ASSESSMENT OF THE DENTIN BOND STRENGTH VALUES OF A RESIN-MODIFIED GLASS IONOMER RESTORATIVE MATERIAL USING DIFFERENT *IN VITRO* TEST METHODS

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ABSTRACT

Anmar Adnan Kensara: Assessment of the Dentin Bond Strength Values of Resin Modified Glass Ionomer Restorative Material using Different *In Vitro* Test Methods
(Under the direction of Ching-Chang Ko and Lee Boushell)

Objective: To assess whether the *in vitro* dentin bond strength values of a resin-modified glass ionomer restorative material (RMGI) are affected by different *in vitro* test methods.

Methods: Mid-depth occlusal dentin of 36-extracted human third molars free of defects was exposed and finished with wet 600-grit silicon carbide paper for 10s. A commercially-available RMGI (Fuji II LC, GC America) was applied to all specimens according to manufacturer's instructions, after which specimens were stored in 100% humidity at 37 °C for 24 h. Specimens were then randomly divided into three different test groups (n=12): shear bond strength (SBS), microtensile bond strength (μTBS), and four-point bending bond strength (4PBBS). Specimens were loaded to failure using universal testing machines and test-specific parameters: Instron for SBS and 4PBBS tests, EZ-Test for the μTBS test. The mode of bond failure (adhesive, cohesive or mixed) was qualitatively assessed with optical stereomicroscopy. Data were analyzed using one-way ANOVA and descriptive statistics.

Results: There was a statistically significant difference between bond strength values for the different test methods ($p<0.05$). The mean bond strength values (± SD, in MPa) were 15.7 (±7.1) for SBS, 9.7 (±5.3) for μTBS and 37.3 (±12.8) for 4PBBS. With respect to the

mode of failure, most SBS failures were adhesive in nature (83%), while the majority of μTBS and 4PBBS failures were mixed (69% and 47% respectively). Several μTBS and 4PBBS specimens failed during processing (before testing).

Conclusion: The *in vitro* dentin bond strength values of a resin-modified glass ionomer material are greatly affected by the test method. The mode of bond failure is also affected by test method. The SBS test method demonstrated the highest percentage of adhesive failure and proved to be less technique sensitive. The majority of μTBS and 4PBBS failures were mixed. Use of the μTBS and 4PBBS may not be optimal laboratory test methods for comparison of the relative bond strength of RMGI materials to dentin. Use of the SBS test may allow more controlled comparison of the adhesive dentin bond among various RMGI formulations, whether already commercially available or under development.

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1. Introduction

Different types of direct restorative materials are used in routine dental practice. The most common are amalgam, resin composites, and glass-ionomer (GI) restorative materials. Amalgam has a proven, long-term successful clinical performance record, is inexpensive and easy to handle.[62] The main drawback of dental amalgam is its poor esthetics and the concern about mercury vapors. Resin-based composites are esthetic materials with satisfactory physical properties. However, material cost and technique sensitivity, along with increased risk of the development of secondary caries (compared with amalgam), may be considered as relative disadvantages of this material.[62] Glass ionomer (GI) materials that can bind with calcium ions of mineral tissues are generally tooth colored and may be used in a wide range of clinical applications.

The clinical application of GI materials depends on the ratio of powder/liquid, which can be identified through the following classification:[55]

Type I: Luting cement with a low powder content.

Type II: Restorative material with high powder content and therefore improved physical properties.

Type III: Cavity lining material (low powder content), or cavity base material (high powder content) with physical properties enabling its use as a dentin substitute.

Glass Ionomer Cement (GIC) is the generic name for the dental restorative material that is formed through an acid-base reaction of fluoroaluminosilicate (FAS) glass (powder) and aqueous polyacrylic or polycarboxylate acid (liquid).[1] Wilson and Kent introduced GIC to

the dental market in the early 1970s.[2] Dental researchers and clinicians have an ongoing interest in GICs because these materials 1) are water based and are able to bond to a moist surface, 2) are able to develop a stable chemical bond with the mineral phase of tooth structure, 3) contain fluoride ions that are released over time with a resultant decrease in the critical pH of immediately adjacent tooth structure, 4) are able to absorb topically applied fluoride for subsequent release 5) are able to seal the cavity and resist microleakage and 6) are biocompatible and not irritating to the pulp.[3,6,57,60,61,70] However, poor mechanical properties such as, brittleness (decreased fracture toughness) and low wear resistance, as compared with composite resin restorative materials, limit their use in dentistry as a permanent filling material in stress-bearing areas.[52] Moreover, GICs have been found to be sensitive to moisture contamination during initial set, are sensitive to subsequent desiccation and have poor polishability.[3,61,64] Modifications in the composition of GICs have resulted in the steady improvement of material properties over time. The addition of light-polymerizable resins to GICs, now generally referred to as resin modified glass ionomer (RMGI) formulations, has been found to improve mechanical/physical properties of the materials without compromising the bond to the mineral phase of tooth structure.[5] The most notable physical property of GI material is the ability to form a stable, adhesive bond with hydroxyapatite. The strength and durability of this bond has been evaluated in the laboratory and by means of clinical trials.

The *in vitro* research studies note that the relative bond strength value, obtained by various test methods, is not considered an inherent material property of a restorative material but is helpful in comparison of various dental material formulations.[36] Laboratory methods used to quantify the strength of the adhesive attachment of RMGI to tooth structure include the Shear

Bond Strength (SBS) test and the Microtensile Bond Strength (μTBS) test. A recently developed laboratory method, referred to as the Four-Point Bending Bond Strength (4PBBS) test, has been used to assess the adhesive attachment of composite resin materials to tooth structure but has not been used to evaluate the strength of RMGI materials to tooth structure.[10,14]

It has been reported that SBS tests may be inadequate for the evaluation of the restorative material bond strength to dental hard tissues as shear stress is not uniformly distributed along the interface. SBS tests result in increased tendency for cohesive RMGI failures.[11,12,50,65] Cohesive RMGI failures represent the inherent strength of the material and, therefore, the measured SBS is not a good representative of interface bond strength between the dentin and the restorative material.[9- 12]

Micro-Tensile Bond Strength tests are able to assess a tensile force at the adhesive interface, which may more closely mimic clinical forces.[9] Other potential advantages of μTBS tests over SBS tests include ability to measure the restorative bond to various regions in dentin as well as the production of more specimens from a given tooth.[8] However, μTBS tests have been found to be laborious and technique-sensitive. One particular draw back of the μTBS test is the potential for the bonded specimen (especially when GIC materials are being tested) to break apart before testing (Pre-test failures). Furthermore, some studies reported cohesive fractures in the RMGI materials being tested.[15,16]

Recent studies have suggested use of the 4PBBS test as a more optimal means of assessing dentin bond strength of dental restorative materials in general.[10,14] The reason for this suggestion is that all specimens tested failed adhesively (i.e. none failed cohesively in dentin or in the dental material) which may allow better assessment of the strength of the

interface.[10,14] The materials tested in the 4PBBS studies where composite resin in nature. It was observed that bond failures were immediately proceeded by fracture at the interface. The authors suggested that this means of bond failure may provide more accurate information about the material behavior at the adhesive interface.[10] However, currently there are no studies in the literature that have utilized the 4PBBS test to evaluate the bond strength of a RMGI to dentin.

2. Objectives

The objectives of this study were to assess whether the *in vitro* dentin bond strength values of a resin-modified glass ionomer restorative material (RMGI) are affected by different in vitro test methods and to assess the modes of bond failure for each type of test.

2.1 Specific Aims:

2.1.1 Primary Aim:

To assess the dentin bond strength values of a RMGI restorative material using SBS, μTBS and 4PBBS tests.

2.1.2 Secondary Aim:

To use optical stereomicroscopy to qualitatively assess the mode of bond failure (adhesive, cohesive or mixed) for each specimen tested.

2.2 Null Hypotheses:

Primary Null Hypothesis: There is no difference in the mean dentin bond strength values of a RMGI restorative material when using SBS, μTBS and 4PBBS tests.

Secondary Null Hypothesis: There is no difference in the mode of dentin bond failure of a RMGI restorative material when using SBS, μTBS and 4PBBS tests.

3. Literature Review

3.1 Resin Modified Glass Ionomer (RMGI) Restorative Material

Clinical limitations of early GICs have forced the development of new hybrid GI materials (see section 3.1.3). Resin monomers (as well as initiators necessary to affect resin polymerization) have been added to these materials in various formulations. As such, these particular GICs are referred to as Resin-Modified Glass Ionomer (RMGI) materials.[5]

3.1.1 Setting Reaction of RMGI Materials:

Glass-ionomer materials have a slow acid-base setting reaction with resultant sensitivity to contamination and desiccation and a need for delayed finishing.[40] The setting process occurs by reaction of an aqueous polyacrylic acid with finely powdered fluoroaluminosilicate glass (Figure 1).[41] RMGI materials, in addition to the acid-base reaction, undergo polymerization as a result of exposure to visible light. The polymerization reaction occurs through interaction of water-soluble resin monomers and methacrylate groups attached to the glass-ionomer acid chains (Figure 2).[40] Some manufacturers (Fuji II LC, GC America) report additional polymerization of HEMA molecules, thus claiming a triple-curing mechanism. Others (Vitremer, 3M ESPE) report a separate setting reaction which is initiated by oxidation and reduction catalysts in the material. This "self-curing" aspect of the RMGI may help to ensure complete cure of the material even in areas that curing light energy cannot reach.[49] Furthermore, ongoing polymerization may occur following exposure to the curing light, as in composite resins. Polymerization of RMGI continues for up to 24 hours after visible light activation ceases.[49]

Light activation allows a longer working time and shorter setting time than are possible with conventional glass-ionomer materials, which results in placement and finishing procedures that are less complex.[40,42]

3.1.2 Adhesion of RMGI Materials:

Glass-ionomers are generally considered to be the only materials that self-adhere to tooth structure.[6,7,19] Clinical protocol recommends that the tooth cavity be pretreated by weak acid solution, Polyacrylic acid (PAA), which removes surface debris (commonly referred to as the "smear layer"), and effects a partial demineralization of the cavity walls, without extensive surface hydroxyapatite removal. This increases the surface area available for chemical interaction of the polyacrylic acid with residual hydroxyapatite (enamel and dentin) and exposes collagen for subsequent micro-mechanical interlocking (hybridization), [6,19] (Figure 3 A).

The adhesion of RMGIs to dentin surfaces is considered twofold:[6] 1) a calcium chelation bond with the calcium hydroxyapatite phase of both dentin and enamel.[7] This chemical bond may be paly a role in the resistance to hydrolytic degradation.[18] 2) Micro-mechanical interlocking of RMGI with the fibril network of the matrix results in a shallow hybrid layer, about .5-1 micrometers deep.[6,43] (Figure 3 B)

3.1.3 Properties of RMGI Materials:

RMGIs have been developed to overcome the limitations of early GIC formulations.[5] Even though there are individual differences from brand to brand, RMGIs generally exhibit 1) greater early flexural strength and diametral tensile strength,[52] 2) less sensitivity to variation in levels of moisture[55] 3) improved finishability,[3] 4) improved translucency,[3] and 5) increased bond strength as compared with conventional GICs.[49,61] Furthermore, RMGIs

release fluoride over extended periods of time.[61] However, when compared with composite resin based restorative materials, RMGIs have much less wear resistance, less rigidity, and are less esthetic.[53] Although RMGIs material have higher coefficient of thermal expansion over conventional GICs, their thermal expansion compares favorably to tooth structure.[61]

Most RMGI formulations contain 2-hydroxyethyl methacrylate (HEMA), which is a hydrophilic component. The presence of HEMA causes these materials to act like a mild hydrogel with rapid water uptake over the first 5-7 days after placement. The uptake of water results in a small amount of expansion of the restoration and increased risk of staining from diet related pigments.[56] Therefore it is recommended that newly placed RMGIs be covered immediately with a layer of varnish or light-activated unfilled resin material.[59]

3.2 Evaluation of the Bond Strength of Dental Materials

3.2.1 Clinical Studies:

Randomized, controlled clinical trials are considered the ultimate means by which to collect scientific evidence on the effectiveness of restorative treatment.[4,9,18] Clinical studies assessing the durability of restorative material adhesive interfaces have primarily focused on restoration of non-carious cervical lesions (NCCLs) of the human dentition. Reasons for use of NCCLs include: 1) little to no macro-mechanical retention is naturally present, 2) margins of the that are located in enamel as well as dentin, 3) natural occurrence is wide spread in the population, 4) reasonable access for restoration and evaluation procedures and 5) minimum or no requirement for preparation prior to restoration. [9,18,38] The presence of various levels of dentinal sclerosis (hypermineralized dentin) in NCCLs may complicate development of the adhesive interface.[18,45]

Many external variables influence the outcome of clinical studies. Among these are patient related factors such as age, oral hygiene, occlusal loading, intra oral temperature and the degree of dentin sclerosis.[18,38,46] Other variables include operator skill, specific restorative material properties, type and effectiveness of the light curing unit, type and effectiveness of isolation, and finishing methods and instrumentation. [18] Interpretation of clinical findings (i.e. restorative outcomes of relative clinical success or failure) must consider all known variables. Identification of which variable(s) contributed most to clinical outcomes may be difficult.[18] The best clinical performance with regard to retention in NCCLs has so far been achieved by glass ionomer restorative materials with an average annual failure rate of 1.9%.[73]

3.2.2 Laboratory Bond Strength Studies:

The goal of laboratory bond strength testing is to measure the amount of force required to separate two bonded materials in a way that is clinically relevant. This quantification of the strength of the interface configuration depends on use of an appropriate test machine[9]. The *in vitro* assumption is that the higher the actual bonding capacity of a material, the better it will clinically withstand similar force vectors i.e. the longer the restoration will survive *in vivo*.[4]

In general, laboratory bond strength tests have many practical advantages over the clinical studies. These advantages include, (1) testing is relatively quick and easy, (2) better control of study variables, (3) ability to directly compare the performance of a new and/or experimental material/technique with that of the current 'gold-standard', (4) ability to simultaneously test multiple materials within one study set-up and (5) relatively low financial investment is required. [4]

Current laboratory test results show large variability among specimens being

evaluated with the same testing method. Experimental factors that influence the results of laboratory bond strength tests, include variation within a group of researchers at the same institution (intra-institute variability) and between different research institutions (inter-institute variability). Reported intra-institute bond strength values varied between 30 and 50% and inter-institute bond strength values varied between 20 & 40 %.[8,9] Additional variability in reported bond strengths may result from the adhesive system being evaluated, the origin of the substrate (bovine or human), how the substrate has been prepared (which would include specifics such as bur type and bur speed) and even the flexural modulus of the dental material that has been bonded to the tooth substrate. Research teams use laboratory tests designed to measure the adhesive bond that occurs at the interface between the dental material and the tooth substrate. When the test results in bond separation at the interface it is referred to as an *adhesive failure*, which represents the best approximation of the strength of the adhesive bond to the substrate. However, testing does not always result in adhesive failure. If the prepared specimen separates within the substrate, then it is referred to as a *substrate cohesive failure*. If the prepared specimen separates within the restorative material, then it is referred to as a *restorative material cohesive failure*. Neither of the types of cohesive failure provide an appropriate approximation of the strength of the adhesive bond. At times both adhesive and cohesive failures occur in a single specimen, which is referred to as a *mixed failure*. It is necessary for researcher to carefully note and report the various types and relative percentages of adhesive, cohesive and mixed failures.

The presence of experimental variables, and the resultant lack of consistent test results, severely limits direct comparison of the reported absolute bond strength test values.[18] However, well designed bond strength testing, performed at multiple different test sites, may

be useful in predicting minimal levels of clinical performance of a test material. For example, an adhesive that performed poorly in several independent laboratory studies was also found to be less clinically effective.[18] Ultimately, the objective of laboratory testing of bond strengths is to use carefully designed methods in such a way that prediction of eventual clinical outcomes becomes more reliable.[19]

The bond strength test methods that have been developed for dental materials are generally categorized based on the size of the bonded area under evaluation. The two basic categories are known as Macro- and Micro-bond strength tests.[4]

3.3 Macro-Bond Strength Tests:

Testing of any bonded area larger than $3mm^2$ is considered to be a Macro-bond strength test. This test is used in protocols designed to measure shear-, tensile- and push-out bond strengths.[4]

3.3.1 Shear Bond Strength (SBS) Test:

It has been reported that 46 % of bond strength studies utilize the SBS test.[9] The reason for its high popularity, as compared with other test methods, is the simplicity of specimen preparation and that no further processing is required after the adhesive interface has been created.[19] The SBS test applies a force designed to slide or twist one material across another, parallel to the interface (Figure 5).[1] This test has been criticized because forces applied to the interface result in non-uniform stresses within the interfacial zone that are unlikely to create pure shear stress. [9]

The SBS test results in a high percentage of restorative material cohesive failures, especially in the case of weak restorative materials. Current studies consistently report that shear stress is not uniformly focused and distributed across the interface.[20,21,36] Furthermore,

it has been reported that SBS testing of a bonded composite cylinder revealed that the bending moment resulted in compression on the side of the cylinder but failed to create adequate tensile forces at the interface.[20,21] Therefore, SBS test results depend on the physical properties of the material. SBS testing of materials with relatively low physical properties, or materials (with relatively high physical properties) that contain some type of flaw (or crack) will guide crack growth into the material that propagates toward a region of high stress near the base of the interface resulting in a cohesive failure.[36] However, if the strength of the adhesive interface is low enough, then the failure will begin at the interface in the region of maximum local stress. In another words, in the case of weak dental materials, the higher the adhesive bond strength, the higher the rate of cohesive failure.[4,36]

Effort have been made to standardized the SBS test protocol so that it is consistent with the ISO Technical Specification (TS) number 11405 entitled "Testing the adhesion to tooth Structure".[28] ISO TS 11405 requires restriction of the bonding area to a limited size. Therefore, in order to control the bonded surface area, specific jigs have been designed accordingly. These include the Ultradent jig (Ultradent, Salt Lake City, UT, USA) and the more recent SDI jig (SDI, Bayswater, Victoria, Australia).[4] Despite such standardization attempts, SBS testing is influenced by crosshead speed of the testing device and the means by which the shear force is actually applied to the specimen by wire loops, points and knife edges.[4] It has been found that SBS test results have a positive correlation with the modulus of elasticity of the restorative material. [27,36] Therefore, it is preferable to use the same dental material for all specimens when comparing the SBS of different adhesive interfaces.[9]

3.3.2 Tensile Bond Strength (TBS) Test:

The TBS test was first used for assessing adhesion of dental materials in by Bowen in 1965.[26] The force in the TBS test results in elongation of the bonded specimen.[1] The TBS test requires that the specimen aligned exactly perpendicular to the tensile force vector.[28] Misalignment of the specimen will result in flexure while under tension and the interfacial stress generated will not be a pure tensile stress.[1] Moreover, other variables, such as inconsistent geometry of the specimens or use of materials with different elastic moduli may lead to non-uniform interface stress application among the test specimens. [36]

3.3.3 Push-out Bond Strength(PoBS) Test:

The PoBS test uses a compressive force to push a material out of a ring made of another material. The force vector results in a shear stress at the interface.[23,37] In dentistry, this test has been used primarily for measuring the bond strength of posts luted to root dentin.[24,25] Complexities of specimen preparation and test methodology have limited the universal use of the PoBS test especially when compared to the traditional SBS testing process.[19]

3.4 Micro-Bond Strength Test:

Micro-bond strength tests utilize specimens that are approximately 1 mm X 1 mm in cross section. A reported advantage of testing a smaller specimen is that it more readily allows measurement of the restorative bond to various regions in dentin. In addition, a smaller specimen size may allow opportunity of increased test numbers per tooth. [19] Generally, it has been noted that the smaller the bonding surface, the higher the bond strength values.[8] Therefore it is essential that the type of bond strength test method be noted when considering the reported bond strength values. Types of Micro-bond strength test methods

include microtensile, micro-shear, fracture toughness, and four-point bending bond strength tests.

3.4.1 Microtensile bond strength (μTBS) Test:

The μTBS test was first reported by Sano and others in 1994. [13] Review of the current literature reveals that approximately 60% of research studies assessing adhesive bond strength used the μTBS test.[4,18] Improved design of the specimen mounting jig, which often has a toggle or freely rotating attachment, allows for tensile forces to be more accurately aligned with the long axis of the specimen.[1] The resultant alignment allows for better tensile stress distribution at the adhesive interface. The resultant mean bond strength values may allow for more accurate comparison and ranking of various adhesive systems. Those systems with consistently higher bond strengths may have a greater likelihood of clinical success. This is in contrast to the results of SBS testing which reveal more variation among studies. It may be that this is part of the reason why the findings of μTBS testing correlate more closely with the reported clinical retention rates of cervical restorations than those identified by SBS testing.[9] Indeed it has been reported that μTBS test results correlate more accurately with the clinical findings, which suggests that the μTBS test discriminates more effectively between different adhesive systems than SBS and other bond strength tests. [8,9]

As with any bond strength test, there are important factors to consider so as to obtain meaningful research findings. These factors include the geometry of the beam, type of jig, specimen trimming, loading speed, specimen alignment and specimen shape. [28,29,32,33]

Dental researchers at the University of Iowa have created a specimen former (the Iowa Micro-Specimen Former) which helps to ensure the tensile stress is maximally concentrated at the interface.[30] This device produces stick-type specimens that are

cylindrically constricted at the interface. It has been reported that the smaller bonding surface area reduces the development of cohesive failures (i.e. increases the tendency to development adhesive failures).[19] Hence, some have recommended that this μTBS-testing protocol become the standard for measure bonding effectiveness in the laboratory.[19]

3.4.2 Micro-shear Bond Strength (μSBS) test:

The μSBS test was introduced in 2002 by Shimada and others.[34,35] This test sought to benefit from the relative simplicity of the SBS while, at the same time, enable increased numbers of specimens per tooth.[4] It has been found that there are no advantages of the μSBS test over the macro-shear test. The reason is that the same problem of non-uniform stress distribution is still present and may be even more pronounced.[4] Specimen formation is more technically demanding as it is very difficult to confine the adhesive to the area that is to be tested.[4] In addition, the μSBS test results revealed similar percentages of the modes of failure as compared with μTBS test results, while, at the same time, the bond strength values detected were lower. [35]

3.4.3 Fracture Toughness (K_{IC}) Bond Strength Test:

The Fracture Toughness Bond Strength test was introduced in 1993 by Tam and Pilliar.[29] It has been used in bond strength studies to characterize the ability of the adhesive interface to resist further fracture propagation from a crack that has been artificially created.[29] Specimen preparation for this test is laborious, time consuming and technique sensitive. The test is particular problematic when testing materials, such as glass ionomer restorative materials, that are already prone to fracture.[4,64] It is not a routine test and any correlation to the retention rates of cervical restorations has not yet been established. Testing the fracture

toughness of the bonding interface may be regarded as a supplementary test to the macro-tensile test.[9]

3.4.4 Four-point Bending Bond strength (4PBBS) Test:

The 4PBBS test measures the net effect of simultaneous tensile, compressive, and shear stress on an adhesive interface.[1] The specimen to be tested must be positioned in the test jig so that a constant stress may be loaded at the central point of the beam which also must be the location of the adhesive interface.[1] Tensile stress concentrates at the interface on the lower aspect of the specimen beam. Compressive stress forms at the adhesive interface on the upper surface of the beam and a natural zone occurs in the center of the interface.[1,10] Additionally, a shear stress is produced close to the supporting ends of the specimen that does not play a significant role in the failure process.[1] Studies suggest that this test may provide more accurate information about the material behavior at the adhesive interface.[10,14]. Even so, this bond strength test has not been commonly used in dentistry to date. Currently there are no studies in the literature that have utilized the 4PBBS test to evaluate the bond strength of a RMGI to dentin.

3.5 Dentin Bond strength of RMGI Restorative Materials

SBS and μTBS test methods have traditionally been used to measure the strength of RMGI materials to dentin. [4,18] However, the failure mode of the SBS studies has been predominantly cohesive in the RMGI material. Therefore, the SBS values associated with cohesive failures are not representative of the interface bond strength between the RMGI and dentin.[11,12,50,65] This indicates that the bond strength of the RMGI material is higher than the inherent strength of this material.[11] Although the stress in μTBS testing is more uniformly distributed at the interface, as compared with SBS testing, the potential for the bonded

specimen to break apart before testing (pre-test failure) is high.[15,16] Furthermore, cohesive failures have still been reported in some μTBS studies.[15.16] High levels of variation in reported RGMI dentin bond strength values, as well as failure modes being predominantly cohesive in nature, suggests potential benefit from exploring the use of an alternative, more recently developed, testing strategy. Therefore, this study sought to assess the human dentin bond strength values of a RMGI restorative material (Fuji II LC, GC America) using SBS, μTBS and 4PBBS test methods.

4. Materials and Methods

The UNC Institutional Review Board determined that this study was not human subjects research and exemption from review was granted (IRB # 15-2558). A commercially available RMGI restorative material was obtained (Table 1). Freshly extracted, non-carious human third molars were obtained from the UNC Oral and Maxillofacial Surgery clinics and placed immediately in a 0.1% aqueous thymol solution and stored at a temperature of 2-5°C until preparation of the dentin surface. Crowns of 36 teeth were separated from the roots using an Isomet diamond saw (Buehler Ltd., Lake Bluff, IL, USA) under running water. The roots were sectioned and all pulp tissue was removed (Figure 4 A, B). The occlusal surfaces of the crowns were ground mechanically (Ecomet 3, Buehler Ltd.) under running water with 180-grit silicon carbide (SiC) paper to obtain a flat dentin surface. The exposed flat dentin surface was examined to be free of enamel using 2.5X magnification loupes (HiRes™ 2, Orascoptic, WI, USA). The dentin surface was then polished mechanically using 320-grit SiC and 600-grit SiC paper under running water for 10 s to create a standardized smear layer.[17] Specimens were then randomly assigned to SBS, μTBS and 4PBBS test methods in three groups of 12 specimen.

4.1 Shear bond strength (SBS) test:

The prepared dentin specimens were embedded in a block of polymethyle methacrylate acrylic resin (Great Lakes Orthodontics, NY, USA). The RMGI (Fuji II LC Capsule, GC America) was bonded to the dentin according to manufacturer's instructions (Table 1) using the Ultradent specimen former which includes a split Teflon mold (Figure

5A). The specimen former created one 2.38 diameter RMGI cylinder per tooth. Light-curing was accomplished using Demi plus (Kerr, CA, USA) for 20s with a light output 1200 mW/cm^2. Specimens then were stored in 100% humidity at 37°C for 24 hours. Shear bond strength tests were then performed using a model 4411 universal testing machine (Instron Corporation, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min using Ultradent notched crosshead contact (Figure 5B). Bond-strength values were calculated by dividing the peak break force (N) by the cross-sectional area of the bonded interface and were expressed in MPa units.

4.2 Micro tensile bond strength (μTBS) test:

The RMGI was applied to the dentin surface to a thickness of 5 mm following manufacturer instructions (Table 1). Specimens were then immediately stored in 100% humidity at 37°C for 24 hours. Specimens were then sectioned mesiodistally using an Isomet diamond saw (Buehler Ltd., Lake Bluff, IL, USA) under running water to obtain around 1 mm thick sections. The specimens were then further sectioned faciolingually to obtain 1 mm X 1 mm rods that were approximately 6 mm long (Figure 6A). The dentin-RMGI interface was located at the center of the rods. Each specimen was fixed to a Geraldeli Jig (EZ-Test, Shimadzu, Kyoto, Japan) using a cyanoacrylate-based adhesive (Figure 6B). The specimens were carefully placed on the jig so that the RMGI-dentin interface was exactly perpendicular to the long axis of the testing assembly. The microtensile bond strengths of all specimens were tested using a universal testing machine (EZ-Test, Shimadzu) with a crosshead speed of 0.5 mm/min. The bond strength of each specimen was determined as the failure load (N) divided by the cross-sectional area of the bonded interface and expressed in MPa units.

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4.3 ***Four-point Bending Bond Strength (4PBBS) test:***

The RMGI was applied to the dentin surface to a thickness of 5 mm following manufacturer instructions (Table 1). Specimens were then immediately stored in 100% humidity at 37°C for 24 hours. Specimens were then sectioned mesiodistally using an Isomet diamond saw (Buehler Ltd., Lake Bluff, IL, USA) under running water to obtain around 1 mm thick sections. The specimens were then further sectioned faciolingually to obtain 1 mm X 1 mm rods that were approximately 10 mm long. The dentin-RMGI interface was located at the center of the rods. A custom stainless steel device was fabricated such that the distance between loading points would be 1.8 mm for upper and 7.2 mm for lower members of the test device (Figure 7A). Prepared specimens were then subjected to the 4PBBS test and loaded to failure (Figure 7B). The test was performed using a model 4411 universal testing machine (Instron Corporation, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min. Bond strengths σb (in MPa), were computed using the standard relationship (ASTM E855/1984): $\sigma b = \frac{3Pa}{bh^2}$.

Where P is the maximum load (in N), a is the spacing (in millimeters) between upper and lower loading points, b and h are, respectively, the specimen width and thickness (in millimeters.

4.4 ***Qualitative Assessment of the RMGI - Dentin Fracture Interfaces***

After bond strength testing, all specimens were examined using optical stereomicroscopy (Nikon SMZ18, Tokyo, Japan), at 8X magnification for SBS and 13.5X magnification for μTBS and 4PBBS, to determine the mode of failure at the fracture interface. The number of "adhesive", "cohesive" and "mixed" failures in each test group was identified and reported as a percentage.

4.5 Statistical Analysis

The mean (+/- SD) SBS, μTBS and 4PBBS test values from each tooth (12 teeth per group) were calculated. Mean bond strengths from multiple beams from each tooth in the μTBS and 4PBBS were calculated so as to provide an average bond strength for each tooth. Specimens showing pre-test or cohesive dentin failures were excluded from the statistical analysis as these do not represent measurements of adhesion.[8] Bond-strength data were analyzed using one-way ANOVA. Data were further analyzed using the Tukey's post-hoc test. Modes of failure were analyzed using Pearson's Chi-squire test after taking into account the range of failures for each tooth that had more than one beam. All statistical tests were performed at the 95% confidence level. Stereomicroscopy images were analyzed and reported using descriptive statistics.

5. Results

The 12 teeth assigned to the SBS group provided 12 cylinders for SBS testing (Table 2). The 12 tooth specimens provided 61 beams for μTBS testing and 43 beams for 4PBBS testing respectively (Table 2). The number of pre-test failures for each test group is presented in Table 2 and Figure 8. Approximately 1/3rd of the beams prepared for μTBS and 4PBBS tests developed pre-test failures. The mean values and standard deviations were as follows: SBS = 15.7 +/- 7.1 MPa, μTBS = 9.7 +/- 5.3 MPa, 4PBBS = 37.3 +/- 12.8 MPa (Table 2 and Figure 9). There was a statistically significant difference between bond strength values of both SBS and μTBS tests and the 4PBBS test ($p<0.0001$). There was no statistically significant difference between SBS and μTBS test results ($p=.24$).

There was a statistically significant difference among the bond strength tests in the mode of failure ($p=.006$) (Table 3). Failure occurred predominantly at the adhesive/dentin interface for SBS (83%) while the majority of μTBS and 4PBBS failures were mixed (69% and 47% respectively). The 4PBBS test showed higher cohesive failure rate than the μTBS (33.3% and 11.1 respectively) (Figure 10). Only one beam (2.2%) of μTBS specimens failed cohesively in the dentin (Figures 10, 11 and 12).

6. Discussion

The present study evaluated the ability of three different test methods (shear-, microtensile-, and 4-point bending bond strength test) to determine the bond strength between a RMGI restorative material and human dentin. Efforts were made to limit intra-institutional variation in methods while conducting the study. Many studies can be found in the literature that compare different bond strength methodologies using dentin as the main substrate. However, no currently reported research studies have compared these methodologies when using RMGI bonded to dentin and no studies have used the 4PBBS test as a method to measure the dentin bond strength of RMGI.

The first null hypothesis was rejected indicating that there was a difference in the strength value among three testing methods. Our data showed that the 4PBBS tests resulted in significantly ($p<0.0001$) higher mean bond strength values than the μTBS and the SBS tests (Figure 9). The finding of higher bond strength values for the 4PBBS test is consistent with other investigations that used this test.[10,14] For example, 4PBBS testing found that the mean bond strength of a self-etching primer (Clearfil SE Bond, Kuraray, Osaka, Japan) to dentin was 90.6 ± 2.5 MPa. This value is considerably higher than the mean values of the same adhesive system using the SBS and the μTBS test (47.1±7.6 and 60.5 ± 7.0 MPa respectively)[47,48]. It is not appropriate to compare the absolute mean bond strength test values among different test methods as each test has different variables.

The mean dentin SBS value of Fuji II LC in the present study (15.7 ± 7.1 MPa) is consistent with the current literature. Research studies of Fuji II LC SBS to human dentin

have reported values in the range of ~ 5 – 22 MPa.[11,49,50,69] The wide range of SBS values may be related to variation in specimen geometry, test load configuration and increased potential for the presence of pre-existing cracks in the RMGI material. Other reasons for high scatter among the mean SBS values may be due to the inclusion of test results from cohesive failures as well as pre-testing failures into the statistical analyses.[8]

The mean μTBS value of Fuji II LC in this study (9.7 ± 5.3 MPa) was lower than the range of values reported in the current literature. Recent μTBS studies report the μTBS value of the dentin/Fuji II LC to be in the range of ~ 18.5 -31 MPa.[15,16,66.67] The wide range of μTBS values may be related to differences in beam geometry, jig type, trimming methods, loading speed, specimen alignment, degrees of dehydration, variations in dentinal tubules, and specimen shape.[28,29,32,33] All of these factors are critical and influence final μTBS test outcomes. Because no internationally recognized standardized test protocol for the testing of adhesive systems is yet available, it can be easily noticed that the absolute values differ widely even when testing the same material and with the same test method. This highlights the influence of the test institute on the bond strength values.[8,9]

The secondary null hypothesis was rejected, since modes of failures of RMGI restorative material were found to be statistically significantly different between the test methods used in this study ($p=0.006$). There is no clear consensus in the literature regarding classification of failure modes.[8] In this study the modes of failure of the tested specimens were classified as adhesive at the interface, cohesive in the material or in the dentin, and mixed (Figure 10).[23] In the present study, SBS test showed a higher percentage of adhesive failure. This finding is in contrast to other SBS studies that assessed the adhesive bond of composite resin based materials to dentin.[11,49,50] This may be related to the relatively low

shear bond strength of RMGI material to the dentin. Based on Schreiner & others, SBS values higher than 20 MPa, will likely result in more cohesive failures.[44] The mean SBS value of 15 MPa found in this study may reveal the adhesive failure during SBS testing. Reduced tendency for cohesive failure within the Fuji II LC may also be secondary to ongoing improvement in the material properties of this RMGI. The SBS findings of this study are similar to others who reported predominantly adhesive failures for this material. [69]

The failure mode of the μTBS test was predominantly mixed. Microscopic assessment of the specimens revealed a half-moon pattern on the border of the bonded area in 69% of the specimens. This type of failure pattern may result from the inherent brittleness and the presence of voids in the RMGI material. Additionally, specimen misalignment during jig assembly and/or testing may also have contributing to increased percentage of mixed failures.

The 4PBBS test showed the highest percentage of RMGI cohesive failure (33%). The majority of this type of failure occurred in the first 8 tested beams. It may be that these beams where not properly aligned in the test apparatus, which requires that the RMGI/dentin interface be exactly in the middle of the apparatus. In general, the majority of the area of the mixed failures of 4PBBS and μTBS tests was located at the adhesive interface with small areas (≤20% of the total surface) of cohesive failure within the RMGI.

SBS testing of Fuji II LC resulted in no pre-test failures. This may be due to the relatively large size of the bonding area and that no post-bonding specimen processing is required. Both μTBS and 4PBBS tests had pre-test failures that occurred at the interface. This finding suggests that these tests may not be appropriate for assessing the dentin bond strength of Fuji II LC. Spontaneous debonding at the interface may be related to small

specimen bonding area (about 1mm^2 or less) and/or post bonding processing required in these tests.[4] The level of RMGI brittleness may limit the usefulness of the μTBS method. Brittle materials are generally considered unsuitable for μTBS testing.[68]

Limitations should always be considered for results of *in vitro* bond strength studies. Clinical conditions cannot be fully simulated *in vitro*.[63] Aspects such as the internal pulpal pressure (and resultant dentinal fluid movement into the bonding interface), tooth stress dynamics and the 3-dimensional nature of cavity preparations can not be fully replicated using *in vitro* protocols.[71] Obviously this study was not able to replicate or even simulate any of these clinical realities. Further limitations of the study include that specimens were tested only at 24 h after the RMGI was placed. Storage time is also known to influence bond strengths. Extended periods of water storage cause mechanical and morphological degradation,[72] which leads to a decrease in bond strength and might better simulate *in vivo* conditions.[4,8,71]

SBS testing demonstrated the highest percentage of adhesive failure, less technique sensitivity, and a lower amount of test material for specimen preparation. It is interesting to note that specimen preparation for SBS testing required less Fuji II LC as compared with the specimen preparation for μTBS and 4PBBS (Table 4). One Fuji II LC capsule was used to create two SBS specimens, while μTBS and 4PBBS specimen preparation required at least two capsules to create one specimen. However, SBS testing creates a non-uniform shear stress at the interface. Test values may not represent a true measurement of the adhesive bond strength of the RMGI. The μTBS test may allow better stress distributed at the interface as compared to SBS methods but the specimen preparation was found to be laborious and technique-sensitive. The 4PBBS is not recommended as a routine dentin bond strength test

for RMGI restorative materials secondary to the difficulty of specimen preparations, orientation, and the resulted cohesive failures in the material. The 4PBBS may be applicable for other types of restorative material.[10,14]

7. Conclusions

Within the limitations of this *in vitro* study, the following conclusions may be made:

1- The relative *in vitro* dentin bond strength value of a resin-modified glass ionomer restorative material is greatly affected by the test method.

2- The SBS test method demonstrated the highest percentage of adhesive failure and lowest technique sensitivity when testing a RMGI material.

3- The majority of μTBS and 4PBBS failures were mixed and therefore the μTBS and 4PBBS tests may not be optimal for comparison of the relative bond strength values of RMGI materials to dentin.

4- Use of the SBS test may allow more controlled comparison of the adhesive dentin bond among various RMGI formulations, whether already commercially available or under development

FIGURES

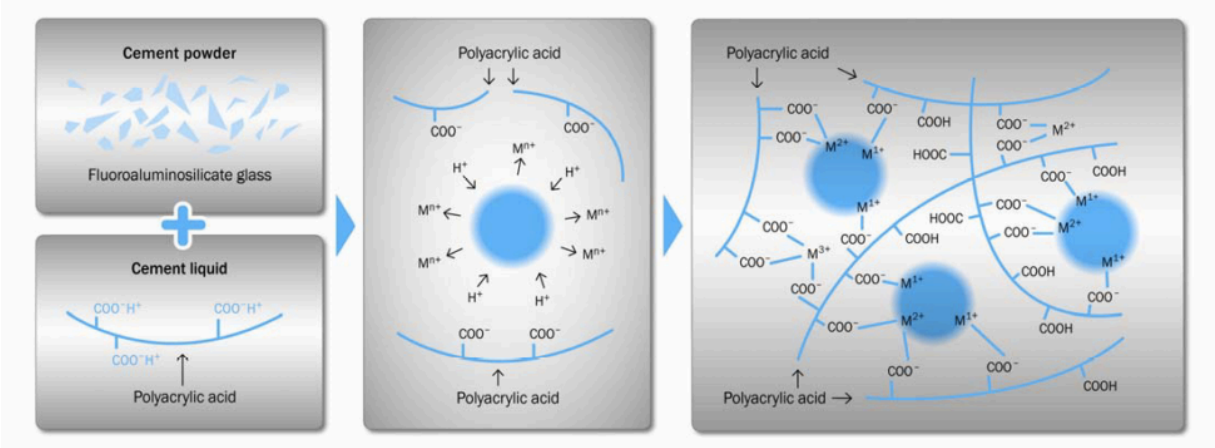

Figure 1: Setting reaction of Conventional GIC.[39]

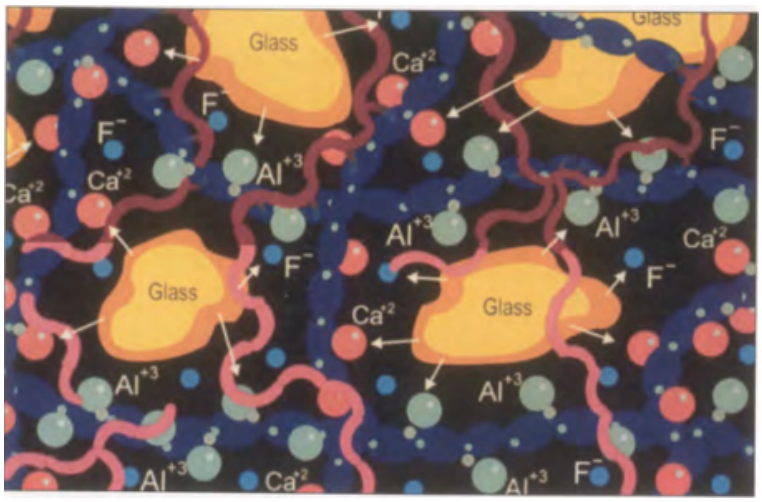

Figure 2: The setting reaction of RMGI material. Red chains represent fully polymerized resin.[55]

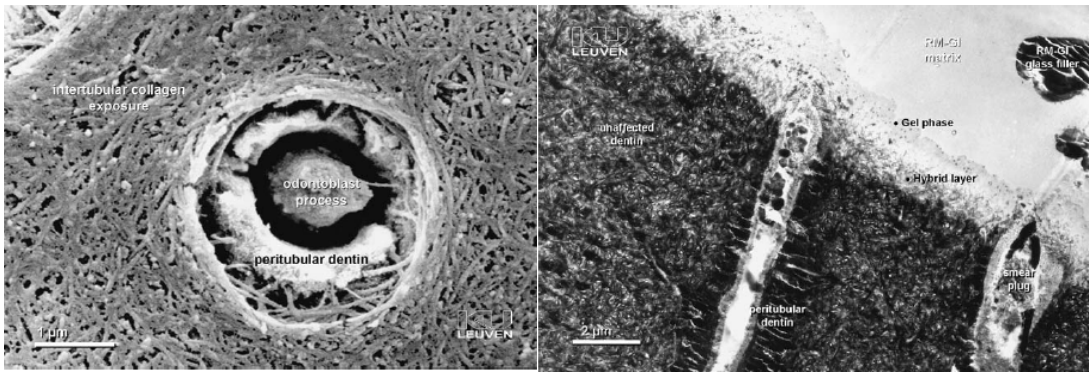

Figure 3: Adhesion of RMGI material to dentin. **A)** The effect of a polyacrylic acid conditioner that was applied for 10 s on dentin previously covered by a smear layer**. B)** Two-fold structural appearance of a glass-ionomer-dentin interface resulting from the application of the resin-modified glass-ionomer adhesive Fuji Bond LC (GC America). [6]

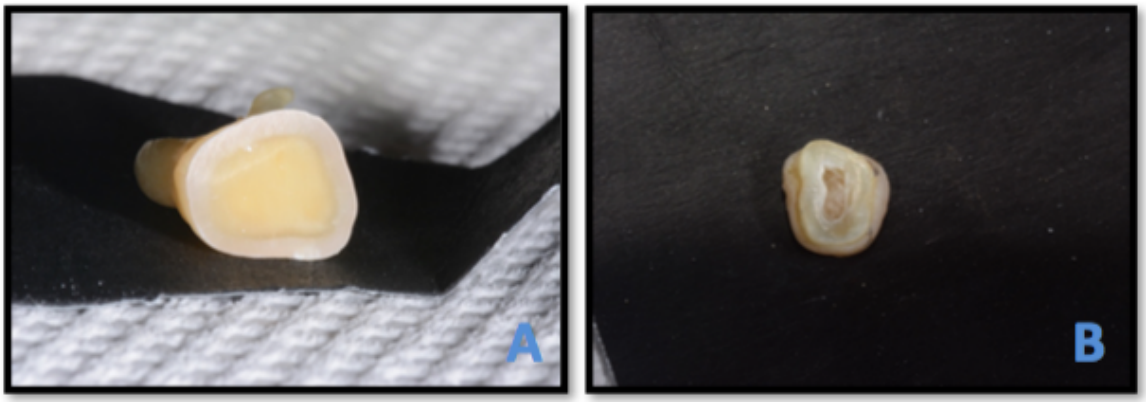

Figure 4: A) Occlusal dentin after exposure and polishing. B) Apical view of a specimens after root separation and debridement.

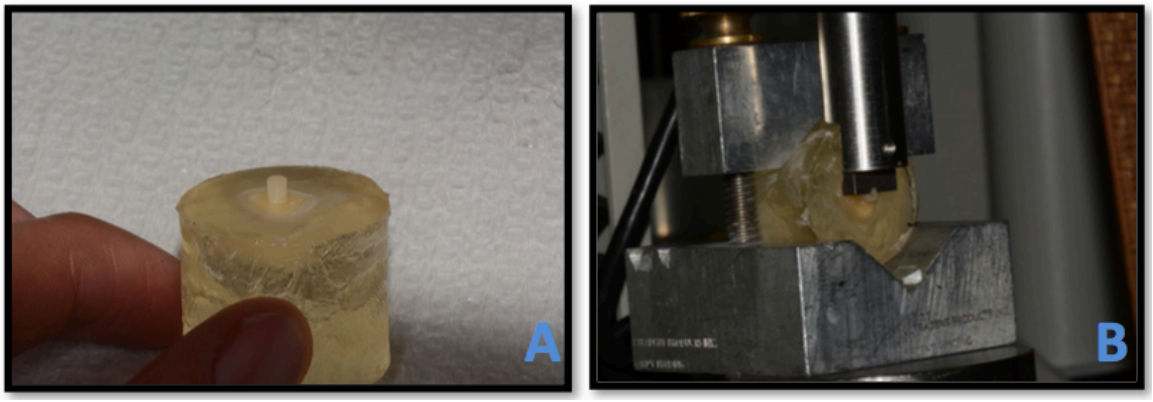

Figure 5: A) Specimen for SBS testing B) A specimen mounted in the Instron machine.

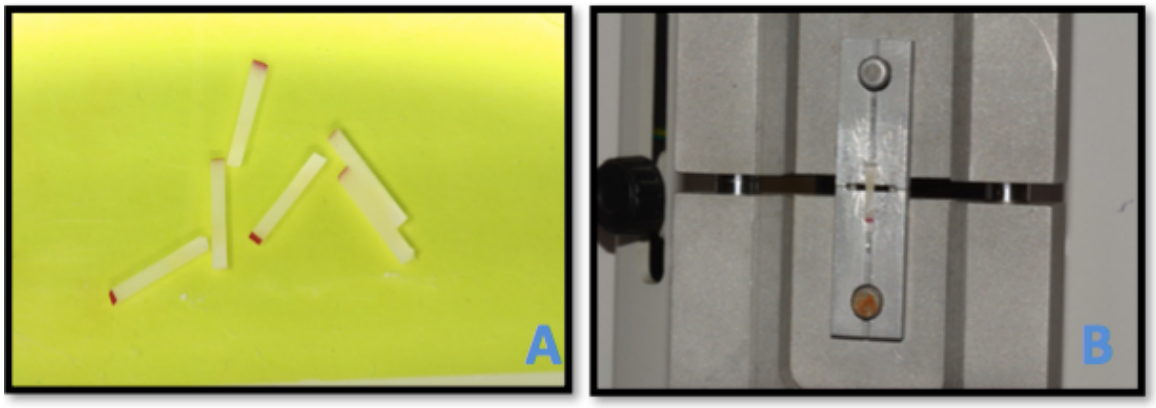

Figure 6: A) Beams (~ 1 mm x 1 mm x 6 mm) prepared for μTBS testing B) A beam mounted in EZ-test machine.

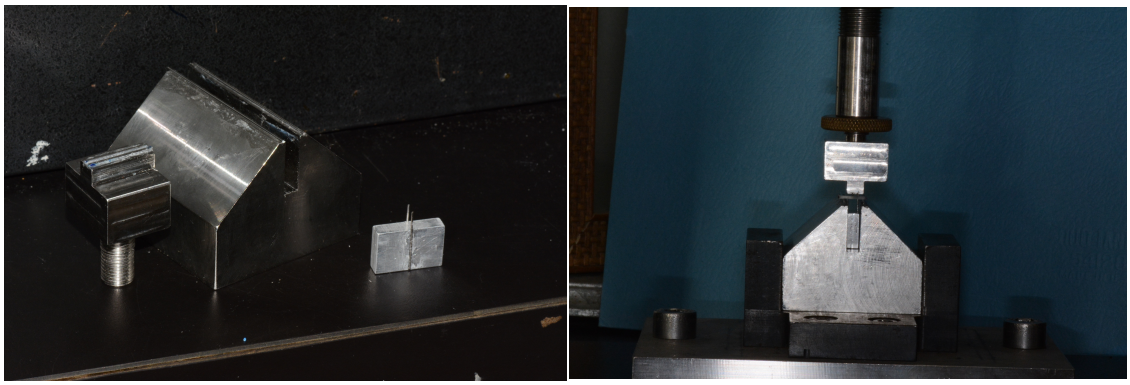

Figure 7: A) 4-Point Bending test assembly **B)** A beam mounted in the Instron machine.

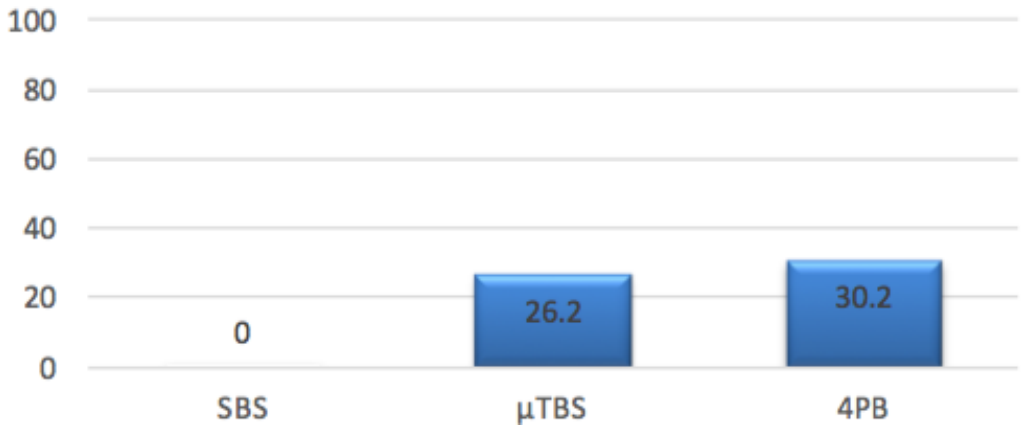

Figure 8: Percentage of pre-test failure by test method.

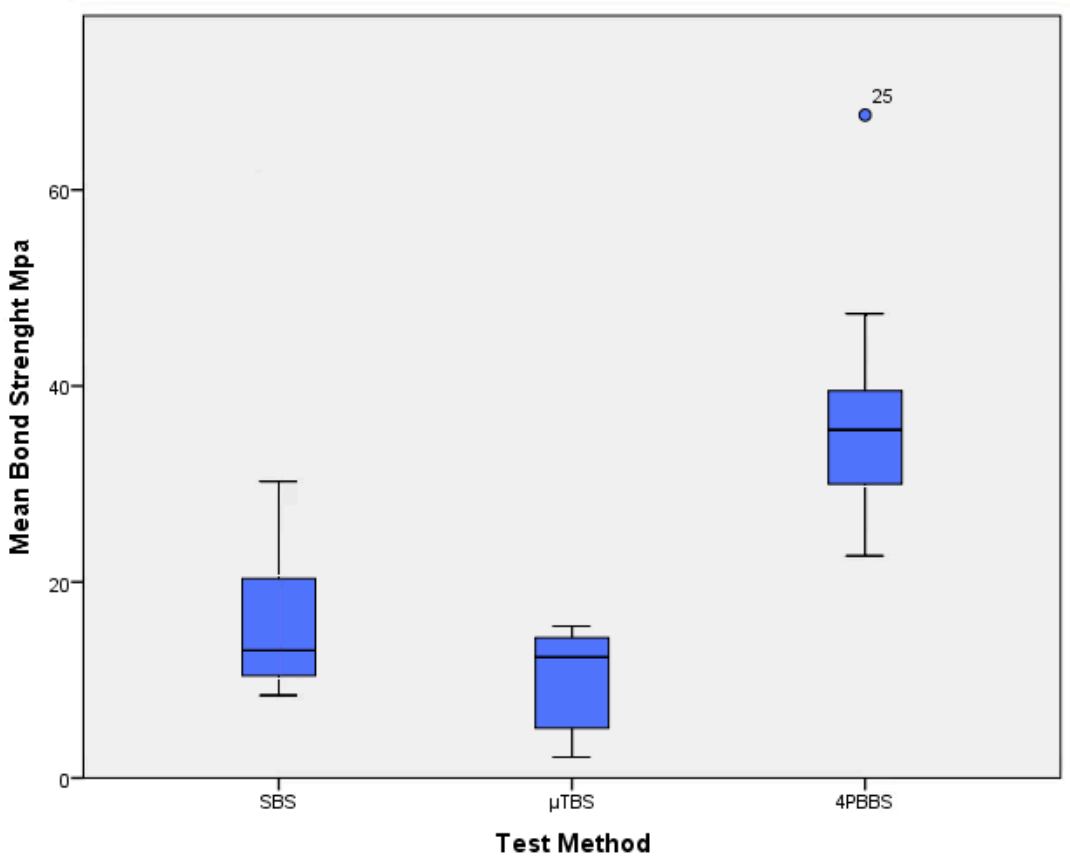

Figure 9: Mean Dentin Bond strength of Fuji II LC by test method.

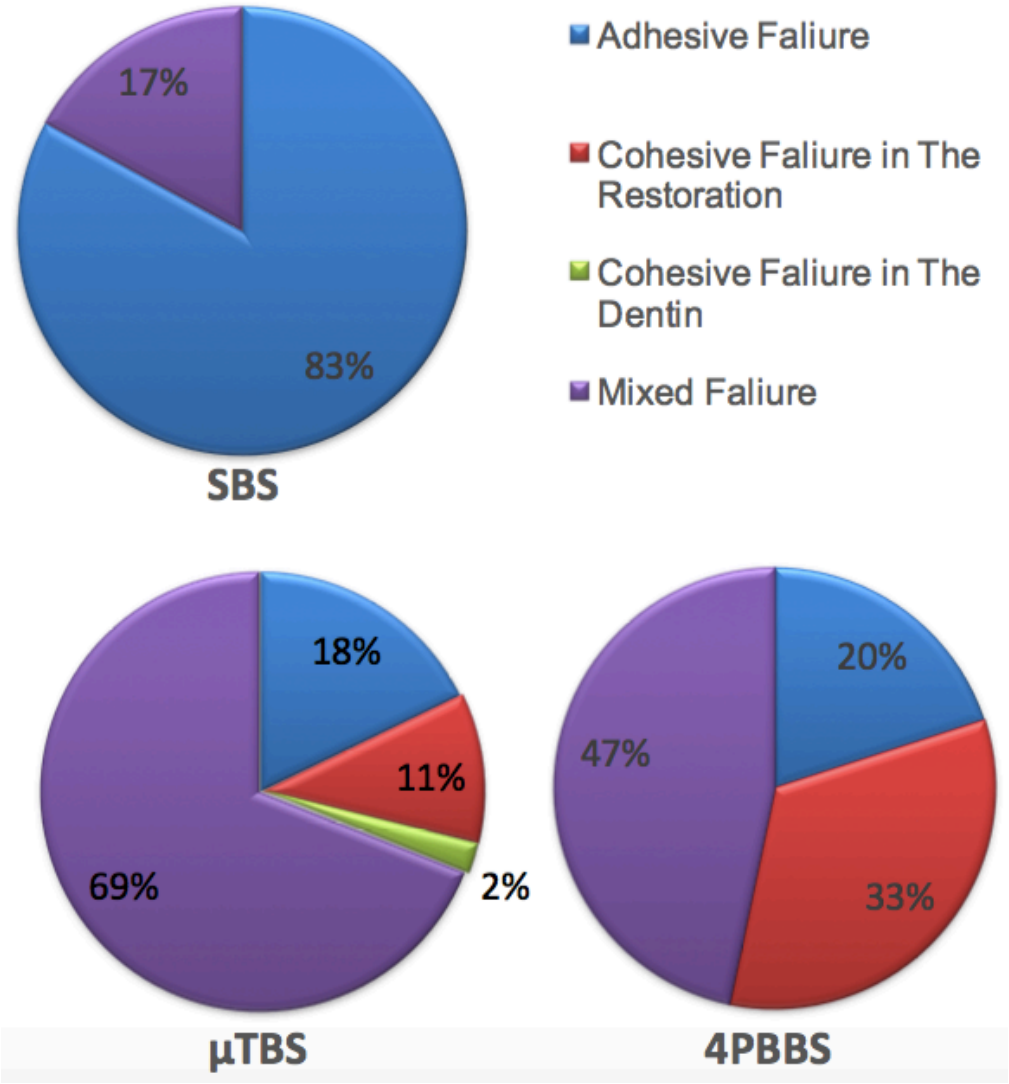

Figure 10: Representation of types and relative percentages of modes of Fuji II LC bond failure as observed by Stereomicroscopy.

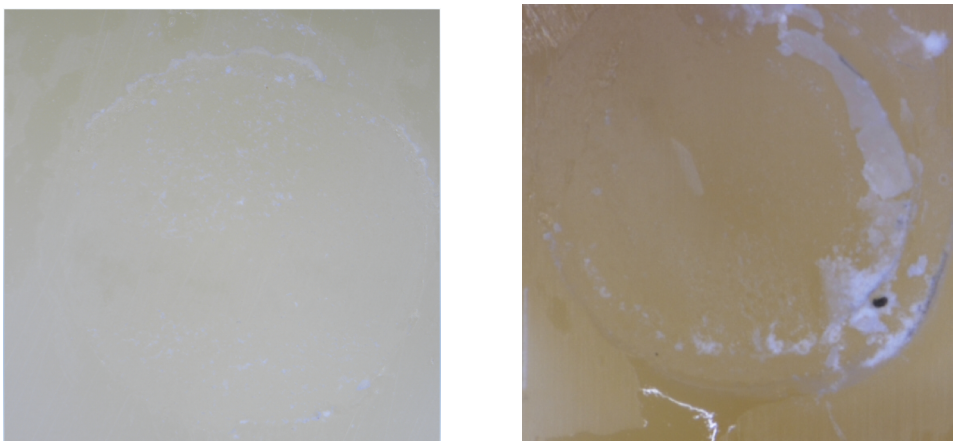

Figure 11: Examples of modes of failure in the SBS test **A)** Adhesive **B)** Mixed failure.

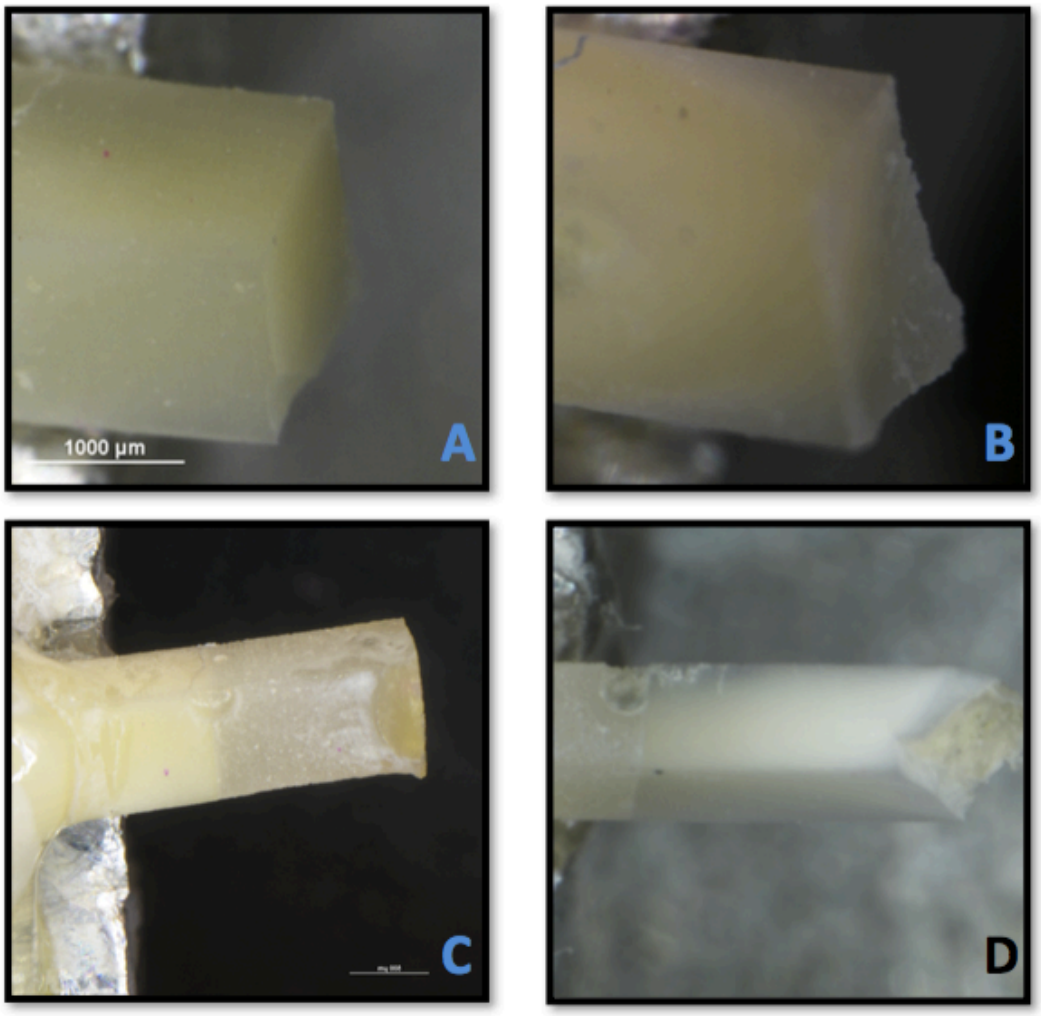

Figure 12: Examples of modes of failure in the µTBS and the 4PBBS tests: A) Adhesive, B) Mixed, C) Cohesive in the RMGI D) Cohesive failure in dentin

TABLES

Table 1: Materials, composition and application directions followed in this study:

Material	Manufacturer	Composition	Application
Fuji II LC Capsule	GC America	Powder: fluoroalumino-silicate glass Liquid: polyalkenoic acid, HEMA, dimethacrylate, camphorquinone, water	Apply cavity conditioner to dentin surfaces and leave undisturbed for 10s; rinse with water for 10s; gently air-dry for 5s, leaving a moist surface. Automatically mix capsules for 10s; apply to dentin surfaces; light-cure was accomplished using Demi plus (Kerr) for 20s with a light output 1200 mW/cm^2. Apply a final coat of EQUIA Coat and light cure for 20s.
Cavity conditioner	GC America	20% Polyacrylic acid, 3% Aluminum chloride hydrate, 77% Distilled water	
EQUIA Coat	GC America	25–50% MMA, 1–5% Photoinitiator, 1-5 %phosphoric acid ester monomer	

HEMA: 2-hydroxyethyl methacrylate.
MMA: methyl methacrylate

Table 2: Mean (+/- SD) dentin bond strengths values of the RMGI by test method. The number of pre-test failures were not included in the calculation of the mean bond strength values:

Test	Teeth	Number of Cylinders or Beams Tested	Number of Pre-test Failures	Mean (+/-SD) Bond Strengths in MPa
SBS	12	12 cylinders	0	15.7 ±7.1
μTBS	12	61 Beams	16	9.7 ± 5.3
4PBBS	12	43 Beams	13	37.3 ± 12.8

Table 3: Number (specimen number, percentage) of modes of RMGI bond failure by test method as observed by Optical Stereomicroscopy:

Test	Adhesive (N, %)	Cohesive in the Material (N, %)	Cohesive in the Dentin (N, %)	Mixed (N, %)
SBS	10 (83)	0 (0)	0 (0)	2 (17)
μTBS	8 (17.8)	5 (11.1)	1 (2.2)	31 (68.9)
4PBBS	6 (20)	10 (33.3)	0 (0)	14 (46.7)

Table 4: Comparison between SBS, µTBS and 4PBBS tests based on the results of this study:

	SBS	µTBS	4PBBS
Test Mechanics	Sliding of two surfaces.	Stretch/elongation of the bonded specimen	Perpendicular loading in the central area of an unsupported span.
Stress Nature	Not uniformly distributed at the interface.	Improved stress distribution at the interface.	Combination of Compressive, Tensile and Shear Stress. More tensile at the interface
Mode of failure	Mostly adhesive with no cohesive failures.	Mostly mixed with some cohesive failures.	Mostly mixed with more cohesive failures than µTBS
Technique	Easy, fast, with no pre-test failures	Difficult, time consuming, with high % pre-test failures	Most difficult, with high % pre-test failures
Material Usage Demands	1 capsule/ 2 teeth	1-2 capsules/ 1 tooth	2 capsules/ 1 tooth

REFERENCES

1) Anusavice K Phillip's Science of Dental Materials, 12th edition. Philadelphia, PA: Saunders; 2013.

2) Wilson AD and Kent BE. The glass-ionomer cement, a new translucent dental filling material. *Journal of Applied Chemistry and Biotechnology*. 1971, 21: 313–313.

3) Mount GJ. Glass ionomers: a review of their current status. *Oper Dent* 1999;24: 115–24.

4) Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A. Relationship between bond-strength tests and clinical outcomes. *Dent Mater*. 2010;26: e100–21.

5) Mitra SB. Photocurable ionomer cement systems. European Patent Application 323120,1988.

6) Van Meerbeek B, Yoshida Y, Inoue S, De Munck J, Van Landuyt K, & Lambrechts P. Glass-ionomer adhesion: the mechanisms at the interface. *Journal of Dentistry*. 2006;34: 615–617.

7) Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellemans L, Lambrechts P, Wakasa K. Evidence of chemical bonding at biomaterial-hard tissue interfaces. *Journal of Dental Research*. 2000;79: 709–14.

8) Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. *Dent Mater*. 2010; 26: e78–93.

9) Heintze SD. Clinical relevance of tests on bond strength, microleakage and marginal adaptation. *Dent Mater*. 2013; 29: 59–84.

10 Staninec M, Kim P, Marshall GW, Ritchie RO, Marshall SJ. Fatigue of dentin-composite interfaces with four-point bend. *Dental Materials*. 2008; 24: 799–803.

11) Fritz UB, Finger WJ, Uno S. Resin-modified glass ionomer cements: Bonding to enamel and dentin. *Dent Mater*. 1996;12: 161–166.

12) Sidhu SK, Sherriff M, Watson TF. Failure of resin-modified glass-ionomers subjected to shear loading. *J Dent.* 1999;27: 373–381.

13) Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength evaluation of a micro-tensile bond test. *Dent Mater.* 1994;10:236–40.

14) Staninec M, Nguyen H, Kim P, Marshall, GW, Ritchie RO, Marshall SJ. Four-point bending evaluation of dentin-composite interfaces with various stresses. *Med Oral Patol Oral Cir Bucal.* 2008,13:81–84.

15) Tanumiharja M, Burrow MF, Tyas MJ. Microtensile bond strengths of glass ionomer (polyalkenoate)cements to dentine using four conditioners. *J Dent.* 2000;28: 361–6.

16) Coutinho E, Cardoso M V, De Munck J, Neves AA, Van Landuyt KL, Poitevin A, Peumans M, Lambrechts P, Van Meerbeek B. Bonding effectiveness and interfacial characterization of a nano-filled resin-modified glass-ionomer. Dent Mater. 2009;25:1347–57.

17) Pashley DH, Tao L, Boyd L, King GE, Horner JA. Scanning electron microscopy of the substructure of smear layers in human dentine. *Arch Oral Biol*. 1988;33:265–270.

18) De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Bream M, Van Meerbeek B. A Critical Review of the Durability of Adhesion to Tooth Tissue: Methods and Results. *J Dent Res*. 2005;84:118–132.

19) Van Meerbeek B, De Munck J. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent*. 2003;28:215–235.

20) De Hoff PH, Anusavice KJ, Wang Z. Three-dimensional finite element analysis of the shear bond test. *Dent Mater*. 1995; 11:126–31

21) Versluis A, Tantbirojn D, Douglas WH. Why do shear bond tests pull out dentin? *J Dent Res.* 1997;76:1298–307.

22) Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. *Dent Mater*.1995; 11:117–25.

23) Otani A, Amaral M, May LG, Cesar PF, Valandro LF. A critical evaluation of bond strength tests for the assessment of bonding to Y-TZP. *Dent Mater*. 2015;31:648–656.

24) Bergoli C, Amaral M, Valandro LF. The disk-specimen height does not influence the push-out bond strength results between fiber post and root dentin. *J Adhes*. 2012;88: 212–23.

25) Zicari F, Couthino E, De Munck J, Poitevin A, Scotti R, Naert I, Van Meerbeek B. Bonding effectiveness and sealing ability of fiber-post bonding. Dent Mater. 2008; 24:967–77.

26) Bowen RL. Adhesive bonding of various materials to hard tooth tissues. Methods of determining bond strength. *Journal of Dental Research.* 1965; 44:690-5

27) Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. *J of Dental Research*. 2001;80:1605–14.

28) Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, Van Meerbeek B. Critical analysis of the influence of different parameters on the microtensile bond strength of adhesives to dentin. *J Adhes Dent*. 2008;10:7–16.

29) Goracci C, Sadek FT, Monticelli F, Cardoso PEC, Ferrari M. Influence of substrate, shape, and thickness on microtensile specimens' structural integrity and their measured bond strengths. *Dent Mater*. 2004;20:643–654.

30) Frankenberger R, Krämer N & Petschelt A. Fatigue behavior of different dentin adhesives. *Clin Oral Investig*, 1999;3:11-7

31) Tam LE, Pilliar RM. Fracture toughness of dentin/resin-composite adhesive interfaces. *Journal of Dental Research* 1993; 72:953–9.

32) Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, Van Meerbeek B. Influence of three specimen fixation modes on the micro-tensile bond strength of adhesives to dentin. Dent Mater. 2007;26:694–9

33) Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, Tay F. The microtensile bond test: a review. *J Adhes Dent* 1999;1:299–309.

34) ISO. Dental materials – testing of adhesion to tooth structure. Technical specification no. 11405; 2003.

35) Barkmeier WW, Erickson RL, Latta MA. Fatigue limits of enamel bonds with moist and dry techniques. *Dent Mater*. 2009;25:1527-31

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

37) Armstrong SR, Keller JC, Boyer DB. The influence of water storage and C-factor on the dentin-resin composite microtensile bond strength and debond pathway utilizing a filled and unfilled adhesive resin, *Dent Mater*. 2001;17: 268- 276

38) Van Meerbeek B, Perdigão J, Lambrechts P, Vanherle G. The clinical performance of adhesives. *J Dent.* 1998;26:1-20.

39) Lohbauer U. Dental Glass Ionomer Cements as Permanent Filling Materials? – Properties, Limitations and Future Trends. *Materials (Basel)*. 2009;3:76–96D.

40) Swift EJ, Perdigao J, Heymann HO. Bonding to enamel and dentin: a brief history and state of the art. *Quintessence Int*. 1995;26:95–110.

41) Wilson AD, Kent BE, Clinton D, Miller RP. The formation and microstructure of dental silicate cements. *J. Mater Sci*. 1972;7:220–238.

42) Glass ionomer resin state of art. *Clin Res Assoc Newsletter* 1993;17:1-2

43) Inoue S, Van Meerbeek B, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H. Effect of remaining dentin thickness and the use of conditioner on micro-tensile bond strength of a glass-ionomer adhesive. *Dent Mater*. 2001; 17:445-455.

44) Schreiner RF, Chappell RP, Glaros AG , Eick JD. Microtensile testing of dental adhesives. *Dent Mater*. 1998;14: 194-201.

45) Van Meerbeek B, Braem M, Lambrechts P, Vanherle. Morphological characterization of the interface between resin and sclerotic dentine. *J Dent.* 1994;22:141-146.

46) Gale MS and Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent.* 1999;27:89-99.

47) Walter R, Swift EJ, Boushell LW, Braswell K. Enamel and dentin bond strengths of a new self-etch adhesive system. *J Esthet Restor Dent*. 2011;23: 390–6.

48) Sheikh H, Heymann HO, Swift EJ, Ziemiecki TL, Ritter AV. Effect of Saliva Contamination and Cleansing Solutions on the Bond Strengths of Self-Etch Adhesives to Dentin. *J Esthet Restor Dent*. 2010;22:402–410.

49) Swift EJ, Pawlus MA, Vargas MA. Shear bond strengths of resin-modified glass-ionomer restorative materials. *Oper Dent*. 1995;20:138–143.

50) Thean HP, Mok BY, Chew CL. Bond strengths of glass ionomer restoratives to primary vs permanent dentin. *ASDC J Dent Child*. 2000;67:112–6, 82.

51) Wilson AD. Resin-modified glass-ionomer cements. *Int J Prosthodont*.1990;3:425–9.

52) Xie D, Brantley WA, Culbertson BM, Wang G. Mechanical properties and microstructures of glass-ionomer cements. *Dent Mater*. 2000;16(2):129–138.

53) Braem MJA, Lambrechts P, Gladys S, Vanherle G. In vitro fatigue behavior of restorative composites and glass ionomers. *Dent Mater*. 1995;11:137—41.

54) McCabe JF. Resin-modified glass-ionomers. *Biomaterials*. 1998;19:521–527.

55) Mount GJ. An Atlas of Glass-Ionomer Cement: A Clinician's Guide, 3rd edition, London: Martin Dunitz. 2002

56) Nicholson JW, Anstice HM & Mclean JW. A preliminary report on the effect of storage in water on the properties of commercial light-cured glass-ionomer cements. *British Dental Journal*. 1992; 173 98-101.

57) Forsten L. Fluoride release of glass ionomers. *Journal of Esthetic Dentistry*. 1994;6:216-222

58) Hume WR, Pulpal responses to glass-ionomers. In Glass-ionomers: the Next Generation; Proceedings of the Second Symposium on Glass-Ionomers edited and published by Peter Hunt, Philadelphia.

59) Earl MS, Mount GJ & Hume WR. The effect of varnishes and other surface treatments on water movement across the glass-ionomer cement surface: II. *Australian Dental Journal*. 1989; 34:326-329.

60) McLean JW. Dentinal bonding agents versus glass-ionomer cements. *Quintessence Int*. 1996;27:659–67.

61) Burgess J, Norling B, Summitt J. Resin Ionomer Restorative Materials: The New Generation. *J Esthet Restor Dent*. 1994;6:207–215.

62) Opdam NJM, Bronkhorst EM, Loomans B a C, Huysmans MCDNJM. 12-Year Survival of Composite Vs. Amalgam Restorations. *J Dent Res*. 2010;89:1063–7.

63) Kelly JR. Evidence-based decision making: Guide to reading the dental materials literature. *J Prosthet Dent*. 2006;95(2):152–60.

64) Nicholson JW. Glass ionomer dental cements: update. *Mater Technol*. 2010;25:8–13.

65) Pereira PNR, Yamada T, Tei R, Tagami J. Bond strength and interface micromorphology of an improved resin-modified glass ionomer cement. *Am J Dent*. 1997;10:128–132.

66) Cook NB, Feitosa SA, Patel A, Alfawaz Y, Eckert GJ, Bottino MC. Bonding Ability of Paste-Paste Glass Ionomer Systems to Tooth Structure: In Vitro Studies. *Oper Dent*. 2014;40:304–312.

67) Pereira PN, Sano H, Ogata M, Zhang L, Nakajima J, Tagami J, Pashley DH. Effect of region and dentin perfusion on bond strengths of resin-modified glass ionomer cements. *J Dent*. 2000;28(5):347–354.

68) Sadek FT, Monticelli F, Muench A, Ferrari M, & Cardoso PE. A novel method to obtain microtensile specimens minimizing cut flaws. *J Biomed Mater Res B Appl Biomater.* 2006;78: 7-14.

69) Imbery AT, Namboodiri A, Duncan A, Amos R, Best AM, Moon PC. Evaluating dentin surface treatments for resin-modified glass ionomer restorative materials. *Oper Dent*. 2013;38:429–38.

70) Kanca J III. Resin bonding to wet substrate -I. Bonding to dentin. *Quintessence International.* 1992;23: 39- 41

71) Oliveira GCB, Oliveira GMS, Ritter AV, Heymann HO, Swift EJ, Yamauchi M. Influence of tooth age and etching time on the microtensile bond strengths of adhesive systems to dentin. *J Adhes Dent*. 2012;14:229–34.

72) Shono Y, Terashita M, Shimada J, Kozono Y, Carvalho RM, Russell CM, Pashley DH. Durability of resin-dentin bonds. *J Adhes Dent*. 1999;1:211-218.

73) Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials. *Dent Mater*. 2005;21:864–881.