wear and improper approximal contour may be considered to be functional failures.

Other modes of failure such as recurrence of initial pathology (for example caries), tooth cracks and fractures as well as effect of the restoration on the periodontium have been classified as biological failures. These types of failures are related to patient factors such oral hygiene, parafunction, presence of periodontal disease (Hickel *et al.*, 2010). However, the failure of a restoration is usually due to a combination of two or more factors. It is therefore necessary to identify the causes of failure as some failed restorations cannot be repaired successfully and would need to be replaced (Blum *et al.*, 2011).

Repair of defective composite restorations

Rationale for repair of restorations

It is estimated that on average, complete removal of a composite restoration results in a loss of tooth structure that is twice the volume lost when amalgam restoration of the same size is replaced (Krejci *et al.*, 1995). This is due in part to the lower optical contrast between composite and natural tooth structure as well as the bonding between the two interfaces. Additional tooth material may also be lost when macro-mechanical retentive features such as undercuts, grooves and slots are incorporated into the cavity preparation (Gordan, 2000). In contrast, with repair of a restoration, very little additional tooth structure is removed. This therefore makes repair a much more conservative alternative to removal of the restoration and placement of a new one.

In addition, repair or refurbishing composite restorations is less time-consuming and relatively inexpensive (Baur and Ilie, 2013). In certain instances, however, repair may not be a viable option and complete replacement of the restoration would provide a more favourable prognosis for the tooth in question. For example, large areas of secondary caries, extensive damage to the original restoration and poor initial cavity design may preclude repair of the restoration and it is therefore recommended to replace these types of restorations (Blum *et al.*, 2011). The selection of cases is thus critical in ensuring the success of repair to composite fillings.

Techniques for repair of defective composite restorations

As a general rule, repair procedures involve superficial modification of the old restoration followed by placement of a new layer of composite, with or without silanization of the interface. Surface modification may involve the creation of undercuts, sandblasting with

abrasive micro-particles, roughening with a bur, etching with an acidic compound (Szep *et al.*, 2000) or any combination of these procedures (Loomans *et al.*, 2011a). This is necessary to create macro- or micro-mechanical areas of retention on the old restoration.

Following surface preparation, a silane solution may be applied onto the old composite followed by an adhesive resin, which is then polymerized. The silane chemically bonds to the silica-coated composite and presents a surface for bonding of the new adhesive resin (Matinlinna *et al.*, 2004). The repair composite may then be applied and polymerized. Another approach involves acid-etching of the composite resin together with the adjacent tooth tissue followed by application of the adhesive and repair composite, in the manner of a new restoration (Blum *et al.*, 2011).

A recent innovation has been the development of the so called ‘universal adhesives’ which aim to simplify the bonding and repair process. Some of these materials, such as Scotchbond Universal (3M ESPE, St Paul, Minnesota) contain a silane which is meant to aid in bonding new to old composite (3M ESPE, 2013).

The composite surface as a bonding substrate

A fresh composite restoration, when free from contamination, presents an excellent surface for bonding due to the presence of an oxygen inhibited layer containing unpolymerized resin. During light curing, the presence of atmospheric oxygen retards the polymerization of the superficial layer of resin composite to a depth of approximately 4-40 μm . The oxygen reacts with radicals from the monomer to form inert peroxy radicals which slow down the polymerization process, resulting in a resin-rich surface layer (Anusavice *et al.*, 2012). It is this resin-rich layer, consisting mainly of unreacted monomers and oligomers, which copolymerizes directly when a subsequent layer of composite is added incrementally onto it. The cohesive bond between two layers of fresh composite is very strong, and ranges from 19.8 to 40 MPa (Staxrud and Dahl, 2011).

In contrast, the surface of an old composite may be significantly altered, depending on the age of the restoration and the material's composition. Like any other restorative material, the composite is exposed to the action of salivary enzymes, intraoral bacterial biofilm, temperature changes, acids in food as well as chewing forces. Each of these factors may cause changes in the structure and properties of the material. Hence older composites do not contain an oxygen inhibition layer, a factor that hinders chemical bonding to fresh composite.

The composite repair interface

As a result of the changes that take place in an old restoration, it is doubtful whether there is any significant chemical bonding between old and new composites. The connection between these two materials is therefore likely to be primarily mechanical or micromechanical in nature.

Scanning electron micrographs of clinically and laboratory aged composites have demonstrated that degradation does occur on the surface of these restorations irrespective of the aging medium used (Figures 5 and 6). These may provide a roughened surface onto which a new resin composite may adhere by micromechanical means. Additional surface roughness can be created by running a diamond bur across the composite surface. Alternatively, sandblasting of the same surface with silica-coated particles increases the surface area for micromechanical retention.

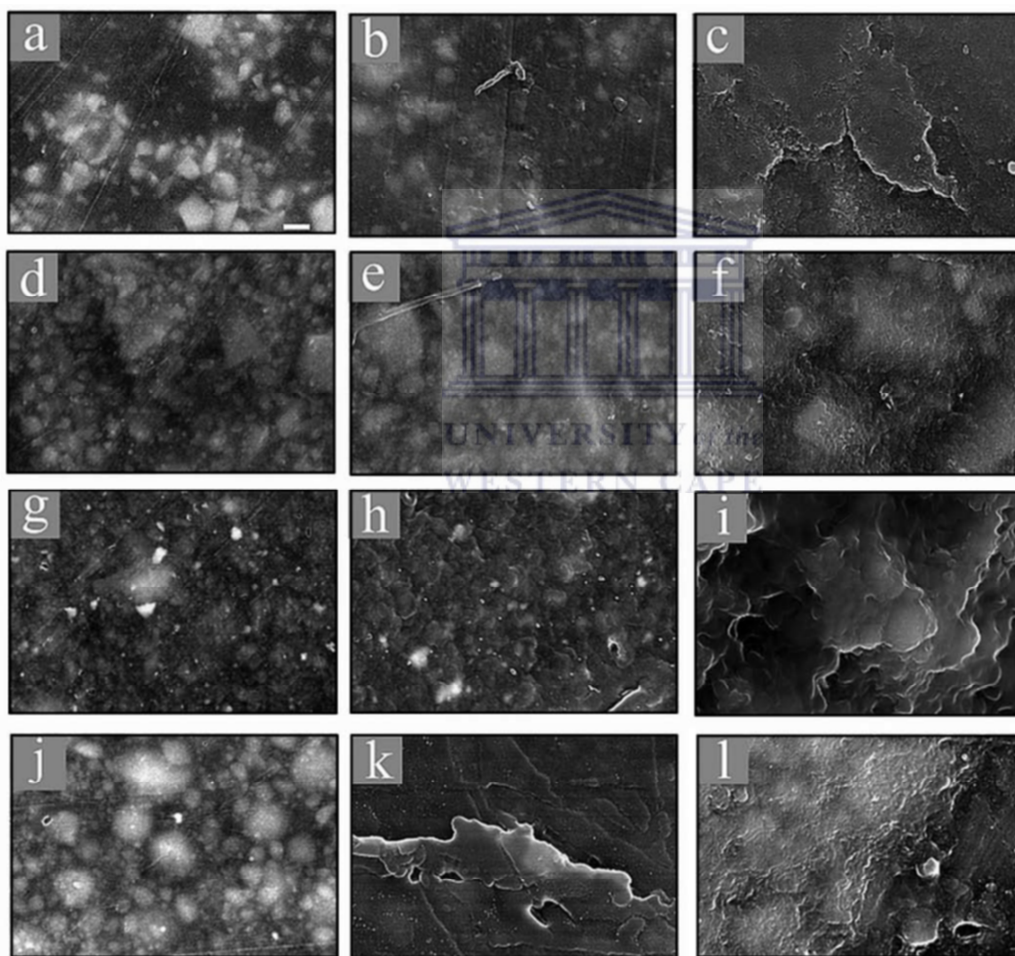


Figure 5: Comparison of non-aged, artificially-aged and clinically aged composite samples. The aging solution used consisted of an artificial saliva biofilm. Representative micrographs taken from a scanning electron microscope (SEM) of all composite resins used in this study. Left column, non-aged; middle row, after aging by in vitro exposure to biofilm; and right row, aging after clinical wear in a palatal appliance. (a-c) Microhybrid Anterior Shine. (d-f) Nanohybrid Grandio. (g-i) Nanohybrid Tetric Evo Ceram. (j-l) Nanofilled Filtek Supreme XTE. From Rinastiti *et al.*, (2010).

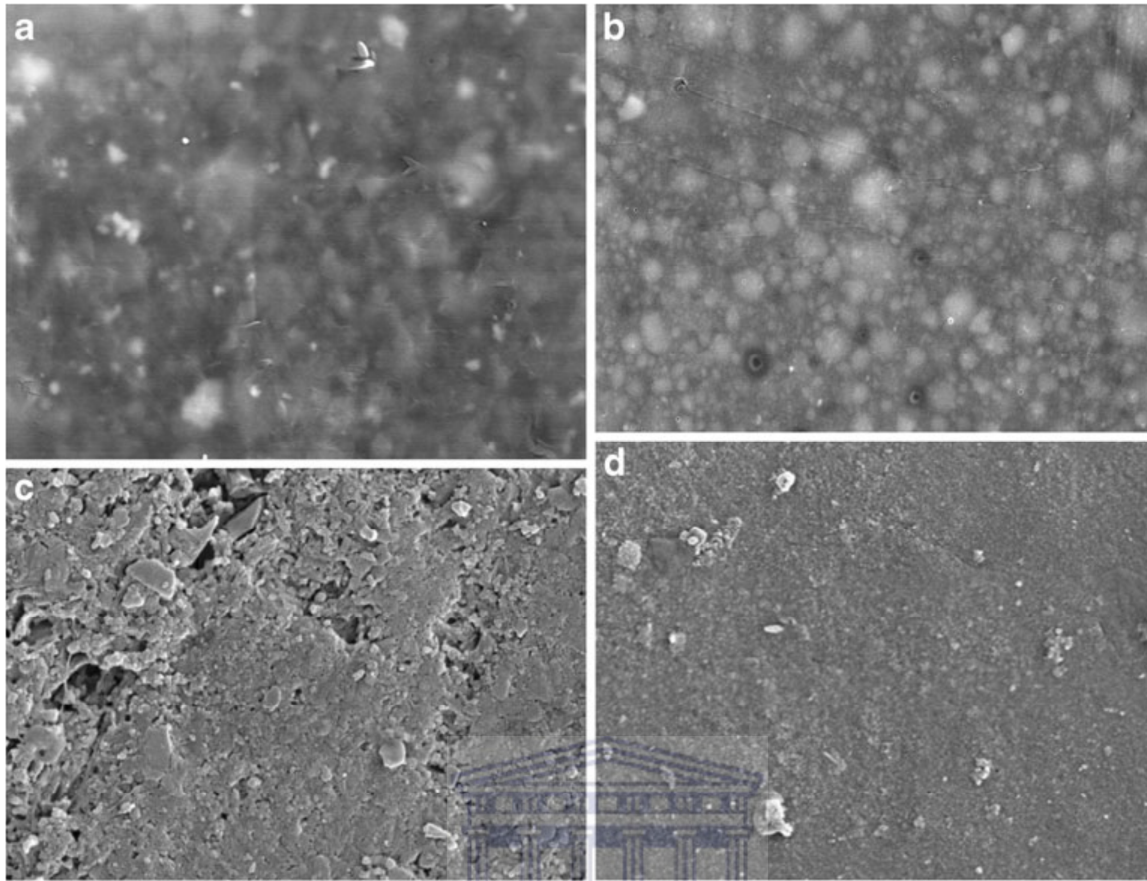


Figure 6: SEM micrograph illustrating the surface degradation observed following aging of composite samples by thermocycling. (a) non-aged Tetric Evo Ceram (TE) (b) non-aged Filtek Supreme XTE (FS). (c) TE following thermocycling (d) FS following thermocycling. From Özcan *et al.* (2013).

Role of the adhesive on the repair bond

In the absence of an adequate amount of unreacted monomer on the surface of an old composite restoration, alternative approaches have to be used to improve the bond strength between old and new composite. Given that the old restoration is likely to have an increase in its water content, direct bonding of the hydrophobic monomers in the fresh composite to the old composite may be difficult. Use of an adhesive containing the hydrophilic primer 2-hydroxyethylmethacrylate (2-HEMA) overcomes this limitation by promoting wetting of the old composite and penetrating its irregular surface, even in areas with a high water content (Curtis, 2008).

This property allows for improved micro-mechanical retention. Any remaining hydrophobic monomers in the composite, as well as un-polymerized resin in the fresh composite then bond chemically to the hydrophobic component of the 2-HEMA. A number of laboratory studies have validated this hypothesis, consistently showing an improvement in bond strength between two composites when an adhesive is used. In a study comparing the mean shear bond strength in composite repairs with and without adhesive, Straxud and Dahl (2011) showed that the use of a bonding agent resulted in a shear bond strength that was 3-4 times greater than in repairs in which no adhesive was used. Loomans *et al.* (2011a) also demonstrated that composite repairs that were carried out without the use of an adhesive had the lowest micro tensile bond strength.

The use of an adhesive is also sometimes necessary when the need for bonding to exposed tooth structure arises. For instance, during repair of restorations, enamel and dentine may be uncovered by the cutting action of burs on defective margins or surfaces, necessitating the use of an adhesive prior to the addition of fresh composite.

Silane in the pre-treatment of composite surfaces and its effect on bond strength

A number of investigators have proposed the silanation of composite surfaces prior as a method to improve the bond strength (Straxud and Dahl, 2011; Özcan *et al.*, 2013). The premise, similar to that of porcelain repair, is for the silane primer to bond itself to the exposed silica or metal fillers and present a surface for application of the adhesive or composite resin. Silane solutions used for ceramic repairs may be employed for this purpose. More recently, solutions that are specifically indicated for composite repairs such as 3M ESPE-Sil have been developed.

The use of this technique has not been universally accepted among practitioners and researchers. Indeed, the data from a number of studies on the effectiveness of silane pre-treatment during composite repairs is conflicting. On the one hand, following a series of experiments using different repair protocols, El-Askary *et al.*, (2012) reported that “the use of a silane did not improve the repair bond strength”. The results of this study corroborated the findings of Papacchini *et al.*, (2007a) and Bonstein *et al.*, (2005) who also noted the limited ability of silane in improving outcomes from repair of RBC restorations.

On the other hand, Özcan *et al.*, (2013) demonstrated that sandblasting followed by silanation of aged composite surfaces prior to repair resulted in micro tensile bond strengths that were comparable to values obtained following repairs made using adhesives alone. This indicates that, at the very least, use of the silane does not have a detrimental effect on the integrity of the bond between composites. Yet another investigation provided evidence of the effectiveness of silanation (Rinastiti *et al.*, 2011). In this study, two different repair protocols (sandblasting with silanation and use of adhesive resin) were compared. The mean bond strength values obtained from the silane/ sandblasting group were significantly higher than

those observed when only adhesive resin was used to repair a similar set of composite samples.

From the results of these studies, it would appear that silanation is only effective when used in conjunction with sandblasting of the composite surface. In this technique, very fine silica or silicized aluminium oxide particles (30-50 μm in diameter) are blasted onto the composite surface prior to application of the silane. Once these particles are embedded in the surface of the old composite, they present a fresh silica surface for adhesion of the silane molecules.

A possible drawback regarding the application of silane solutions and adhesive separately is that it results in a thick and multi-layered interface that may present a weakened bond (El-Askary *et al.*, 2012). Moreover, the risk of introducing defects into the bonding interface increases with each working step.

Incorporation of silane into adhesives and their influence on repair bond strength

Recently, universal adhesives (UAs) such as Scotchbond Universal (3M ESPE, Maplewood, U.S.) and Tetric-N-Bond Universal (Ivoclar Vivadent, Schaan, Liechtenstein) have been introduced into the dental market. The main aim for developing these bonding agents was to make the clinical application of adhesives more user-friendly. These materials have a wide variety of applications such as bonding ceramics, metal and resin-based composite restorations. In addition, they can be used as part of the etch-and-rinse, selective etch and self-etch protocols when bonding to tooth structure.

In general, the so-called universal adhesives contain hydrophilic monomers and may incorporate functional monomers such as 10-methacryloyloxydecyl dihydrogenphosphate (MDP) which demonstrate an effective and durable bond to dentine. In addition, the UAs are

mildly acidic, a property which allows direct chemical bonding to enamel when used in the self-etch mode (McLean *et al.*, 2013).

Universal adhesives such as Scotchbond Universal also contain silane, which facilitates adhesion to composites and ceramics. As such, these materials are indicated for the repair of composites as well as other restorations, doing away with the need for a separate silanation stage of the surface to be repaired. However, there is currently very little published information on the in-vitro and clinical efficacy of these types of adhesives during the repair of RBCs.



***In-vitro* assessment of the effectiveness of composite repairs**

Artificial aging of composites in-vitro

Clinically, repair of a composite restoration involves bonding a fresh composite onto an old and possibly degraded filling. In order to simulate the defective restoration, laboratory aging of composite test samples is necessary. A number of methods have been used for this purpose, including thermocycling, immersion in distilled or deionized water, boiling or even in vitro exposure to citric acid and sodium chloride solutions (Hickel *et al.*, 2013). Of these, thermocycling appears to be the most commonly employed and is regarded as an effective method of aging the material. However, there is as yet no standardization as to the number of cycles or the duration of time for which the substrate should be thermocycled (Hickel *et al.*, 2013).

A method in which composite blocks were aged in a mixed-species biofilm was recently reported by Rinastiti *et al.* (2010). This particular biofilm was formulated according to the Zürich biofilm model proposed by Guggenheim *et al.* (2004) and consisted of a variety of organisms, including *Streptococcus oralis*, *Actinomyces naeslundii*, *Fusobacterium nucleatum* and *Candida albicans* together with 5% fetal bovine serum. In this case, scanning electron microscope scans showed that composite surfaces exposed to this medium were degraded in a manner similar to that of composite blocks left intraorally for eight weeks within a palatal appliance. Within the literature surveyed, very few studies have utilized this aging method and studied its influence on bond strengths, representing a knowledge gap that this study will attempt to bridge.

It is important to note that this use of artificial saliva only simulates the hydrolytic degradation of composite. The effect of temperature variation may be tested using thermocycling while exposure to citric acid attempts to mimic the action of acids within

foods. However, the cyclic loading of composite from repeated chewing is more difficult to reproduce and is not factored in these types of aging.

Bond-strength testing of the composite repairs

For the evaluation of the interface between new and aged composite, the shear and micro-tensile bond strength tests may be employed. Of these two, the shear bond strength test appears to be the more widely used, perhaps owing to its relative ease of application (Hickel *et al.*, 2013). However, results based on experiments employing this test may be influenced by the modulus of elasticity of the material under investigation. For this reason, it is recommended that the same composite material be used as the substrate in all test specimens when the shear bond test is applied (Heintze, 2013).

Loomans *et al.* (2011a) conducted an experiment to assess the bond strength of five composite restoratives using nine different preparation protocols for each material. None of the protocols tested was found to be statistically superior to the others. However, the authors recommended that an ideal situation involved bonding a repair composite of the same type as the adherend. Where the original composite was unknown to the dentist, etching the old composite surface with phosphoric acid and sandblasting with micro fine powder was recommended (Loomans *et al.*, 2011a).

Available data from laboratory studies shows that the repair bond strength is dependent on the repair technique used and type of composite used (Loomans *et al.*, 2011; Özcan *et al.*, 2013). This was also confirmed by Rinastiti *et al.* (2010) who concluded that application of a silica coating was more effective than use of intermediate adhesives in all three types of composites tested.

Durability of the repair interface

Following repair of the restoration, the bond between old and new composite may be susceptible to degradation owing to the same factors that promote the chemical and physical breakdown of the composite. In the presence of saliva and moisture, both the adhesive and new composite are likely to undergo water sorption. In addition, the carboxyl and hydroxyl groups present in each of these resins may be broken down leading to a weakening of the bond interface. Preventing or slowing down this hydrolytic degradation will obviously improve the durability of the repaired restoration (Papacchini *et al.*, 2007b).

It has been suggested that use of flowable resin during the repair may stabilize the repair bond and improve its resistance to hydrolysis (Staxrud and Dahl, 2011). This may be due to the hydrophobic nature of the flowable resins. Certainly, improved bond strength values have been demonstrated following the use of flowable resin in the repair (Staxrud and Dahl, 2011).

The etch-and-rinse bonding procedure provides the best hydrolytic stability following repair, owing to the fact that most of the hydrophilic resin within the primer is eliminated during air-drying. In the study by Staxrud and Dahl (2011), after aging of the test samples for 60 days, the three step etch-and-rinse approach produced the highest mean repair bond strength values when compared to other bonding strategies.

Clinical evidence for effectiveness of repair of damaged restorations

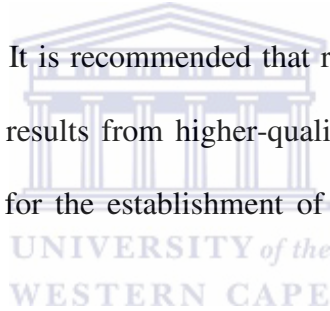
The repair of defective composite restorations has recently been the subject of numerous clinical studies. The evidence available appears to indicate that repair may prolong the lifespan of these restorations while having the advantage of being minimally-invasive.

In a prospective clinical trial, Moncada *et al.* (2009) repaired 78 defective Class I and II restorations in 66 patients and followed the cases over a three-year period. It was found that repair and refurbishment procedures carried out on deteriorating composite restorations led to improved survival rates when compared to an untreated group of restorations. Data from this same cohort of patients after four years (Fernandez *et al.*, 2011) provided findings consistent with results provided at 3 years, namely improved survival of repaired restorations. The same patients were followed up for a period of ten years and results showed that composite restorations that had been repaired had the same survival rate as those that had undergone total replacement (Fernandez *et al.*, 2015)

In another study based on a seven-year follow-up of 38 patients with 88 restorations, Gordan *et al.* (2009) concluded that repair resulted in an increase in the longevity of failing composite restorations. It was found that, within this cohort, no restorations failed following repair or marginal sealing compared with 23% of similar unrepaired restorations showed evidence of failure. Even with this limited sample, it appears that repairing damaged restorations may play a role in prolonging their lifespans.

Summary

Composite repair is a viable and more conservative alternative to replacement of restorations (Blum *et al.*, 2011). However, the evidence on the expected longevity of repaired restorations is still weak, with no randomized clinical trials investigating outcomes following composite repair (Sharif *et al.*, 2014). The studies investigating these outcomes have tended to use non-standard criteria for reporting failure which may distort the results. Reference is made to the publications by Hickel *et al.* (2007 and 2010) in which detailed criteria for evaluating the quality of restorations is provided. In the studies by Moncada *et al.* (2009) and Gordan *et al.* (2009), for example, chipped or fractured restorations were not included in the 'failed' categories. It may be postulated that these omissions led to skewed data; hence the reported survival rates may be inaccurate. It is recommended that results from non-randomized trials be interpreted with caution until results from higher-quality studies are available (Sharif *et al.*, 2014). There is also a need for the establishment of standard protocols for composite repairs.



CHAPTER 3

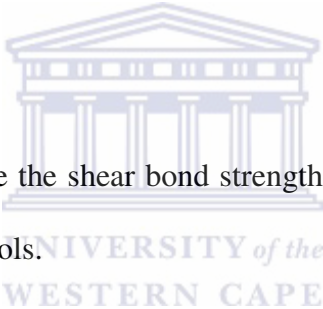
AIM & OBJECTIVES

Aim of the study

To evaluate the effect of three different factors on the shear bond strengths between new and old composite. These factors are:

1. Repair protocols,
2. Repair composite,
3. Aging.

Objectives

- 
1. To determine and compare the shear bond strengths of repaired composite following five different repair protocols.
 2. To determine and compare the shear bond strengths when cylinders of Filtek Supreme XTE were repaired with two different composites.
 3. To determine whether aging of composites in artificial saliva affects the shear bond strengths of the repaired composites.

Null hypotheses

1. There will be no difference in the shear bond strength when five different repair protocols are applied to aged composite specimens.
2. There is no difference between the shear bond strength when either Filtek Supreme XTE or Tetric N-Ceram is used as the repair composite.

3. Aging of the composite cylinders in artificial saliva has no effect on the shear bond strength following repair.



CHAPTER 4

MATERIALS AND METHODS

The different materials that were used in this study are listed in Table 2.

Table 2: Materials used for this study

Material	Description	Manufacturer
Filtek Supreme XTE, shade A3 Body (A3B)	Nano-filled restorative composite	3M ESPE, St. Paul, MN, USA
Tetric N-Ceram, shade A3	Nano-hybrid restorative composite	Ivoclar Vivadent AG, Schaan, Liechtenstein.
Adper Scotchbond 1XT Adhesive	Etch-and-rinse adhesive	3M ESPE, St. Paul, MN, USA
Scotchbond Universal Adhesive	Single-bottle universal adhesive	
Tetric N-Bond Univeral	Single-bottle universal adhesive	Ivoclar Vivadent AG, Schaan, Liechtenstein.
Ultra-Etch	Phosphoric acid etchant	Ultradent Products, Inc., South Jordan, UT, USA
COJET Sand	Silicized abrasive particles	3M ESPE, St. Paul, MN, USA

Filtek Supreme XTE

A visible light-activated composite designed for anterior and posterior restorations. This product is available in dentine, enamel, body and translucent shades. The polymeric matrix consists of bis-GMA, UDMA, TEGDMA and bis-EMA resins. To reduce the shrinkage, PEGDMA has been substituted for a portion of the TEGDMA resin in Filtek Supreme XTE restorative (3M ESPE, 2010)



Figure 7: Filtek Supreme XTE

The inorganic fillers include non-agglomerated/non-aggregated 20nm silica, non-agglomerated/ non-aggregated 4 to 11nm zirconia filler and aggregated zirconia with silica cluster filler of between 4 to 11nm and 20 nm diameters respectively. The inorganic filler loading is about 72.5% by weight (55.6% by volume) for the translucent shades and 78.5% by weight (63.3% by volume) for all other shades (3M ESPE, 2010).

Tetric-N-Ceram

A light-curing, nano-hybrid composite indicated for both anterior and posterior restorations.



Figure 8: Tetric N-Ceram

Its resin matrix is made up of bis-GMA and UDMA (15% by weight) and ethoxylated bis-EMA (3.8%) as well as prepolymers (17%). The inorganic fillers (63.5% of the composite by weight) consist of barium glass, ytterbium trifluoride and silicon dioxide. Pigments, additives, stabilizers and catalysts are also present in the material and comprise 0.7% of the composite. This product is available in a variety of tooth shades (Ivoclar Vivadent, 2010).

Adper Scotchbond 1XT

A total-etch, visible-light activated dentine bonding agent incorporating silica filler. This adhesive is indicated for use with direct light-cured restorative materials as well as for the treatment of cervical dentine hypersensitivity.



Figure 9: Adper Scotchbond 1XT

This product contains bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system together with a methacrylate functional copolymer of polyacrylic and polyitaconic acids. In addition, Adper Scotchbond 1 XT adhesive incorporates 10% by weight of 5 nanometer-diameter spherical silica particles (3M ESPE, 2004).

Scotchbond Universal



Figure 10: Scotchbond Universal

A single-bottle light-activated universal adhesive that has a wide variety of applications; including the repair of composite and ceramic restorations. It consists of MDP Phosphate monomer, dimethacrylate resins, HEMA, filler, ethanol, water, initiators and a silane coupling agent.

According to the manufacturer, the presence of silane allows the adhesive to chemically bond to glass ceramic and composite surfaces without using a separate ceramic primer or silane (3M ESPE, 2013).

Tetric N-Bond Universal

A single component, light-cured universal adhesive indicated for the repair of composite restorations (Ivoclar Vivadent, 2015). Its matrix is based on a combination of HEMA, decandiol dimethacrylate (D3MA) and bis-GMA in addition to MDP and methacrylated carboxylic acid polymer (MCAP). In total, the methacrylates comprise 60-70% of the adhesive by weight. Other components include ethanol and water (23-28%), highly dispersed silicon dioxide (3-5%), initiators and stabilizers (3-5%).



Figure 11: Tetric N-Bond Universal

Ultra-Etch

A solution of 35% phosphoric acid used for etching tooth and resin composite surfaces.



Figure 12: Ultra-Etch

COJET Sand

This is a specially developed sand for coating all conventional dental materials such as composite, metal and ceramic surfaces intraorally during their repair. Consisting of silica coated aluminium oxide particles; the manufacturer claims a low abrasion rate of surfaces owing to its very fine particle (30 μ m) size (3M ESPE, 2000).



Figure 13: COJET Sand

During the coating process, the silicized particles are blasted onto the surface of the restoration with the silica anchoring to the material being repaired. This formation of a chemical bond using mechanical energy is known as tribochemistry. In this manner, the components of the blast coating agent (silica) are embedded into the substrate for a maximum depth of 15 μ m. When this surface is conditioned by the addition of a silane primer and then followed by the addition of a repair resin, a very strong bond results between the methacrylated monomer and the substrate being repaired (composite, metal or ceramic). Superior bond strengths have been demonstrated when COJET Sand is compared to conventional composite repair techniques (3M ESPE, 2000).

The steps followed in the fabrication of the samples and collection of data were as follows:

1. Fabrication of 240 composite cylinders using Filtek Supreme XTE .
2. Aging of 120 cylinders in artificial saliva at 37° C for 28 days.
3. Embedding of all cylinders in acrylic within PVC blocks.
4. Aged and non-aged composite cylinders divided randomly into six groups each, corresponding to the repair protocol applied.
5. Repair of the samples with either Filtek Supreme XTE or Tetric N-Ceram.
6. Shear bond strength testing of repaired samples.
7. Data collection (shear bond strengths) and analysis.



Sample preparation

Two hundred and forty composite cylinders were fabricated with the aid of a silicone matrix using Filtek Supreme XTE, with each cylinder having a diameter of 5mm and height of 5mm. To fabricate these samples, composite was applied into the silicone matrix and cured incrementally to a depth of 2mm until the full height of the sample was achieved. The curing was carried out using a light-emitting diode (LED) light with a power output 1000mW/cm² (Ellipar Freelight 2, 3M ESPE, St. Paul). The output of this light was verified prior to use with a Cure Rite Light Meter (Caulk, USA) and confirmed after fabrication of every 10 composite cylinders. All the composite cylinders were prepared by a single operator.

One hundred and twenty samples were randomly selected and allocated to the ‘non-aging’ (group A) while the remainder (n=120) were allocated to the ‘aging’ (group B). The samples for aging were then immersed into a solution of artificial saliva (Table 3) which was manufactured according to the method described by van der Bijl & de Waal (1994) and inoculated with *Streptococcus mutans* (1 X 10⁶ CFU) and *Lactobacilli acidophilus* (1 X 10⁶ CFU).



Figure 14: Composites cylinders undergoing aging in artificial saliva within an incubator

Table 3: Composition of artificial saliva. Adapted from van der Bijl & de Waal (1994).

Component	Weight in grams or volume in mL
Carboxymethylcellulose	9.0g
Potassium chloride	1.2g
Sodium chloride	0.84g
Magnesium chloride hexahydrate	0.06g
Calcium chloride dihydrate	0.16g
Dipotassium phosphate	0.34g
Sorbitol solution (70%)	42.80g
Methyl p-Hydroxybenzoate	2.0g
Solution of egg yellow (1%)	2.0g
Alcohol	2.5ml
Oil of lemon	0.4g
Distilled water	1000ml

The pH of this solution before inoculation with the bacteria was 6.7. Following this, the 120 samples were stored in an incubator for 28 days at 37° C (Figure 14). The non-aged composite cylinders were stored at room temperature in a dark storage area for 48 hours before repair.

All the composite cylinders within each of the two groups were embedded in acrylic resin within PVC pipes (Figure 15). They were then divided into twelve different sub-groups representing six different surface preparation protocols in each group (A1-A6 consisting of the non-aged composite cylinders and B1-B6 consisting of the aged cylinders). Thus, each of the 12 sub-groups had 20 composite cylinders. The six surface preparation protocols are described below.

1. Roughening with a medium-grit diamond bur what type (MANI Inc., Tochigi, Japan) and application of Scotchbond 1XT to the roughened surface.
2. Roughening with a medium-grit diamond bur what type (MANI Inc., Tochigi, Japan), application of 35% phosphoric acid (Ultra Etch) for 20 seconds, cleaning and drying with a water and air spray. Application of a single layer of Scotchbond 1XT using a microbrush after surface was completely dry.
3. Cleaning of the surface with a water and air spray for 5 seconds. Application of a single layer of Scotchbond Universal onto the dried surface using a microbrush.
4. Surface cleaning with a water and air spray. Application of a single layer of Tetric N-Bond Universal onto the dried surface using a microbrush.
5. Cleaning of the surface with an air-water spray. Application of COJET Sand for 5 seconds with the tip of the microblaster (Rocatec Junior, 3M ESPE) oriented perpendicular to the composite surface at a distance of 2mm. The pressure of the microblaster was set at 2 bar in conformity with the instructions from the manufacturer of COJET Sand (3M ESPE).
6. No surface treatment. For group A with the non-aged samples, this set represented the positive control while group B (aged samples) was used as the negative control.



Figure 15: 5mm X 5mm composite cylinders after embedding in acrylic resin

After the test samples were subjected to their respective surface treatments, repair of all two hundred and forty specimens was carried out using fresh composite. Within each subgroup of 20 samples (A1 to A6 and B1 to B6), 10 samples each were repaired by the addition of Filtek Supreme XTE and the remaining 10 samples with Tetric N-Ceram. The intention of this part of the study was to assess if repairing composite fracture with the same composite or a different composite from the original fractured composite would make a difference to the bond strength of the repaired composite. In this case all samples were prepared with Filtek composites bases and then half the group was repaired with the same material i.e. Filtek and the other half was repaired with a different composite i.e. Tetric N-Ceram. The objective of this step was to simulate the clinical conditions as quite often dentists repairing a fractured composite may not know what the original fractured composite material is.

All the repair composites were applied in 2mm increments using a silicon mould of 3mm diameter, resulting in a height of 4mm for the repair composite (Figure 16). The repair interface was also therefore standardized to 3mm of bonded surface area to the bottom composite base. The purpose of this step was to determine whether using a brand of

composite that was different from the original restoration had any effect on the repair bond strength. Clinically, the brand of composite used for the initial restoration may be unknown to the practitioner and a different type of composite might be used for the repair.

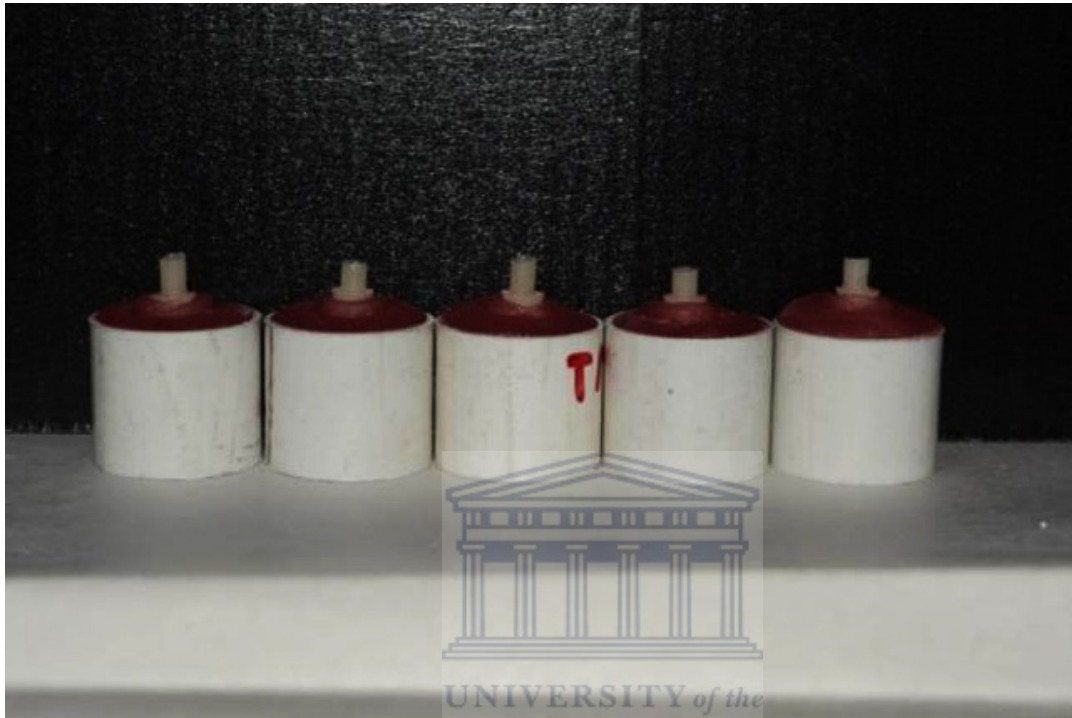


Figure 16: Repaired composite cylinders

The samples were then mounted on a jig and a shear bond test of the repair interface carried out using a universal testing machine (Zwick Roell International, Germany). For the purpose of the shear bond test, the blade was oriented perpendicular to the long axis of the composite block as close to the repair interface as possible (Figure 17). A force was then applied onto the blade at a crosshead speed of 0.5mm/minute until fracture of the composite occurred. The shear bond test was carried out in accordance with the recommendations on bond strength testing by Heintze (2013).

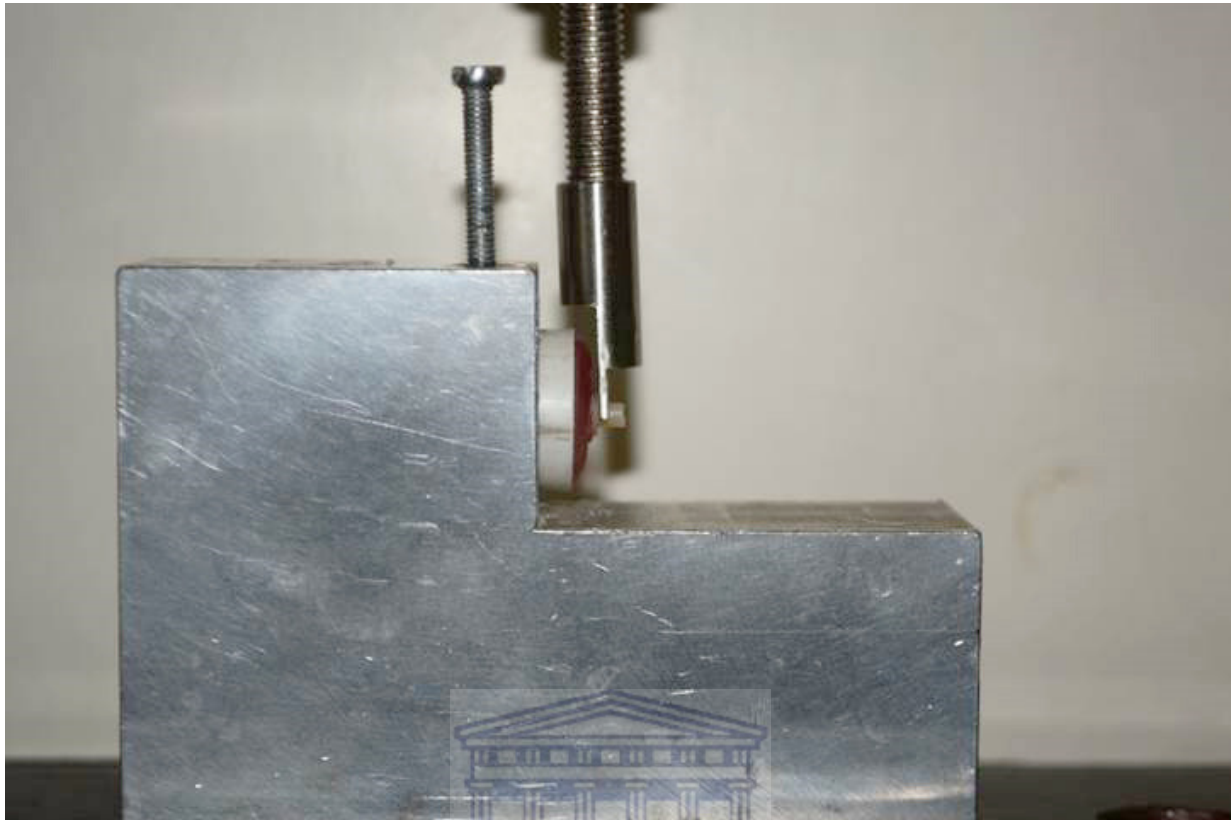


Figure 17: Shear bond strength testing



Data acquisition and analysis

The force at breaking point was then measured in megapascals (MPa) for each sample and recorded on a data collection sheet. The data obtained was then subjected to statistical analysis using computer software, IBM SPSS Statistics version 20 (IBM Corporation, Armonk, U.S.A.).

The effects of each preparation protocol on the shear bond strengths were compared using two-way Analysis of Variance (ANOVA). Post hoc testing was used to determine where the specific differences between the groups lay. The significance level was set at 0.05.

CHAPTER 5

RESULTS

The mean shear bond strengths (SBS) from each of the two groups; Group A (non-aged) and Group B (aged) composite samples are summarized in Tables 4 and 5 respectively. The raw data from all two hundred and forty test samples is presented in Appendix 2.

For the results of the effect of different repair protocols and type of repair composite on the shear bond strength, all 120 samples from Group B were analysed together with a positive control (Table 6 and Figure 18). In this case, the positive control served as an indicator of the cohesive strength of the composite being repaired.

For the effects of the aging in artificial saliva on the shear bond strength, the combined mean SBS values of the samples in group A were compared to the means of the corresponding samples in group B (Table 7 and Figure 19). There was a statistically significant difference between the aged and non-aged group with the non-aged group showing higher mean bond strength values ($p < 0.05$ ANOVA).

Table 4: Group A, non-aged samples: Mean shear bond strength (SBS) in megapascals (MPa) of non-aged composites. SD = standard deviation from the mean.

Group	Total	Repair protocol	SBS (Filtek Supreme XTE)	SD	SBS (Tetric N-Ceram)	S.D.
A1	20	Bur roughening + Scotchbond 1XT	18.59	4.82	16.67	3.17
A2	20	Bur roughening + phosphoric acid + Scotchbond 1XT	15.85	4.67	16.81	2.18
A3	20	Scotchbond Universal	19.05	2.70	17.74	1.65
A4	20	Tetric N-Bond Universal	18.16	4.58	21.55	2.46
A5	20	No surface preparation or adhesive	19.45	3.57	18.26	2.70
A6	20	COJET Sand + Scotchbond Universal	18.02	3.70	18.24	3.40

For the non-aged composites, when Filtek Supreme XTE was used as the repair composite, the highest shear bond strength (19.45MPa) was observed in the positive control group in which no intermediate adhesive was used (Table 4). This value was taken as the cohesive strength of Filtek Supreme XTE for the purpose of this study. With Tetric N-Ceram, the highest shear bond strength (21.55MPa) was observed when its own recommended bonding agent was used i.e. Tetric N-Bond Universal was used as the intermediate adhesive.

The lowest bond strength for Filtek Supreme XTE was 15.85MPa and was observed in group A2 in which the surface of the composite was first roughened by a diamond bur followed by etching with 35% phosphoric acid and the application of a single layer of Scotchbond 1XT. The lowest bond strength for Tetric N-Ceram was 16.67MPa and this was measured in group A1 where the surface was roughened by a diamond bur and a single layer of Scotchbond 1XT applied.

Table 5: Group B, aged samples: Mean shear bond strength (SBS) in megapascals (MPa) of aged composites. SD = standard deviation from the mean.

Group	Total	Repair protocol	SBS (Filtek Supreme XTE)	SD	SBS (Tetric N-Ceram)	S.D.
B1	20	Bur roughening + Scotchbond 1XT	14.17	1.28	14.83	1.11
B2	20	Bur roughening + phosphoric acid + Scotchbond 1XT	14.75	1.87	15.09	1.93
B3	20	Scotchbond Universal	15.45	1.39	17.83	2.06
B4	20	Tetric N-Bond Universal	15.55	3.06	17.35	1.49
B5	20	No surface preparation or adhesive	6.52	2.08	6.89	2.32
B6	20	COJET Sand + Scotchbond Universal	19.71	3.10	20.83	2.87

With the aged composite cylinders, the highest shear bond strength (SBS) occurred in group B6 for both Filtek Supreme XTE and Tetric N-Ceram i.e. when both samples groups were treated with COJET Sand and Scotchbond Universal.

These SBS values were 19.71MPa and 20.83MPa respectively (Table 5).

The lowest bond strengths in the aged group also occurred in group B5 where there was neither surface preparation nor application of an adhesive. Therefore, for the purpose of this study, this group was used as the negative control. For Filtek Supreme XTE in this group, the SBS was 6.52MPa while for Tetric N-Ceram it was 6.89MPa.

Effectiveness of different repair protocols on the shear bond strength of composite resin

For this part of the study, the mean shear bond strength from each of the five repair protocols were compared with a negative and a positive control to determine the most effective method of repair (Table 6 and Figure 18). In this case, Groups B1-B6 consisted of the aged composites while the positive control from Group A5 was non-aged, with no surface preparation.

For both repair composites, the highest mean bond strength was observed in the COJET Sand/ Scotchbond Universal group. When using Filtek Supreme XTE, the repair bond strength of 19.71MPa was achieved (Table 6). There was no statistical difference when this value was compared with the cohesive strength of the non-aged composite (19.45MPa) (Two way ANOVA, $p>0.05$).

With Tetric N-Ceram, the repair bond strength in the COJET Sand + Scotchbond Universal group was 20.83MPa. This was greater than the cohesive strength between the Filtek Supreme XTE and Tetric N-Ceram (18.26MPa). This difference was statistically significant (Two way ANOVA, $p=0.01$)

The lowest SBS values (14.17MPa for Filtek Supreme XTE and 14.83MPa for Tetric N-Ceram) were obtained from group B1 in which the composite cylinders were roughened by passing a diamond bur across the top surface followed by application of a single layer of Scotchbond 1XT (Figure 18).

The highest bond strength (19.71Mpa for Filtek Supreme XTE and 20.84MPa for Tetric N-Ceram) was observed following repair using COJET Sand in conjunction with Scotchbond Universal. The repair bond strength obtained after repair with COJET Sand and Scotchbond

Universal was statistically comparable to the substrate's cohesive strength, regardless of the repair composite used.



Table 6: Shear bond strength (MPa) of aged specimens following repair with Filtek Supreme XTE and Tetric N-Ceram. SD = Standard deviation from the mean.

Group	Repair protocol	Number	Filtek Supreme XTE	SD	Tetric N-Ceram	SD
B1	Bur roughening + Scotchbond 1XT	20	14.17	1.28	14.83	1.11
B2	Bur roughening + phosphoric acid + Scotchbond 1XT	20	14.75	1.87	15.09	1.93
B3	Scotchbond Universal	20	15.45	1.39	17.83	2.06
B4	Tetric N-Bond Universal	20	15.55	3.06	17.35	1.49
B5	Negative control	20	6.52	2.08	6.89	2.32
B6	COJET Sand + Scotchbond Universal	20	19.71	3.10	20.83	2.87
A5	Positive control	20	19.45	3.57	18.26	2.70

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Use of phosphoric acid etching did not appear to improve the repair bond strength. There was no significant difference between Groups B1 and B2 which had identical surface treatments except for the application of phosphoric acid in Group B2 (Two way ANOVA, $p>0.05$).

Repair with both universal adhesives resulted in larger bond strength values than when Scotchbond 1XT was used (Figure 18). For Scotchbond Universal, the mean repair bond strength with Filtek Supreme XTE was 15.45MPa. This value was not statistically significant from those obtained when Scotchbond 1XT was used in groups B1 and B2 (Two way ANOVA, $p=0.08$).

When Tetric N-Ceram was used in conjunction with Scotchbond Universal, the repair bond strength was 17.83MPa which statistically, was significantly greater than the values obtained when the same composite was used together with Scotchbond 1XT (Two way ANOVA, $p=0.01$).

With Tetric N-Bond Universal, the repair bond strength was 15.55Mpa for Filtek Supreme XTE. There was no statistically significant difference between the SBS from this group and groups B1 and B2 in which Scotchbond 1XT was used (Two way ANOVA, $p=0.08$). For Tetric N-Ceram, the mean SBS with Tetric N-Bond Universal 17.35Mpa. This value was significantly greater than when Tetric N-Ceram was used as the repair composite in groups B1 and B2 (Two way ANOVA, $p=0.001$).

Scotchbond Universal and Tetric N-Bond Universal were equally effective (ANOVA, $p > 0.05$) when used as repair adhesives with either repair composite (Figure 18).

Influence of repair composite on shear bond strength

Overall there was no significant difference between the two composites used for both aged and non-aged repair protocols (Two way ANOVA, $p=0.06$).

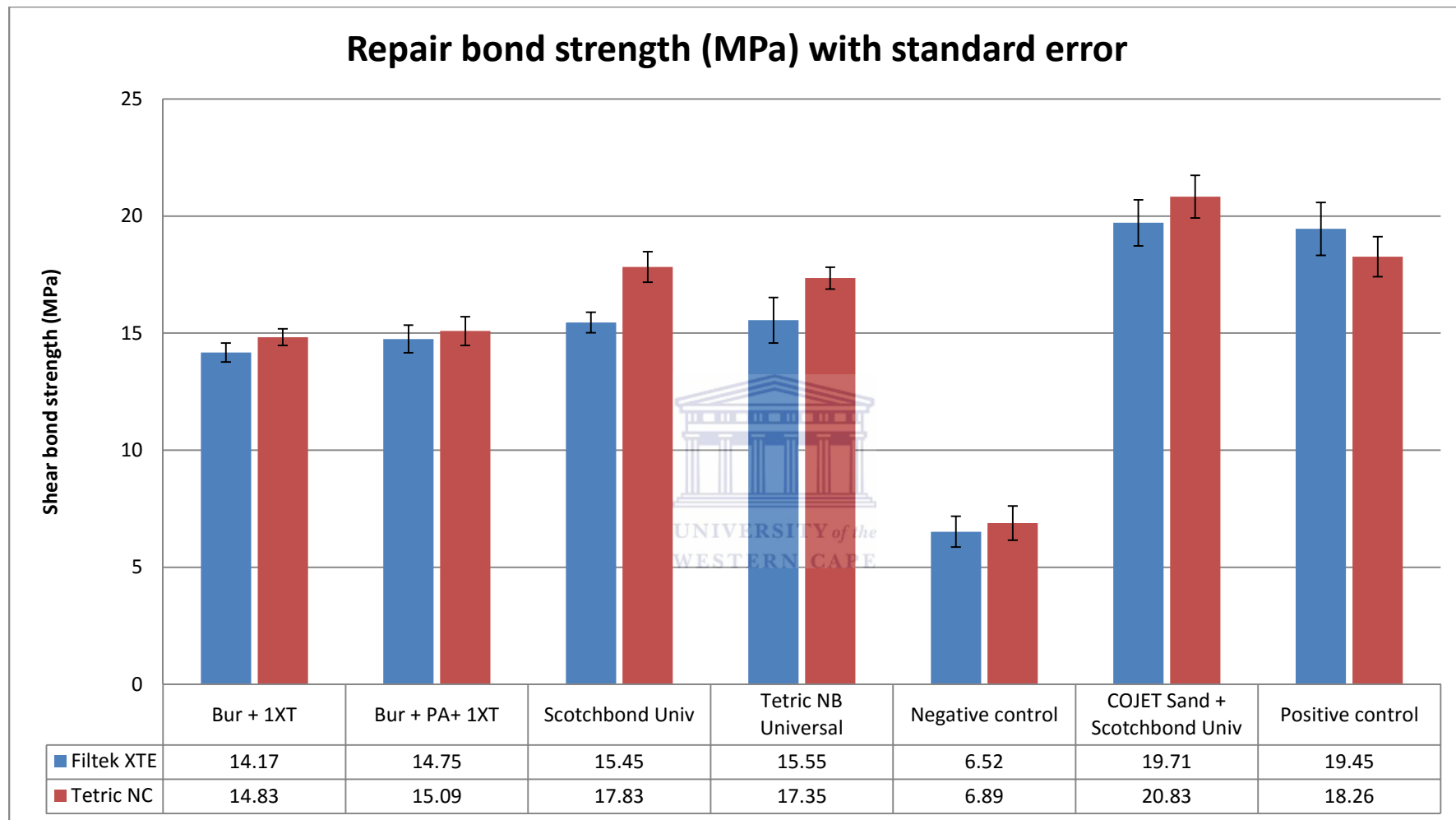


Figure 18: Repair bond strength following application of different surface preparation protocols

Effect of aging in artificial saliva on the repair bond strength

Table 7 highlights the differences between shear bond strength values obtained from aged and non-aged composite samples. When the combined mean SBS of the non-aged (18.20MPa) and aged (14.91MPa) was compared the shear bond strength of aged composites was 18.08 % lower than the non-aged samples.

Table 7: A comparison of the mean repair bond strength between non-aged and aged composite

Repair protocol		Mean	Std. Deviation	N
Bur roughening + PA + Scotchbond 1XT	Aged	14.92	2.24	20
	Non-aged	16.33	3.58	20
Bur roughening + Scotchbond 1XT	Aged	14.50	1.29	20
	Non-aged	17.63	4.09	20
COJET Sand + Scotchbond Universal	Aged	20.27	3.12	20
	Non-aged	18.13	3.46	20
No preparation	Aged	6.70	2.27	20
	Non-aged	18.85	3.30	20
Scotchbond Universal	Aged	16.64	2.16	20
	Non-aged	18.39	2.28	20
Tetric N-Bond Universal	Aged	16.45	2.64	20
	Non-aged	19.85	3.98	20
Total (aged and non-aged)	Aged	14.91	4.73	120
	Non-aged	18.20	3.59	120
	Total			240

The bond strength values of the non-aged composites had a narrow range of between 16.33 and 19.85 MPa (Figure 19). On the other hand, the aged samples showed a much wider variation, with results ranging from as low as 14.50 MPa to a maximum of 20.27 MPa.

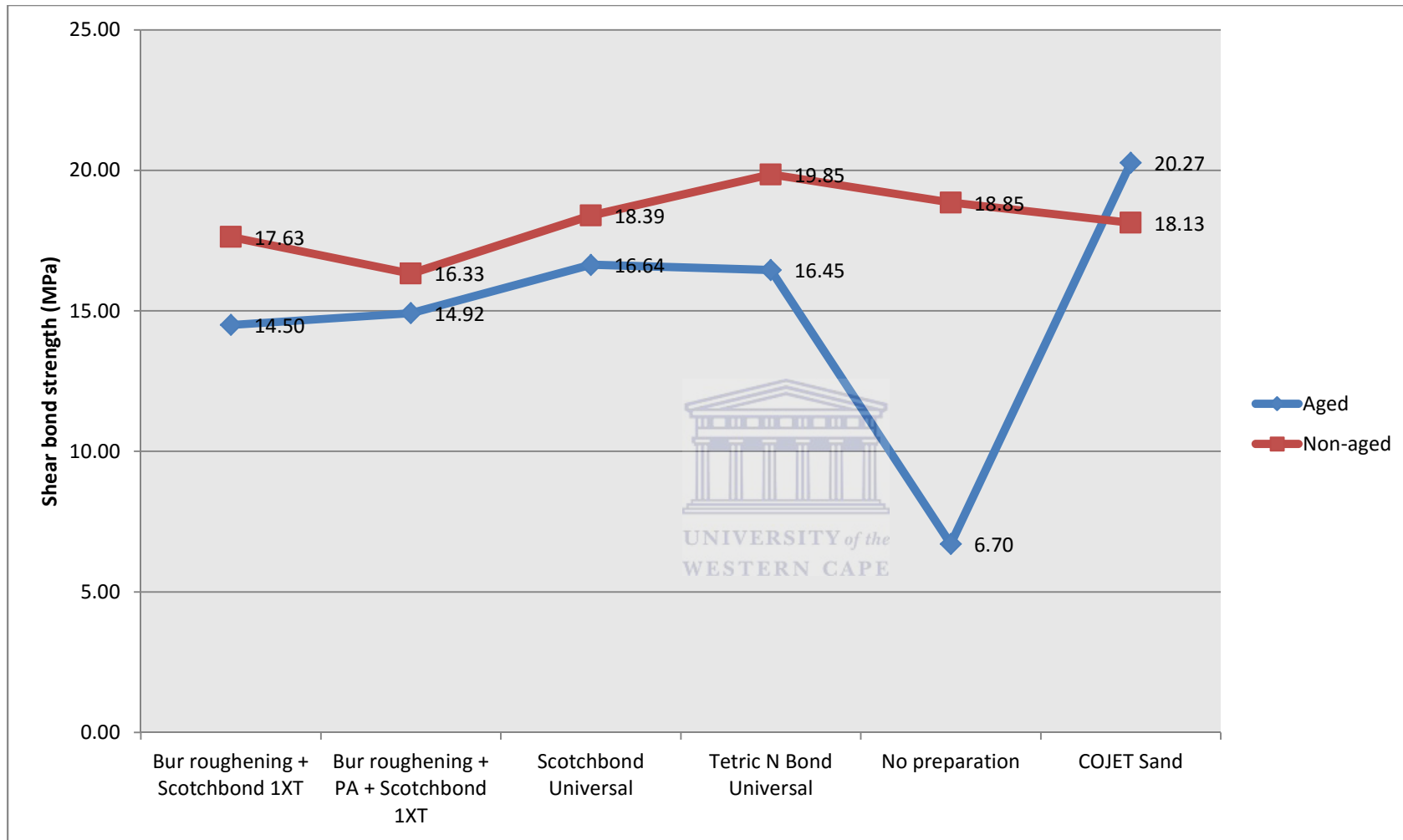


Figure 19: Effect of aging on the repair bond strength of composite samples

CHAPTER 6

DISCUSSION

From the results, it was evident that surface treatment is a crucial step during repair that helps to create a strong bond between aged and fresh composite. When no adhesive or surface modification was applied, the shear bond strength (6.7Mpa) was found to be about 46.2 % lower than the least effective adhesive repair technique (14.5Mpa, Figure 19). Due to the changes in the surface morphology and chemical composition of the aged resin composite, the bonding of a fresh composite onto this surface is compromised in the absence of an intermediate adhesive and surface treatment.

Artificial saliva as an aging medium

In many of the studies on composite repair, researchers tended to use plain water with or without thermocycling for the purpose of artificially aging composite blocks. Only in the study by Rinastiti *et al.* (2010) was an artificial saliva biofilm used to simulate the clinical aging of resin composite. They reported that filler particle exposure occurred to a lesser degree when an artificial biofilm was used compared to aging by thermocycling or immersion in citric acid. In a laboratory study to determine the effect of different repair protocols, it seems logical to age the composites in saliva first followed by the application of the repair protocol. This would more accurately simulate the clinical situation.

In this study, it was found that in general, the repair strength of aged samples was less than that of the corresponding non-aged composites except in the COJET Sand group. The decreased bond strength may be due to a number of factors.

First, acids produced by bacteria in the artificial saliva may induce softening and surface swelling of the composite with a subsequent reduction in surface roughness (Asmussen,

1984). Secondly, water sorption may lead to degradation of the resin matrix and reduce residual monomer availability for bonding to fresh composite.

Another finding was that blasting with COJET Sand produced the most effective method of cleaning this biofilm to produce an effective adhesive interface. This is in agreement with Rinastiti *et al.* (2010) who reported that application of a silica coating from COJET Sand was the best method to condition composite restorations prior to repair.

Effect of phosphoric acid etching on the repair bond strength

Previously, it has been proposed that phosphoric acid etching of a composite restoration may dissolve glass filler particles on the composite resin and leave a roughened surface that promotes adhesion of a fresh composite (Anusavice *et al.*, 2012). In this study, the use of phosphoric acid on the surface of the aged composite prior to application of an adhesive layer did not appear to have any effect on the repair bond strength. Furthermore, phosphoric acid for etching also contains thixotropic silica particles which increase its viscosity. After acid etching and rinsing, these particles may be left behind on the composite surface and compromise the repair bond strength (Loomans *et al.*, 2011b). Clinically, not using phosphoric acid may also have the advantage of saving chairside time since an intermediate adhesive may be applied without acid etching and obtaining better bond strengths.

In a study on the effect of different acids on the surface topography of aged resin composites, Loomans *et al.* (2011b) showed that phosphoric acid on its own had no effect on the surface roughness. However, when phosphoric acid was applied in conjunction with hydrofluoric acid, the surface roughness was greater than that of the individual acids.

Nevertheless, the filler particles in the specific composite may contribute to the material's resistance to etching. For instance, Filtek Supreme XTE has a high percentage of zirconia and

silica nanoparticles which are resistant to acid etching. Tetric N-Ceram does contain some barium glass filler particles but it appears that their quantity is not high enough for the overall effect of phosphoric acid etching to be observed.

Phosphoric acid may play a role in cleaning the surface of a restoration owing to its effect of removing surface impurities (Fawzy *et al.*, 2008). For instance, a composite surface may be contaminated by a smear layer consisting of hydroxyapatite. Clinically, this effect is observed in the process of repairing a restoration which involves cutting of enamel and dentine. The significance of this effect was not tested in this study as only composite blocks, rather than composite restorations within teeth were used. Therefore, it can be recommended that when repairing a restoration, phosphoric acid should be applied only to the tooth structure and not on the remaining part of the composite.

Universal adhesives in the repair of composites

Universal adhesives are a new class of adhesive materials that are aimed at providing an all-in-one product for multiple applications, including dentine bonding and repair of composite restorations. Scotchbond Universal contains a silane coupling agent that is claimed by the manufacturer to aid in the chemical adhesion to composite (3M ESPE, 2013). Tetric N-Bond Universal has no silane component but comprises silicon dioxide filler particles that are also meant to promote adhesion to the resin matrix (Ivoclar Vivadent, 2015).

In this study, Scotchbond Universal (SU) was compared to a different brand of universal adhesive (Tetric N-Bond Universal) and a sixth generation adhesive (Scotchbond 1XT). It was observed that there was no significant difference in the shear bond strength between the SU and phosphoric acid/ Scotchbond 1XT groups and Tetric N-Bond Universal groups (ANOVA, $p=0.29$) when repairing the aged composite.

All three adhesives used in this study were equally effective in the repair of composites, in spite of the fact that Scotchbond Universal and Tetric N-Bond had a different composition from Scotchbond 1XT. This may be due in part to the change in the chemical and physical composition of the aged composite. Tetric N-Bond Universal which contains silicon dioxide may assist in creating additional bonding to the MDP and silane coupling agent. Decandiol dimethacrylate hydrophobic crosslinking dimethacrylate (D3MA), a constituent of Tetric N-Bond Universal, enables the reaction of the adhesive with the less polar monomers of the filling or luting composite

COJET Sand in conjunction with Scotchbond Universal

The most effective repair protocol observed was the use of COJET Sand in conjunction with Scotchbond Universal. The mean SBS for COJET Sand with Scotchbond Universal was significantly greater than all the other repair protocols (Two way ANOVA, $p=0.001$) regardless of the repair composite used and compare favourably with the cohesive strength of the substrate.

COJET Sand consists of aluminium oxide particles with an average diameter of 30 μm which are coated with a layer of silica. When these particles are air-blasted onto the RBC surface, the silica coating is chemically anchored onto the restoration to a depth of about 15 μm (Figures 20-22). The composite surface modified in this way is then conditioned by addition of a silane or silane-containing adhesive such as Scotchbond Universal. This creates a chemical bond similar to that observed in the resin composites, where the filler particles are bound to the resin matrix by a silane coupling agent (Figure 23). The result is a very strong bond between the old and new composites which is mediated by both micromechanical and chemical adhesion. This process, the creation of chemical bonds by the use of mechanical energy as applied with COJET Sand, is referred to as tribochemistry (3M ESPE, 2000).

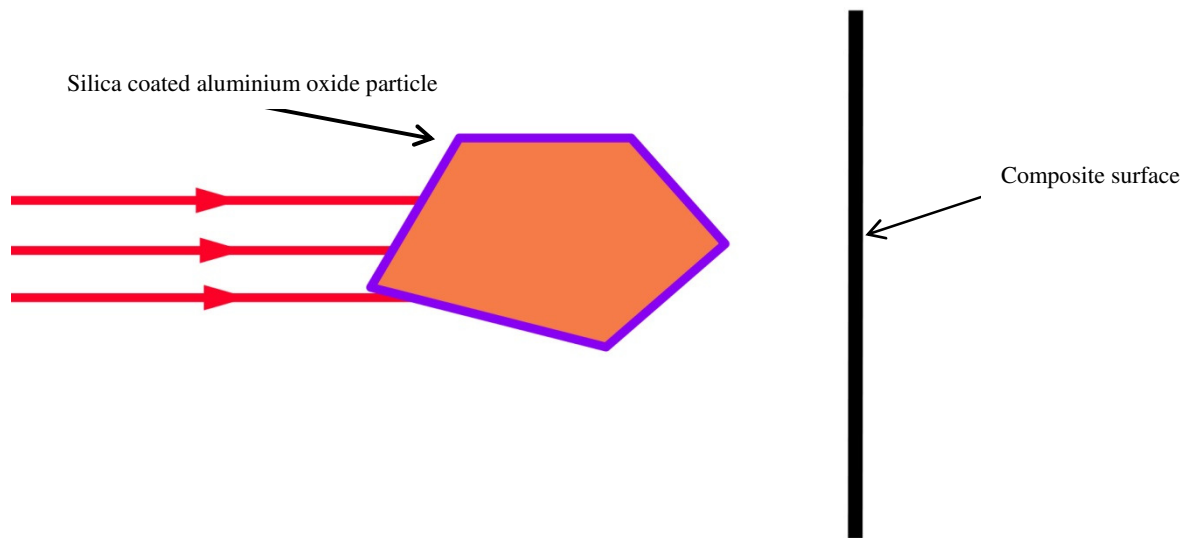


Figure 20: Tribochemical coating during microblasting

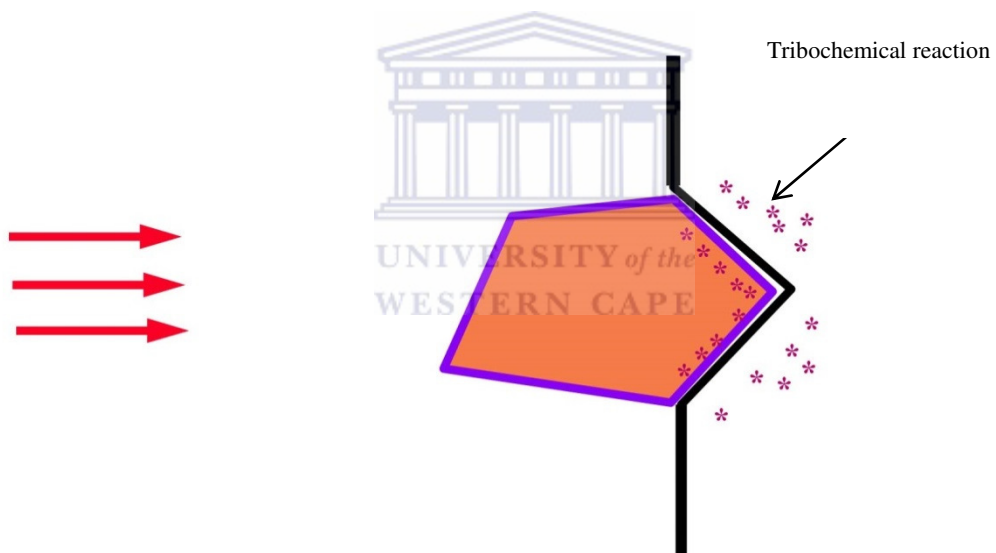


Figure 21: Tribochemical coating on impact

Silica layer deposited on the composite surface

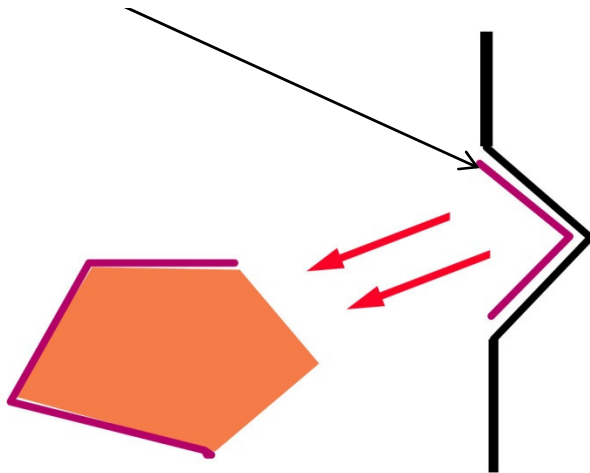


Figure 22: Tribochemical coating (silicized surface of composite).

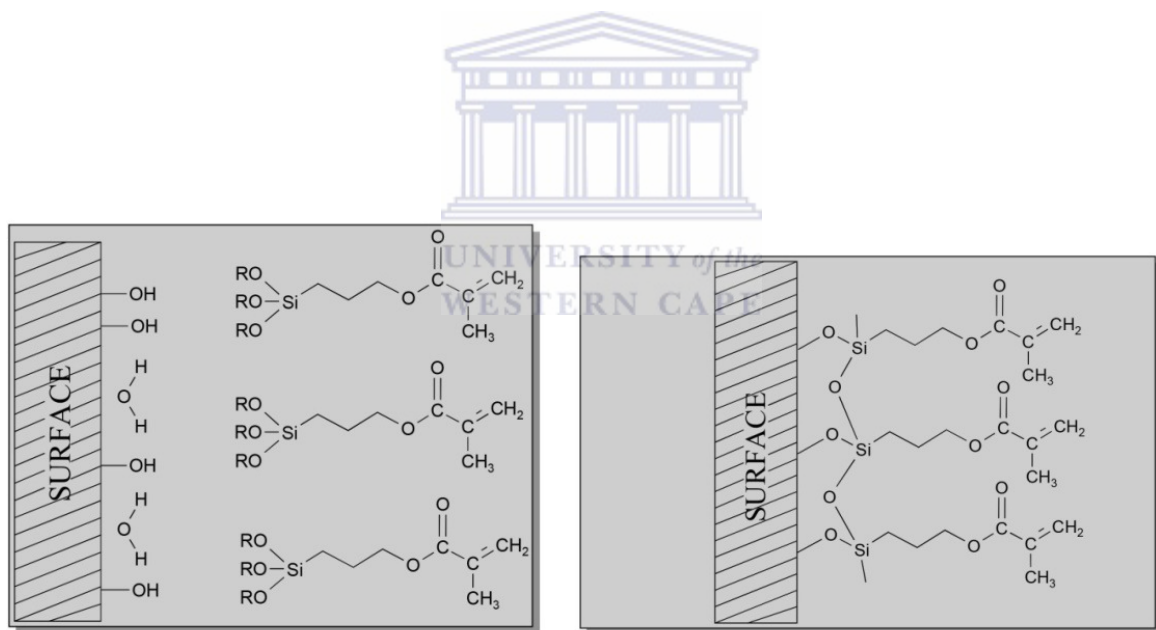


Figure 23: Silanization reaction.

Effect of repair composite on the shear bond strength

Overall, in the repair of aged composite, there was no difference in the repair bond strengths for both materials Filtek Supreme XTE and Tetric N-Ceram. Therefore, repairing a damaged composite restoration with a different composite material does not seem to affect its bond strength.

Both these composites incorporate bis-GMA, UDMA and bis-EMA in their resin matrices. The main difference in their composition lies in the type of filler particles used, with Filtek Supreme XTE composed of nanoscale zirconia and silica particles, some of which are aggregated in 'nanoclusters'. On the other hand, Tetric N-Ceram contains barium glass, ytterbium trifluoride, mixed oxide and silicon dioxide filler particles. However, the differences in the types of filler particles in the two materials did not seem to have a significant effect on the repair bond strength. It appears, therefore, that the primary bonding mechanism occurs between the resin matrices of composites; the fillers within the repair composite may not play a significant role in maintaining the repair interface.

These finding suggests that, clinically, the type of composite used to repair a restoration may be less important than the repair protocol applied. In other words, a restoration can be successfully repaired without the practitioner having prior knowledge of the brand of the existing composite restoration.

Limitations of the study

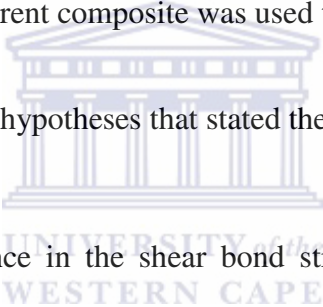
1. Although aging the specimens in artificial saliva offers the most realistic in vitro scenario, the findings may not exactly mimic the intraoral behaviour of composite restorations. The systematic review by Bayne (2012) showed that for resin based composites, the in vitro results of shear bond strength testing do not always correlate to the clinical performance and longevity of the same materials. This may be due to the large variety of clinical factors that influence material longevity and the difficulty in replicating all of them in vitro. Should an in vivo study be conducted, a randomized clinical trial may be a more effective method of evaluating the effectiveness of different repair protocols.
2. The study evaluated only one aspect of the chemical degradation of composite restorations i.e. the use of saliva and bacteria. In addition, the effects of temperature variation and acids present in food were not considered in this study. Within the oral environment, the restoration is subjected to both mechanistic and chemical degradation simultaneously. These two factors work in tandem in the breakdown of the restoration but the effects of this complex interaction were not taken into account in this study.
3. Based on the results obtained from the COJET Sand in aged composite samples, the use of Tetric N-Bond Universal would have provided a good comparison to the COJET Sand + Scotchbond Universal protocol.

CHAPTER 7

CONCLUSIONS

In this in vitro study, different repair protocols were evaluated and compared for their effectiveness when composite was aged in artificial saliva. The influence of aging the composite blocks in artificial saliva was also evaluated. The differences in the shear bond strength between the five repair protocols were found to be statistically significant. COJET Sand used in conjunction with Scotchbond Universal produced the best repair bond strength. In addition, there was a significant reduction in the bond strengths when composite blocks were aged as compared to the non-aged samples. There was no difference in the bond strengths when the same or a different composite was used to repair the composite.

In view of these findings, the null hypotheses that stated the following were rejected:

- 
1. There will be no difference in the shear bond strength when five different repair protocols are applied to aged composite specimens.
 2. Aging the composite cylinders in artificial saliva has no effect on the shear bond strength following repair.

The following null hypothesis was not rejected:

1. There is no difference between the shear bond strength when either Filtek Supreme XTE or Tetric N-Ceram is used as the repair composite.

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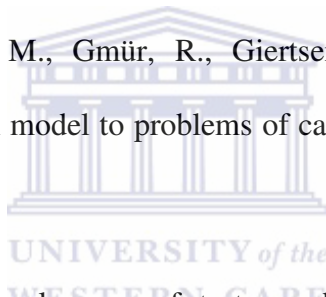
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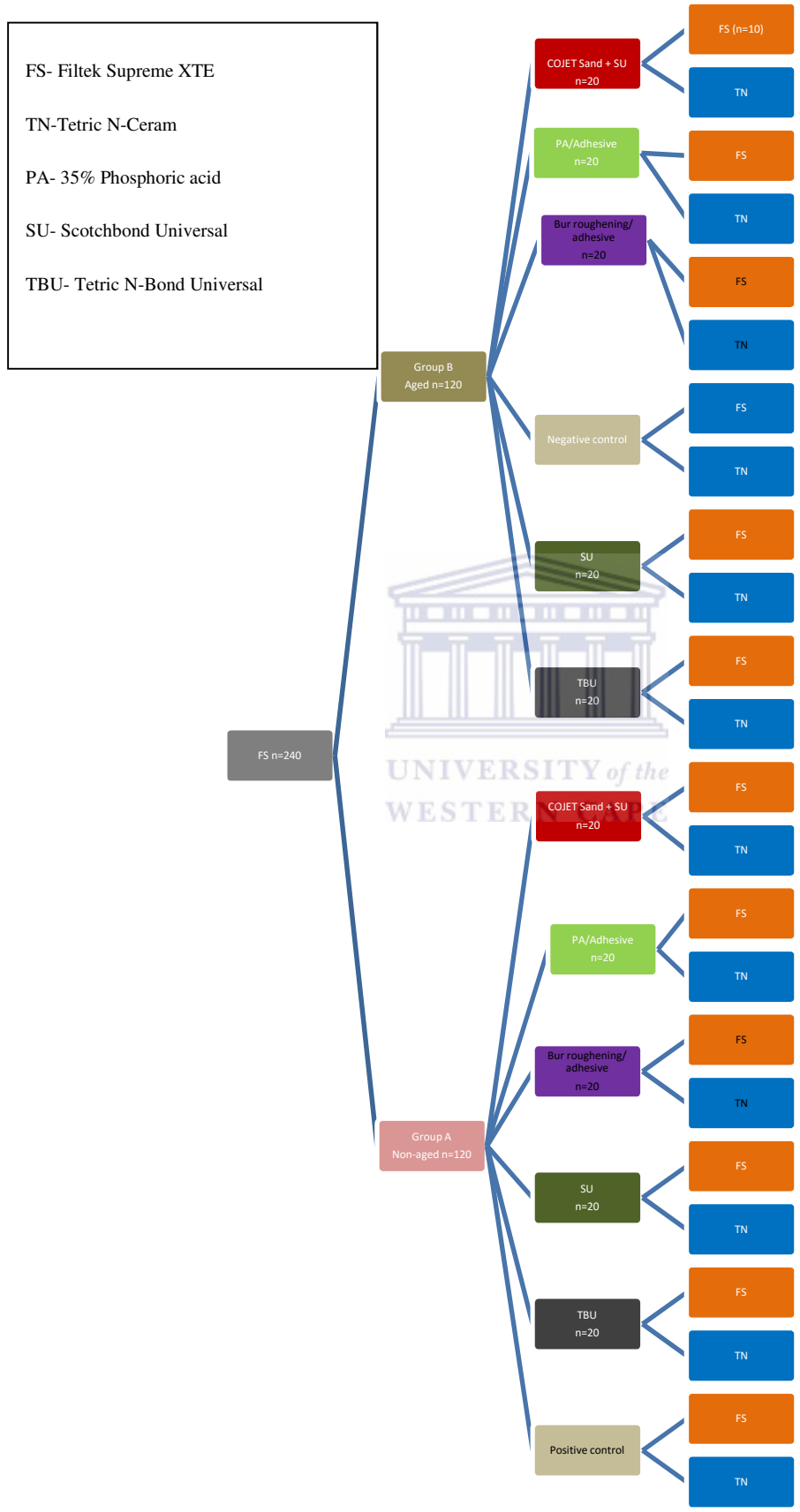
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APPENDIX 1: FLOWCHART FOR SPECIMEN PREPARATION



APPENDIX 2: Raw data of shear bond strength from 240 composite repair samples

Repair protocol	Repair composite	Shear bond strength (MPa)	Aged or non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	12.82	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	15.48	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	16.15	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	12.42	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	13.68	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	13.43	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	13.42	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	14.88	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	13.57	Aged
Bur roughening + Scotchbond 1XT	Filtek XTE	15.82	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.05	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.59	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	13.59	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.29	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	15.79	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.04	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	15.34	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	13.30	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	17.18	Aged
Bur roughening + Scotchbond 1XT	Tetric NC	16.15	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	15.69	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	17.35	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	14.36	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	10.83	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	18.12	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	11.91	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	16.62	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	12.79	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	16.88	Aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	12.93	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	17.43	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	16.55	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	12.24	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	14.72	Aged

Bur roughening + PA + Scotchbond 1XT	Tetric NC	14.50	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	18.10	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	13.29	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	15.49	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	16.20	Aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	12.37	Aged
Scotchbond Universal	Filtek XTE	18.69	Aged
Scotchbond Universal	Filtek XTE	16.52	Aged
Scotchbond Universal	Filtek XTE	15.59	Aged
Scotchbond Universal	Filtek XTE	14.85	Aged
Scotchbond Universal	Filtek XTE	14.34	Aged
Scotchbond Universal	Filtek XTE	15.75	Aged
Scotchbond Universal	Filtek XTE	15.36	Aged
Scotchbond Universal	Filtek XTE	13.86	Aged
Scotchbond Universal	Filtek XTE	15.24	Aged
Scotchbond Universal	Filtek XTE	14.27	Aged
Scotchbond Universal	Tetric NC	18.73	Aged
Scotchbond Universal	Tetric NC	17.92	Aged
Scotchbond Universal	Tetric NC	21.76	Aged
Scotchbond Universal	Tetric NC	17.35	Aged
Scotchbond Universal	Tetric NC	19.95	Aged
Scotchbond Universal	Tetric NC	16.19	Aged
Scotchbond Universal	Tetric NC	16.14	Aged
Scotchbond Universal	Tetric NC	19.63	Aged
Scotchbond Universal	Tetric NC	15.50	Aged
Scotchbond Universal	Tetric NC	15.11	Aged
Tetric N-Bond Universal	Filtek XTE	9.80	Aged
Tetric N-Bond Universal	Filtek XTE	15.76	Aged
Tetric N-Bond Universal	Filtek XTE	18.70	Aged
Tetric N-Bond Universal	Filtek XTE	19.35	Aged
Tetric N-Bond Universal	Filtek XTE	15.58	Aged
Tetric N-Bond Universal	Filtek XTE	16.28	Aged
Tetric N-Bond Universal	Filtek XTE	11.45	Aged
Tetric N-Bond Universal	Filtek XTE	19.61	Aged
Tetric N-Bond Universal	Filtek XTE	14.06	Aged
Tetric N-Bond Universal	Filtek XTE	14.94	Aged
Tetric N-Bond Universal	Tetric NC	18.10	Aged
Tetric N-Bond Universal	Tetric NC	16.98	Aged
Tetric N-Bond Universal	Tetric NC	16.67	Aged
Tetric N-Bond Universal	Tetric NC	17.16	Aged
Tetric N-Bond Universal	Tetric NC	18.88	Aged

Tetric N-Bond Universal	Tetric NC	14.93	Aged
Tetric N-Bond Universal	Tetric NC	19.35	Aged
Tetric N-Bond Universal	Tetric NC	19.49	Aged
Tetric N-Bond Universal	Tetric NC	15.65	Aged
Tetric N-Bond Universal	Tetric NC	16.26	Aged
Aging no prep (negative control)	Filtek XTE	9.12	Aged
Aging no prep (negative control)	Filtek XTE	5.86	Aged
Aging no prep (negative control)	Filtek XTE	5.29	Aged
Aging no prep (negative control)	Filtek XTE	3.57	Aged
Aging no prep (negative control)	Filtek XTE	4.89	Aged
Aging no prep (negative control)	Filtek XTE	10.55	Aged
Aging no prep (negative control)	Filtek XTE	6.89	Aged
Aging no prep (negative control)	Filtek XTE	8.45	Aged
Aging no prep (negative control)	Filtek XTE	5.14	Aged
Aging no prep (negative control)	Filtek XTE	5.42	Aged
Aging no prep (negative control)	Tetric NC	3.59	Aged
Aging no prep (negative control)	Tetric NC	5.72	Aged
Aging no prep (negative control)	Tetric NC	7.28	Aged
Aging no prep (negative control)	Tetric NC	11.21	Aged
Aging no prep (negative control)	Tetric NC	5.73	Aged
Aging no prep (negative control)	Tetric NC	7.90	Aged
Aging no prep (negative control)	Tetric NC	3.58	Aged
Aging no prep (negative control)	Tetric NC	7.16	Aged
Aging no prep (negative control)	Tetric NC	6.81	Aged
Aging no prep (negative control)	Tetric NC	9.92	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	23.92	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	19.28	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	19.72	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	20.97	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	19.12	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	14.86	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	16.09	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	16.51	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	24.71	Aged
COJET Sand + Scotchbond Universal	Filtek XTE	21.88	Aged
COJET Sand + Scotchbond Universal	Tetric NC	20.65	Aged
COJET Sand + Scotchbond Universal	Tetric NC	19.59	Aged
COJET Sand + Scotchbond Universal	Tetric NC	21.69	Aged
COJET Sand + Scotchbond Universal	Tetric NC	26.78	Aged
COJET Sand + Scotchbond Universal	Tetric NC	20.66	Aged
COJET Sand + Scotchbond Universal	Tetric NC	17.61	Aged
COJET Sand + Scotchbond Universal	Tetric NC	21.34	Aged
COJET Sand + Scotchbond Universal	Tetric NC	22.28	Aged

COJET Sand + Scotchbond Universal	Tetric NC	22.32	Aged
COJET Sand + Scotchbond Universal	Tetric NC	15.36	Aged
No aging no prep (positive control)	Filtek XTE	16.91	Non-aged
No aging no prep (positive control)	Filtek XTE	20.62	Non-aged
No aging no prep (positive control)	Filtek XTE	21.32	Non-aged
No aging no prep (positive control)	Filtek XTE	20.81	Non-aged
No aging no prep (positive control)	Filtek XTE	21.55	Non-aged
No aging no prep (positive control)	Filtek XTE	18.83	Non-aged
No aging no prep (positive control)	Filtek XTE	14.2	Non-aged
No aging no prep (positive control)	Filtek XTE	26.02	Non-aged
No aging no prep (positive control)	Filtek XTE	13.39	Non-aged
No aging no prep (positive control)	Filtek XTE	20.82	Non-aged
No aging no prep (positive control)	Tetric NC	18.86	Non-aged
No aging no prep (positive control)	Tetric NC	17.73	Non-aged
No aging no prep (positive control)	Tetric NC	24.36	Non-aged
No aging no prep (positive control)	Tetric NC	14.74	Non-aged
No aging no prep (positive control)	Tetric NC	21.12	Non-aged
No aging no prep (positive control)	Tetric NC	16.51	Non-aged
No aging no prep (positive control)	Tetric NC	18.3	Non-aged
No aging no prep (positive control)	Tetric NC	18.21	Non-aged
No aging no prep (positive control)	Tetric NC	17.84	Non-aged
No aging no prep (positive control)	Tetric NC	14.9	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	17.65	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	19.54	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	11.46	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	16.3	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	25.16	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	27.83	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	18.32	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	18.87	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	16.51	Non-aged
Bur roughening + Scotchbond 1XT	Filtek XTE	14.29	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	12.46	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.33	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	23.59	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	17.34	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	15.26	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	17.71	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	19.35	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	14.08	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	17.17	Non-aged
Bur roughening + Scotchbond 1XT	Tetric NC	15.42	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	9.38	Non-aged

Bur roughening + PA + Scotchbond 1XT	Filtek XTE	23.29	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	17.73	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	13.01	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	11.6	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	14.43	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	17.37	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	23.1	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	12.34	Non-aged
Bur roughening + PA + Scotchbond 1XT	Filtek XTE	16.2	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	18.4	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	19.33	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	15.32	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	15.94	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	19.39	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	18.51	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	16.86	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	12.61	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	14.88	Non-aged
Bur roughening + PA + Scotchbond 1XT	Tetric NC	16.81	Non-aged
Scotchbond Universal	Filtek XTE	20.38	Non-aged
Scotchbond Universal	Filtek XTE	18.63	Non-aged
Scotchbond Universal	Filtek XTE	19.6	Non-aged
Scotchbond Universal	Filtek XTE	19.74	Non-aged
Scotchbond Universal	Filtek XTE	15.27	Non-aged
Scotchbond Universal	Filtek XTE	24.15	Non-aged
Scotchbond Universal	Filtek XTE	20.58	Non-aged
Scotchbond Universal	Filtek XTE	14.83	Non-aged
Scotchbond Universal	Filtek XTE	19.7	Non-aged
Scotchbond Universal	Filtek XTE	17.65	Non-aged
Scotchbond Universal	Tetric NC	17.23	Non-aged
Scotchbond Universal	Tetric NC	17.21	Non-aged
Scotchbond Universal	Tetric NC	14.93	Non-aged
Scotchbond Universal	Tetric NC	18.77	Non-aged
Scotchbond Universal	Tetric NC	18.91	Non-aged
Scotchbond Universal	Tetric NC	19.84	Non-aged
Scotchbond Universal	Tetric NC	19.99	Non-aged

Scotchbond Universal	Tetric NC	15.79	Non-aged
Scotchbond Universal	Tetric NC	17.6	Non-aged
Scotchbond Universal	Tetric NC	17.08	Non-aged
Tetric N-Bond Universal	Filtek XTE	10.98	Non-aged
Tetric N-Bond Universal	Filtek XTE	18.83	Non-aged
Tetric N-Bond Universal	Filtek XTE	15.79	Non-aged
Tetric N-Bond Universal	Filtek XTE	23.02	Non-aged
Tetric N-Bond Universal	Filtek XTE	24.24	Non-aged
Tetric N-Bond Universal	Filtek XTE	16.81	Non-aged
Tetric N-Bond Universal	Filtek XTE	23.86	Non-aged
Tetric N-Bond Universal	Filtek XTE	18.6	Non-aged
Tetric N-Bond Universal	Filtek XTE	17.22	Non-aged
Tetric N-Bond Universal	Filtek XTE	12.25	Non-aged
Tetric N-Bond Universal	Tetric NC	24.01	Non-aged
Tetric N-Bond Universal	Tetric NC	21.84	Non-aged
Tetric N-Bond Universal	Tetric NC	21.53	Non-aged
Tetric N-Bond Universal	Tetric NC	22.4	Non-aged
Tetric N-Bond Universal	Tetric NC	18	Non-aged
Tetric N-Bond Universal	Tetric NC	18.24	Non-aged
Tetric N-Bond Universal	Tetric NC	21.59	Non-aged
Tetric N-Bond Universal	Tetric NC	21.88	Non-aged
Tetric N-Bond Universal	Tetric NC	19.84	Non-aged
Tetric N-Bond Universal	Tetric NC	26.14	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	12.56	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	17.74	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	12.71	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	22.53	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	22.71	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	18.95	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	18.96	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	21.2	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	14.68	Non-aged
COJET Sand + Scotchbond Universal	Filtek XTE	18.19	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	11.61	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	16.33	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	22	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	17.5	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	18.25	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	15.73	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	19.43	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	18.87	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	18.71	Non-aged
COJET Sand + Scotchbond Universal	Tetric NC	24	Non-aged



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