

EFFECTS OF DELAYED POLYMERIZATION TIME AND BRACKET MANIPULATION
ON ORTHODONTIC RESIN MODIFIED GLASS IONOMER ADHESIVE

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ABSTRACT

This study examined the effect of varying delayed polymerization times in combination with bracket manipulation on shear bond strength (SBS), degree of conversion (DC), and adhesive remnant index (ARI) score when using a resin modified glass ionomer (RMGI) adhesive. Specimens were divided into three groups of clinically relevant delay times (0.5, 2, and 4-min) to simulate the delay that frequently occurs between bracket placement and manipulation and subsequent light curing.

Based on an analysis of variance ($\alpha=.05$), the SBS was not significantly different between the three groups. While one of the goals of this study was to be the first study to quantify DC of RMGI using Raman microspectroscopy, several challenges, including weak peak signal with and without fluorescence, were encountered and as a result, DC could not be determined. A significant difference ($p<0.05$) in ARI score was detected between the 0.5-min and 4.0-min delay groups with more adhesive remaining on the bracket with increasing delay time. A Spearman correlation between SBS and ARI indicated no positive association between SBS and ARI measures across delay times.

The results of this study suggest that clinically relevant delay times of 0.5, 2, and 4-min do not negatively impact the SBS of a RMGI adhesive. However, with increasing delay

time, the results suggest that more adhesive might remain on the bracket during debonding. With more adhesive remaining on the bracket, this could be beneficial in that less adhesive needs to be removed from enamel by grinding at the time of bracket removal when orthodontic treatment is completed.

APPROVAL PAGE

The faculty listed below, appointed by the Dean of the School of Dentistry have examined a thesis titled “Effects Of Delayed Polymerization Time and Bracket Manipulation on Orthodontic Resin Modified Glass Ionomer Adhesive,” presented by Danielle W. Gilbert, candidate for the Master of Science degree, and certify that in their opinion it is worthy of acceptance.

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CHAPTER 1

INTRODUCTION

Orthodontic Brackets

Orthodontic tooth movement occurs via forces applied to the teeth to direct tooth movement through alveolar bone to a desired location. Fixed or removable appliances may be used to apply orthodontic forces to the dentition. Fixed appliances are less dependent on patient compliance and can deliver more precise tooth movements when compared to removable appliances. Fixed orthodontic appliances generally consist of bands and brackets. Bonding brackets largely replaced bands in the 1970's due to improvements in enamel etching and bonding materials (Buonocore 1955; Zachrisson and Buyukyilmaz 2012). Today, maxillary first molars are the only teeth routinely banded by most orthodontists in the US, and banding of molars and premolars is less common than in the past (Keim et al. 2008). Brackets have several advantages over orthodontic bands including improved esthetics, greater ease in maintaining oral hygiene, and do not require tooth separation before treatment or closure of spaces after treatment (Zachrisson and Buyukyilmaz 2012). Brackets may be metal, plastic, or ceramic, with most orthodontists preferring metal brackets (Keim et al. 2008). Metal bracket bonding depends on mechanical retention provided by mesh pads, etched surfaces, or undercuts present on the bracket base (Zachrisson and Buyukyilmaz 2012).

Orthodontic Bracket Adhesives

Fixed orthodontic appliances must have adequate bond strength to maintain attachment to enamel while being subjected to a variety of forces in the oral cavity. An ideal adhesive would have adequate bond strength to prevent premature bond failure, but also

allow for removal of appliances without damaging tooth structure. Other desirable qualities include adequate working time, ease of use, prevention of enamel decalcification, and reasonable price (Millett et al. 2007). A multitude of orthodontic adhesives are currently available and consist of filled or unfilled acrylic or diacrylate resin polymers (Zachrisson and Buyukyilmaz 2012). Adhesives undergo a setting reaction by means of a chemical reaction, light polymerization, or a combination of both (dual-cured). Bonding orthodontic brackets to tooth structure using light-cured resin materials (composites) began in the 1970's and is currently the most commonly used bonding technique (Tavas and Watts 1979). Light-initiated adhesives have the advantage of curing rapidly, which is important when bonding brackets in areas where moisture control is difficult (Zachrisson and Buyukyilmaz 2012). Dual-cure resins are often used with metal brackets or bands because both light and chemical catalysts initiate the setting reaction (Zachrisson and Buyukyilmaz 2012).

Conventional Resin Adhesives

Conventional resin adhesives have been used in orthodontics since the 1960's and gained popularity in the 1970's. Commonly used resin adhesives consist of diacrylate oligomers, dimethacrylate monomers, fillers of silica or glass, and polymerization initiators or accelerators. Most diacrylic resins are based on the bisphenol A glycidyl dimethacrylate (Bis-GMA) monomer (Read 1984). Two-paste resin cements involve mixing prior to application to the bracket base and undergo a chemical-cure reaction. One-step cements do not require mixing and polymerization is initiated by a light source such as quartz tungsten halogen lights, plasma arc lights, or light emitting diodes (Mitra and Sakaguchi 2012). Bond strength of various resin adhesives has been examined extensively in *in vivo* and *ex vivo* studies and most results show conventional resin adhesives to have superior bond strength

and lower bond failure rates when compared to glass ionomer adhesives or resin-modified glass ionomer (RMGI) adhesives (Miguel et al. 1995; Komori and Ishikawa 1997; Rix et al. 2001; Sfondrini et al. 2001; Hegarty and Macfarlane 2002; Summers et al. 2004).

Resin Modified Glass Ionomer Adhesives

Glass ionomer adhesives have been utilized in dentistry since 1972 (Wilson and Kent 1972) and contain basic glass and acidic polymer components that cure via an acid-base reaction (McLean et al. 1994). Major advantages of glass ionomer adhesives are the fluoride releasing and recharging capability; however, they exhibit poor bond strength and greater bonding failures in clinical trials than composite resin adhesives (Miguel et al. 1995; Norevall et al. 1996). RMGI adhesives were developed to combine the favorable properties of conventional glass ionomer adhesives and resin adhesives (Rix et al. 2001). Light-cured RMGI adhesives have been used in orthodontic bonding procedures since the 1990's (Croll and Nicholson 2002).

RMGI adhesives have two setting reactions: polymerization of the composite resin particles and an acid-base reaction (Silverman et al. 1995; Sfondrini et al. 2001; Cheng et al. 2011). Therefore, an ionic bond and hybrid layer bond occurs at the enamel-adhesive interface (Braga and Mitra 2012). The curing process reflects the dual nature of the components of the adhesive; glass ionomers cure via acid-base reactions while traditional resin composite adhesives cure via light-initiated polymerization. The acid-base reaction begins as protons from the polyacrylic acid attack the basic glass particles, resulting in the release of metal and fluoride ions. The free ions form polysalt precipitates as the pH rises. The precipitates form linkages between polyacrylic acid chains (Pearson and Atkinson 1991).

Composite resins undergo free-radical polymerization, which involves chemical bond formation between monomer components to form high molecular weight polymer chains. Free-radical polymerization, a type of addition polymerization, involves the conversion of double bonds to single bonds as monomers are added to the molecule. This takes place in three stages: initiation, propagation, and termination. Free-radicals produced from light, heat, or chemicals serve as the initiator. During propagation, monomers are added to the molecule via the free-radical, and the free-radical moves to the end of the growing polymer chain. Finally, termination occurs (Mitra and Sakaguchi 2012).

The two curing reactions of RMGI occur simultaneously, and a 2010 study showed that the reactions compete (Berzins et al. 2010). As polymerization produces increasing amounts of cross linked polymer chains (after light curing) the diffusion of acid-base reactants is significantly reduced (Young 2002). The acid-base reaction also influences the polymerization. As the acid-base reaction progresses, the material becomes more opaque, reducing the effectiveness of light curing (Nicholson 1998).

The addition of resin particles to conventional glass ionomer adhesives to produce RMGI adhesives resulted in increased bond strength, while also maintaining the ability to uptake and release fluoride (Forsten 1995; Bishara et al. 2007). Plaque adjacent to brackets has a greater concentration of fluoride when RMGI adhesives are used (Chung et al. 1998). Additionally, a previous study reported that using a RMGI adhesive resulted in a lower proportion of *S. mutans* and lactobacilli in plaque samples adjacent to brackets at 1 week and 5 months when compared to conventional resin cements (Wright et al. 1996). Increased fluoride concentration in plaque and reduced proportion of cariogenic bacteria may be beneficial in reducing the incidence of enamel decalcification.

The shear bond strength (SBS) of RMGI adhesives depends on the method of tooth preparation prior to bonding (Newman et al. 2001). While the SBS of RMGI adhesives is less than conventional resin adhesives (Sfondrini et al. 2001), numerous studies have shown that its SBS is sufficient to result in successful bracket bonding (Komori and Ishikawa 1997; Rix et al. 2001; Chitnis et al. 2006). Also, a study demonstrated that the *in vitro* SBS of RMGI adhesives (prepared with 10% polyacrylic acid) and conventional resin adhesives (prepared with 37% phosphoric acid) was not significantly different (Chitnis et al. 2006). An *in vivo* study showed no significant difference in a RMGI adhesive and conventional resin adhesive regarding bracket failure rates at 1.3 years (Summers et al. 2004).

Bracket Bonding Process

Bracket bonding to enamel is a technique sensitive process and requires cleaning, conditioning, and sealing of the enamel surface just prior to bonding of the orthodontic appliances (Zachrisson and Buyukyilmaz 2012). Pumice prophylaxis removes plaque and the organic biofilm from the enamel surface. At this point, proper isolation and moisture control must be established. The enamel is then conditioned with 10-20% polyacrylic acid or 37% phosphoric acid for 15-30 seconds and thoroughly rinsed and dried with oil-free air. The type of enamel conditioner used depends on the bracket adhesive system chosen. In most cases, 37% phosphoric acid is utilized when bonding with conventional resin adhesives, while 10-20% polyacrylic acid is used when bonding with RMGI adhesives (Zachrisson and Buyukyilmaz 2012). Phosphoric acid roughens the enamel surface, increases the bonding surface area, and allows for mechanical bond formation between the enamel and adhesive (Komori and Ishikawa 1997). Mechanical bond formation does not occur with polyacrylic acid because the enamel structure is not altered (Komori and Ishikawa 1997). Also,

phosphoric acid penetrates the enamel surface to a greater depth and results in greater loss of enamel when compared to 10% polyacrylic acid (Summers et al. 2004).

Next, sealants, primers, or self-etching primers may be applied. The adhesive is then applied to the bracket base and placed on the tooth in the desired position. The manufacturer of a conventional resin orthodontic adhesive recommends that the adhesive be immediately light cured once the bracket is in final position and excess adhesive has been removed. If a time delay is anticipated before light curing, a dark mask should be placed over the mouth to prevent premature curing due to ambient light (Unitek 2012). The manufacturer of a RMGI adhesive recommends that the adhesive be light cured after one quadrant or full arch is bonded, and “tack curing” can be done if drift of the bracket is a concern (GC America 2010). Tack curing involves quickly light curing brackets with the goal of preventing bracket drift due to the viscosity of the adhesive; however, complete light curing is still required.

Bracket Bond Strength

Bond strength is a significant factor in selecting an orthodontic adhesive because premature bond failure is problematic for the practitioner and patient. Bond failure results in an increased number of office visits, longer appointments, increased use of materials, and increased overall treatment time (Zachrisson and Buyukyilmaz 2012). Bond strength is evaluated by assessing tensile bond strength and SBS, with SBS being the most prevalent testing method (Powers and Sakaguchi 2006). In 1975 Reynolds recommended that the minimum bond strength required for most clinical orthodontic situations is 6-8 MPa, however this was not based on specific data (Reynolds 1975). Since that time many studies have examined bond strength of various orthodontic adhesives and the results vary greatly

within studies and between studies. The reported bond strength of conventional orthodontic resin adhesives ranges from 11.6–25.8 MPa (Rix et al. 2001; Sfondrini et al. 2001; Summers et al. 2004; Cheng et al. 2011; Pereira et al. 2013). The reported bond strength of RMGI adhesives varies based on many factors, including enamel surface pretreatment method utilized. Enamel treatment with phosphoric acid of varying concentrations results in a SBS of RMGI adhesives of 10–19MPa (Sfondrini et al. 2001; Valente et al. 2002; Cheng et al. 2011). Enamel pretreatment with polyacrylic acid results in a SBS of RMGI of 8.6–13.6MPa (Rix et al. 2001; Cacciafesta et al. 2004; Summers et al. 2004; Al Shamsi et al. 2006). One study showed that RMGI adhesives achieve a SBS of 21MPa when using a self-etching primer (Cacciafesta et al. 2004).

One reason for the large range of reported bond strengths is the inconsistency in study design and protocol, testing mode, and data analysis. This inconsistency of *in vitro* bond strength studies makes it difficult to compare studies or make clinical recommendations (Eliades and Brantley 2000). Faster loading rates when debonding brackets result in higher observed bond strengths and must be considered when comparing study results (Powers and Sakaguchi 2006). Further complicating the matter, a recent review and meta-analysis stated that studies frequently fail to report on many factors related to testing design that influence bond strength (Finnema et al. 2010).

Factors Related to Bracket Bond Strength

Many factors affect the bond strength achieved with any adhesive. Tooth preparation, clinical technique, materials, and patient factors are just a few of the variables impacting bond strength. Many studies have examined these factors and often report conflicting results. As previously mentioned, conventional resin adhesives are consistently reported to have

higher bond strengths than RMGI adhesives; however, a 2011 study showed that a RMGI adhesive can achieve higher bond strength than a conventional resin adhesive, with or without acid etching and moisture contamination (Cheng et al. 2011). Other studies have evaluated the relationship between enamel conditioner or etchant used and bracket bond strength. One study found no significant difference in bracket bond tensile strength when a RMGI adhesive was used with 37% phosphoric acid, 10% phosphoric acid, or 10% polyacrylic acid prior to bonding; however, significantly lower tensile strength occurred when no etchant was used (Valente et al. 2002). An *in vitro* study showed that 37% phosphoric acid etchant results in increased bond strength of a RMGI adhesive when saliva contamination occurs (Godoy-Bezerra et al. 2006).

The thickness of adhesive may impact bracket bond strength. A 2005 study demonstrated that RMGI adhesive had the highest tensile and SBS at 0.25mm thickness. The SBS of a light-cured conventional resin adhesive increased as thickness increased from 0 to 0.5mm, while tensile strength decreased (Arici et al. 2005). Studies often examine the impact of thermocycling on the mechanical properties of adhesives. Thermocycling attempts to simulate the intra-oral environment so that information can be clinically applicable. *In vitro* testing has shown that SBS for RMGI adhesives and conventional resin adhesives decreased following thermocycling. After 20,000 cycles the RMGI adhesive had a 26.5% reduction in SBS while the conventional resin adhesive showed a 17.9% reduction (Arici and Arici 2003).

Degree of Conversion

Polymerization occurs when monomer units join via chemical bonds to form polymers of higher molecular weight (Mitra and Sakaguchi 2012). Degree of conversion

(DC) is a measure of polymerization and describes the percentage of carbon-carbon double bonds that have been converted to single bonds to produce a polymer. Reduced polymerization may result in substandard mechanical properties, reduced bond strength, and increased degradation (Eliades 2006). The DC may also be influenced by photo-initiator type and amount, and quantity and composition of the organic matrix (Cerveira et al. 2010; Corekci et al. 2011).

The DC can be measured using techniques such as Fourier transform infrared (FTIR) spectroscopy and Raman microspectroscopy. Both FTIR and Raman microspectroscopy are vibrational techniques that require composite molecules to undergo changes in vibrational energy state due to excitation radiation; however, the method of energy transfer to the composite differs. In FTIR the material absorbs photon energy while in Raman microspectroscopy the energy is transferred via a scatter technique. When analyzing conventional resin adhesives, both instruments compare the C=C vinyl stretching mode at 1640 cm^{-1} to an aromatic reference band at 1610 cm^{-1} before and after polymerization. Both instruments have the same peaks for specific compounds, but the peaks differ in relative intensities (Young et al. 2000). FTIR has a few disadvantages when compared to Raman microspectroscopy. FTIR samples require technique sensitive and time-consuming preparation prior to analysis while Raman microspectroscopy is generally considered easier to perform. Moreover, when using FTIR, the infrared spectra of C=C can be masked by the deformation of H_2O molecules, which also appears at the 1640 cm^{-1} . Furthermore, silica fillers can obscure the infrared spectra in the $1100 - 1000\text{ cm}^{-1}$ range when using FTIR (Pianelli et al. 1999).

Several studies have evaluated the DC of orthodontic resin adhesives producing a wide range of results. The DC for a light-cured conventional resin adhesive was found to be 48% with metal brackets and 58% with ceramic brackets, while a dual-cured cement achieved a DC of 68% under metal brackets (Eliades et al. 2000). Another study showed that DC of several conventional resin adhesives containing Bis-GMA was 57% to 88% and was not related to percentage of filler present in the adhesive (Corekci et al. 2011).

Adhesive Remnant Index

Following orthodontic bracket debonding, the residual adhesive on the tooth or bracket base can be evaluated to determine the location of the bond failure. Bond failure may occur at the bracket-adhesive interface, the adhesive-enamel interface, or within the adhesive. An adhesive that tends to adhere to the bracket base during the debonding procedure is desirable because less adhesive must then be removed from the enamel surface. The Adhesive Remnant Index (ARI) is a visual qualitative assessment developed in 1984 to describe the location of bond failure using a 4-point scale. The scale is defined as: 0 = all adhesive remains on the bracket base, 1 = >50% of the adhesive remains on the bracket base, 2 = <50% of the adhesive remains on the bracket base, and 3 = no adhesive remains on the bracket base (Artun and Bergland 1984). More precise information regarding the location of adhesive after debonding can be obtained with quantitative methods; however, they require specialized equipment or software, more time to analyze the samples, and the information gathered may not be clinically useful (Montasser and Drummond 2009).

Delayed Polymerization and Manipulation of Brackets

Orthodontic brackets are often placed by assistants and subsequently are checked by the orthodontist prior to light curing the adhesive. During this delay, ambient light may begin the polymerization reaction of light-cured or dual-cured adhesives. Additionally, dual-cured adhesives may begin to polymerize due to the chemical reaction that also takes place during the setting reaction.

Beyond the delay time between the assistant placing the brackets and the orthodontists checking placement, the orthodontist will also manipulate the bracket to improve the placement location before adhesive polymerization is initiated. The delay in polymerization in combination with bracket manipulation could potentially have a negative impact on bracket bond strength. According to the most recent edition of *Orthodontics: Current Principles and Techniques*, it is recommended that after the bracket has been placed in the proper location and excess adhesive removed, additional manipulation should be avoided to decrease the chance of bracket bond failure (Zachrisson and Buyukyilmaz 2012).

In a previous *in vivo* study it was reported that depending on the enamel pre-treatment, bracket manipulation could affect bond failure. With a self-etching primer, the bond failure rate increased as the number of bracket manipulations increased; in particular, the bond failure rate doubled when brackets were manipulated more than three times prior to light polymerization. However, with conventional etching using 37% phosphoric acid, there was no effect of bracket manipulation on bond failure rates (Murfitt et al. 2006). Nonetheless, that study only focused on conventional light-cured orthodontic resin adhesives.

Problem Statement

While conventional orthodontic resin adhesives are commonly used, the fluoride release and recharge capabilities of RMGI adhesives make them a potentially attractive option for orthodontists. As already indicated, there is frequently a time delay from bracket placement to manipulation and subsequent adhesive light curing. However, to date, there have been no studies evaluating the effect of bracket manipulation and delayed light polymerization on RMGI adhesives with regard to bond strength. This is of particular importance with a RMGI adhesive, because it sets through a dual-cure process; an acid-base reaction along with the light polymerization reaction. If the adhesive begins to polymerize or set before bracket manipulation occurs, then a decrease in bond strength may result.

Hypotheses

1. The shear bond strength of the resin modified glass ionomer adhesive will vary as a function of delayed polymerization time.
2. The degree of conversion of the resin modified glass ionomer adhesive will vary as a function of delayed polymerization time.
3. The adhesive fracture pattern determined by the Adhesive Remnant Index will vary as a function of delayed polymerization time.

CHAPTER 2

MATERIALS AND METHODS

Tooth Specimen Collection

Extracted premolar teeth are frequently utilized in *in vitro* orthodontic bonding studies because these teeth are commonly extracted as part of an orthodontic treatment plan. Due to limited availability of extracted premolars at the UMKC School of Dentistry, extracted maxillary third molars were used in this study. Tooth specimens with no patient identifiers were collected from various departments within the UMKC School of Dentistry and from oral surgery offices in the Kansas City area. The specimens were stored in containers of phosphate buffered saline¹ (PBS) for up to 4 months prior to testing. PBS was chosen as the storage media because it has been shown to better maintain enamel surface hardness and modulus of elasticity when compared to physiological saline and deionized water (Anjum et al. 2009). Once collected, the teeth were examined and any remaining soft-tissue removed. Teeth with cracks, fluorosis, decay, abnormal morphology, or evidence of surface damage were discarded. After visual inspection, the remaining teeth were stored in PBS with 0.002% sodium azide at 4°C to limit bacterial growth.

Resin Modified Glass Ionomer Adhesive

The RMGI adhesive² used was a dual-cured reinforced glass ionomer orthodontic adhesive that is commercially available to practicing orthodontists. The RMGI adhesive consists of two pastes that were combined prior to placement on the bracket base via an auto-mix syringe system. One paste of the two paste system contains amorphous fluoro-alumino-

¹ Dulbecco's Phosphate Buffered Saline, Sigma-Aldrich, 3050 Spruce St., St. Louis, MO 63103

² Fuji Ortho LC, GC America Inc. 3737 West 127th Street, Alsip, IL 60803 USA

silicate glass, dimethacrylate, silicon dioxide, and urethanedimethacrylate. The second paste contains polyacrylic acid, distilled water, amorphous silicon dioxide, polybasic carboxylic acid, and an initiator. Fluoro-alumino-silicate glass particles and polyacrylic acid are components of conventional glass ionomers that are found in RMGI adhesives (Berzins et al. 2010).

Brackets

Metal maxillary universal premolar brackets³ with a concave bracket base with mesh pad were utilized in this study. Maxillary universal premolar brackets are designed to be used on first or second premolars. Maxillary premolar brackets have been bonded to maxillary third molars in studies evaluating shear bond strength of various adhesives (Chitnis et al. 2006).

Bracket Bonding

To facilitate bracket bond strength testing, each tooth specimen was mounted in self-curing acrylic resin⁴ using a mounting jig and plastic mounting ring⁵ (fig. 1). Each tooth was oriented so that the flattest portion of the mesio-buccal surface was perpendicular to the mounting ring. A level⁶ was used to ensure that the buccal surface of the crown was oriented perpendicular to the horizontal plane. Proper positioning of the teeth allowed for vertical shear force to be applied during shear bond strength testing.

The RMGI adhesive manufacturer's recommendations were followed during testing. The bonding procedure was performed in an environmental chamber at 33°C ($\pm 2^\circ$) and 85%

³ Victory Series™ Low Profile MBT Universal Bicuspid Twin Bracket, 3M Unitek, 2724 South Peck Road, Monrovia, CA 91016

⁴ Biocryl #040-016, Great Lakes, 200 Cooper Ave., Tonawanda, NY 14150

⁵ Item #20-8180, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044

⁶ Johnson Level & Tool Mfg. Co., Inc, 6333 W. Donges Bay Road, Mequon, WI 53092-4456

(±5%) humidity to simulate oral conditions (Plasmans et al. 1994). Immediately prior to bonding, each specimen was polished with fluoride-free pumice⁷, rinsed with water, and dried with oil-free air. Enamel conditioner⁸, a 20% polyacrylic acid, was applied to the buccal surface of the specimen for 10 seconds, and then rinsed and dried with oil-free air. The teeth remained slightly moist as the manufacturer recommends. A jig consisting of digital calipers⁹, protractor and fine tip permanent marker were used to mark the teeth (outside of the bracket bonding area), at 2 locations, to indicate the 10° manipulation (fig. 2). The digital calipers were used to measure the distance from the center to the mesial/distal width of the bracket. That distance and a protractor was then used to mark the tooth at two locations indicating the desired position for initial bracket placement and 10° of manipulation. The adhesive was dispensed onto the bracket base and applied to the mesial-buccal surface of the specimen using a bracket placement instrument. A hand instrument¹⁰ was then used to apply pressure to the bracket allowing excess adhesive to be expressed and removed with the carver. From this point forward, the teeth were treated according to group assigned.

⁷ 1st & Final® pumice , Reliance Orthodontic Products, 1540 West Thorndale Ave, Itasca, IL 60143

⁸ ORTHO GEL CONDITIONER, Fuji Ortho LC,GC America Inc. 3737 West 127th Street, Alsip, IL 60803

⁹ Mitutoyo Corporation, Model # CD-4”CSX, 965 Corporate Boulevard, Aurora, IL 60502

¹⁰ Hollenbeck Carver, CVHL 1/2, Hu-Friedy, 3232 N. Rockwell, Chicago, IL 60618-5982

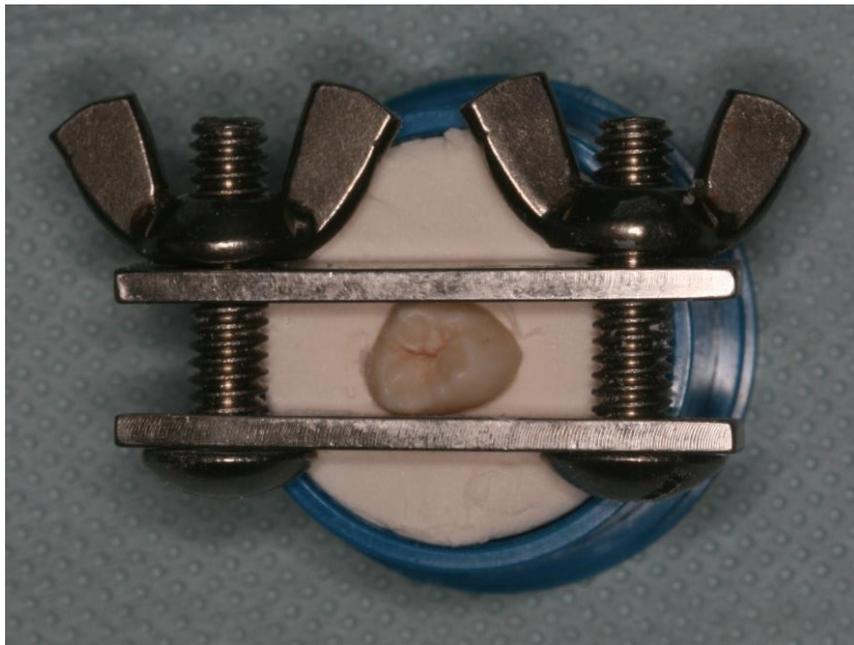


Figure 1. Maxillary third molar mounted in self-cure acrylic resin.

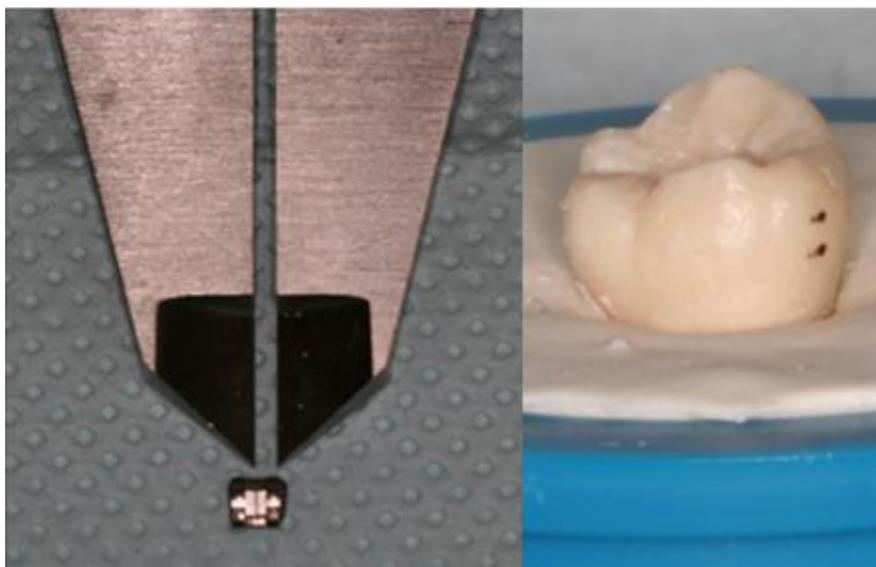


Figure 2. Digital calipers and tooth marked to show 10° manipulation.

Polymerization Protocol

For group one, the bracket was manipulated 10° clockwise using a hand instrument⁷ and then light cured¹¹ 30 seconds (0.5 min) after placement. The light-curing unit was applied for 20 seconds each at the mesial, distal, gingival, and incisal bracket surfaces as recommended by the adhesive manufacturer. Group two followed the same protocol, except that there was a 2-min time delay after bracket placement before bracket manipulation and light curing. Group three followed the same protocol, except that there was a 4-min time delay after bracket placement before bracket manipulation and light curing. The 2-min and 4-min delays were selected based on preliminary testing of the working time of the RMGI adhesive. This data is presented in the appendix. While the working time was reported by the manufacturer to be 5 minutes, preliminary testing showed that manipulation was ideally performed before 4 minutes, when the material becomes firm. Prior to each day's use, the light-curing unit output was measured with a radiometer¹² to ensure consistent output of the curing light.

Following light curing, teeth with bonded brackets were stored for 24-hrs at 37°C in PBS. Waiting 24-hrs allowed the chemical component of the reaction to be completed before shear bond strength testing. A plastic container with a plastic 1.5 cm x 1.5 cm x 1.5 cm grid¹³ was used with the mounted tooth inverted onto the plastic grid so only the crown of the tooth was submerged in the PBS solution (fig. 3). The container with teeth in PBS solution was covered with a plastic film coating¹⁴ during the 24-hr storage period.

¹¹ Ortholux™ LED Curing Light, 3M Unitek, 2724 South Peck Road, Monrovia, CA 91016

¹² OrthoLux LED Radiometer, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

¹³ Egg Crate Lighting Panel Model # LP2448EGG-5, The Home Depot, Kansas City, MO 64111

¹⁴ Parafilm M, SPI Supplies/Structure Probe, Inc., West Chester, PA 19380

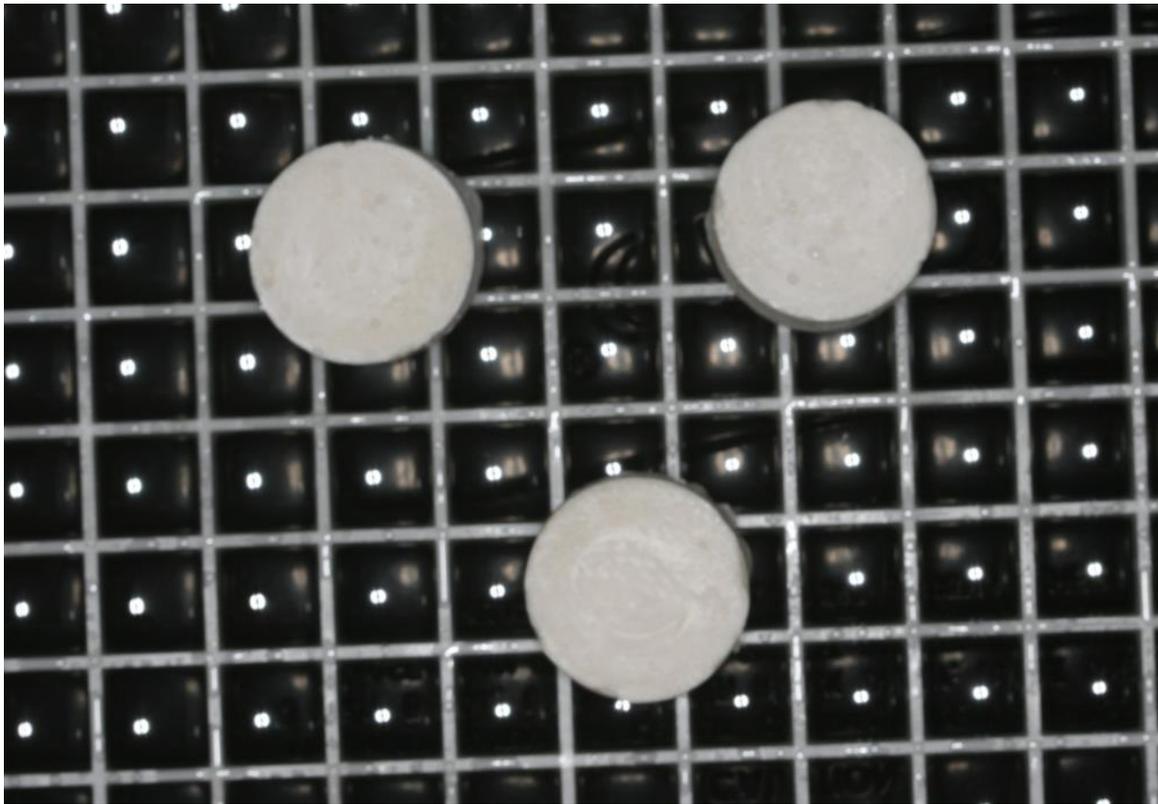


Figure 3. Plastic storage container with grid for bonded specimens. Tooth crowns/bonded brackets submerged in PBS for 24-hr storage at 37°C.

Shear Bond Strength

After 24-hr storage in PBS at 37°C, SBS testing was performed using a universal testing machine¹⁵. The mounted specimen was secured to the universal testing machine platform. The knife-edge rod attachment of the universal testing machine crosshead was positioned at the occlusal edge of the bonded bracket base so that the load was applied in the occlusogingival direction paralleling the buccal surface of the tooth (fig. 4). The crosshead speed was set at 1mm/1min to apply shear force to the bracket-tooth interface. The maximum force applied when the bracket was sheared off the enamel surface was recorded in

¹⁵ Model 5967, Instron Corporation, 825 University Ave., Norwood MA 02062-2643

Newtons (N). The shear bond strength was determined using the equation: Shear Bond Strength (MPa) = debonding force (N) /surface area of bracket base (mm²) (Rajagopal et al. 2004). The bracket base surface area (mm²) = width of bracket base (mm) * height of bracket base (mm) = 3.45 * 3.01= 10.38mm². A representative load/displacement curve is presented in figure 5.



Figure 4. Bonded, mounted tooth ready for shear bond strength testing.

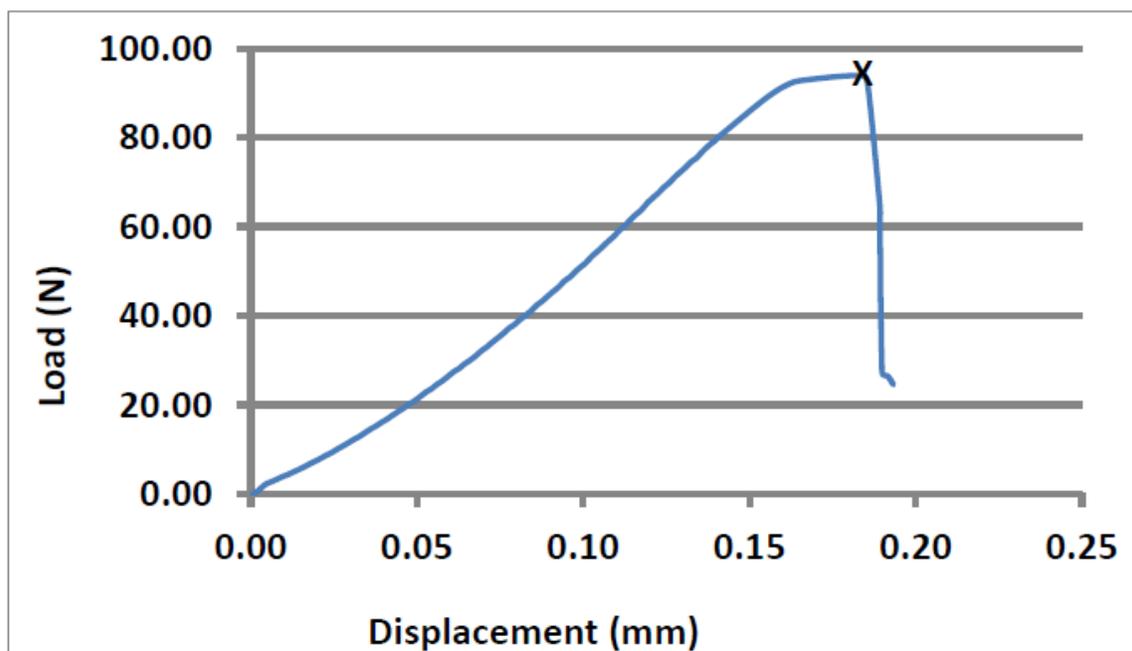


Figure 5. Representative load-displacement graph for a bracket debonding test. Maximum load was used to calculate shear bond strength.

Degree of Conversion Measurements

Raman microspectroscopy data was collected within 60 minutes after SBS testing so that the degree of conversion measurements corresponded to the actual degree of conversion of the adhesive close to the time of shear bond testing. Point measurements of the adhesive were recorded at three locations on the bracket base. However, only 34 of 123 spectra were acceptable due to interfering fluorescence of the bonding material and lack of visible peaks on the spectra.

For the 34 spectra, DC was determined using the following equation adapted for RMGI: $DC (\%) = 100 * [1 - (R_{\text{polymerized}} / R_{\text{unpolymerized}})]$, where R = peak area at 1639 cm^{-1} / peak area at 1709 cm^{-1} (Pianelli et al. 1999). Figure 6 includes representative examples of

unpolymerized and polymerized Raman microspectroscopy spectra that was obtained during preliminary testing.

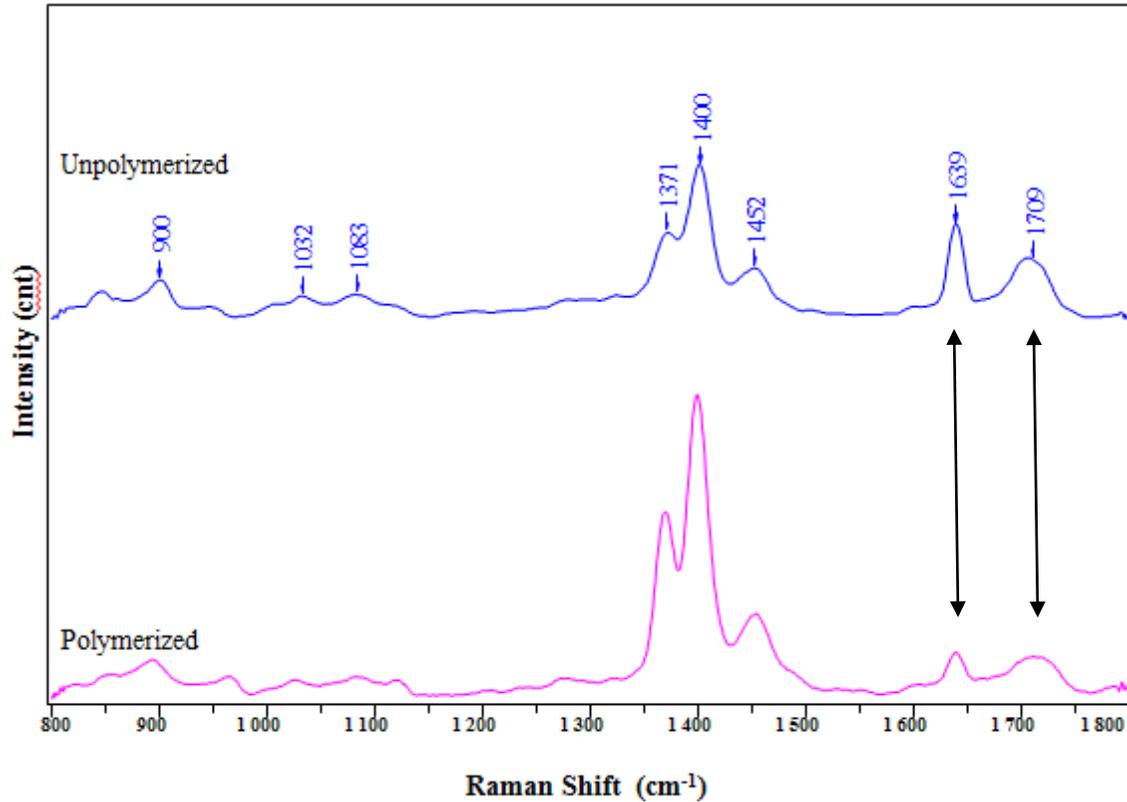


Figure 6. Representative Raman microspectroscopy spectra. Unpolymerized and polymerized RMGI. Peaks at 1639 and 1709 cm^{-1} (arrows) were used for degree of conversion calculations.

Adhesive Remnant Index

Photos were taken of the debonded brackets immediately prior to Raman microspectroscopy analyses. The photos were used to implement the 4-point ARI scale to describe the location of bond failure and the subsequent location of the adhesive (fig. 7). The scale was defined as: 0 = all adhesive remains on the bracket base, 1 = >50% of the adhesive remains on the bracket base, 2 = <50% of the adhesive remains on the bracket base, and 3 = no adhesive remains on the bracket base (Artun and Bergland 1984). A transparent grid was

placed over the image to allow for accurate determination of the amount of adhesive remaining on the bracket base.

Prior to ARI evaluations, the examiner (DG) was calibrated. Ten photographs of debonded bracket bases were scored on two different occasions, 48 hours apart. The examiner was blinded to identifying information for each image. ARI scores were determined for each image and intra-rater reliability was calculated. There was 100% agreement between the two scoring sessions.

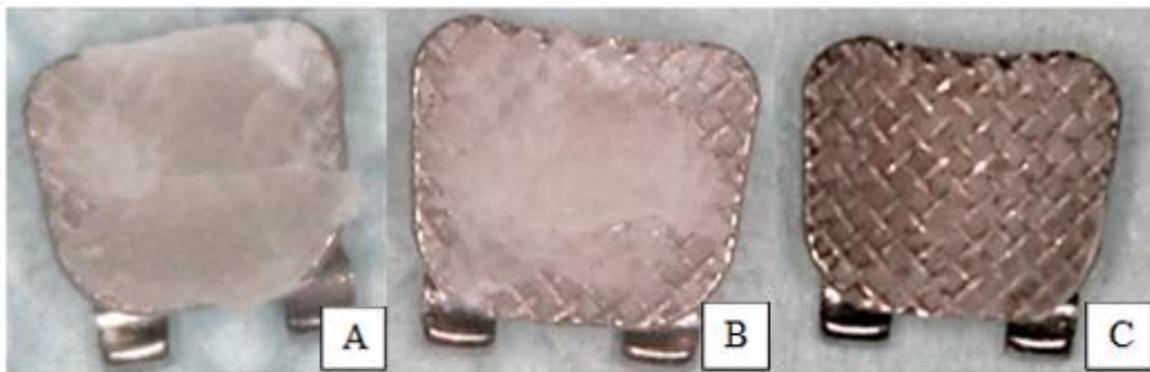


Figure 7. Representative images of debonded bracket bases assigned ARI Scores. ARI Score 0 (A), ARI Score 1 (B), and ARI Score 2 (C).

Experimental Design and Sample Size

This study utilized a 1-factor design with the independent variable of delay time with three levels. A sample of 10 teeth was randomly assigned to each of the three experimental groups for this pilot study (N=30). The dependent variables measured were shear bond strength (MPa), degree of conversion (%), and the Adhesive Remnant Index score (0-3). The experimental design is presented in Table 1.

TABLE 1
EXPERIMENTAL DESIGN

Experimental Group (N=10 teeth/group)	Delay Time	Shear Bond Strength (MPa)	Degree of Conversion (%)	Adhesive Remnant Index Score
1	0.5 min			
2	2 min			
3	4 min			

Data Analysis

A one-factor ANOVA was used to analyze the dependent variables of shear bond bracket strength as a function of delay time. To determine if the ARI scores vary as a function of delay time, a Kruskal-Wallis one-way analysis of variance by ranks was used. A Mann-Whitney paired comparison was included as a post-hoc evaluation approach. A Spearman correlation was done to test any relationship between shear bond strength and ARI scores. All statistical analyses was performed using a statistical analysis software program¹⁶ with significance set at $\alpha = 0.05$ for all testing.

¹⁶ SPSS version 21, 233 S. Wacker Dr., Chicago IL 60606

CHAPTER 3

RESULTS

Shear Bond Strength Measurements

Means and standard deviations of shear bond strength for the three testing groups are presented in figure 8. Based on the 1-factor ANOVA, there was no significant difference ($p > 0.05$) in the shear bond strength between the delay times. This did not support the hypothesis that shear bond strength of the resin modified glass ionomer would vary as a function of delayed polymerization time.

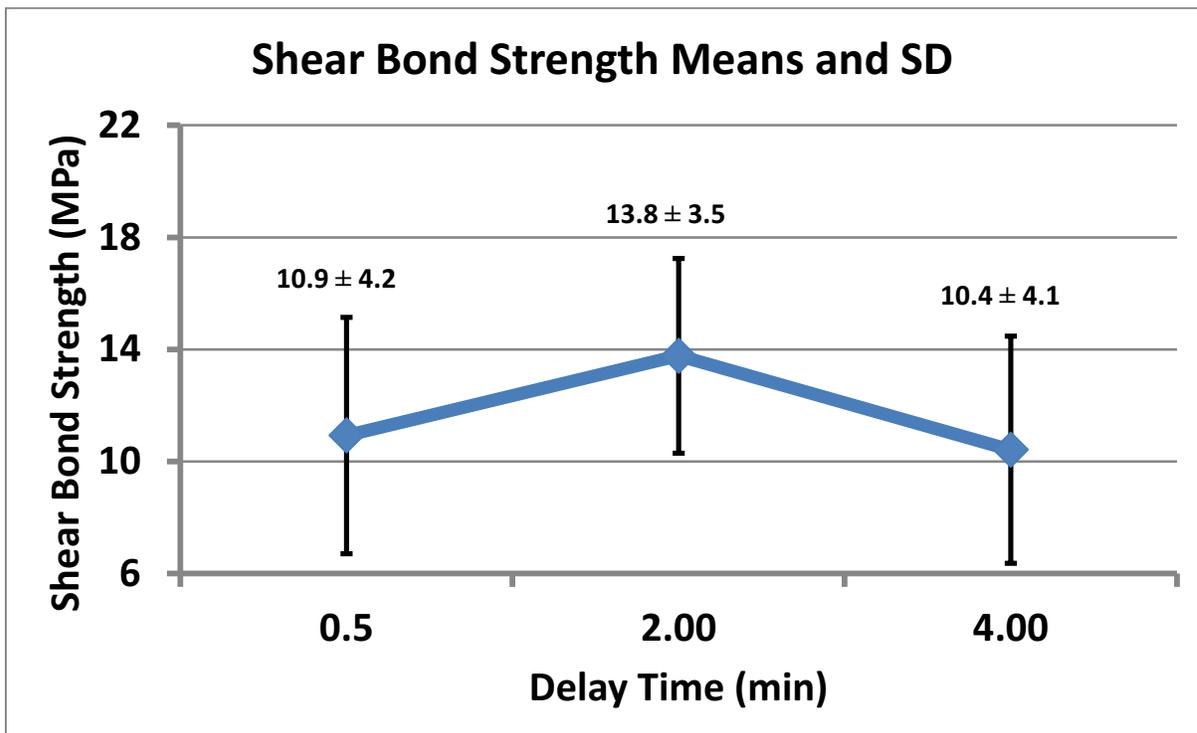


Figure 8. Means and standard deviations of shear bond strength measures. There were no statistically significant differences between the time delay groups.

Degree of Conversion Measurements

The data for degree of conversion was collected for all specimens. However, only 34 of 123 spectra were acceptable due to interfering fluorescence of the bonding material and lack of visible peaks on the spectra. The acceptable spectra were not equally distributed among the experimental groups with 15 acceptable spectra from the 0.5-min delay group, 9 acceptable spectra from the 2-min delay group and 10 acceptable spectra from the 4-min delay group. Because of this complication, DC was not a reliable evaluation measure and was not included in the data analysis. The hypothesis that degree of conversion of the resin modified glass ionomer would vary as a function of delayed polymerization time could not be tested.

Adhesive Remnant Index Measurements

The ARI results are presented in table 2. Representative images of each ARI score are shown in figure 7. Based on the Kruskal-Wallis one-way ANOVA, there was a significant difference in ARI scores as a function of delay time. The Mann-Whitney post hoc comparisons indicated that the significant difference ($p < 0.05$) was between the 0.5-min and 4.0-min delay groups. As noted in the table, there were more ARI scores of 2 in the 0.5-min delay group (30%) as compared to the 4.0-min delay group (0%). Additionally, there were fewer ARI scores of 0 in the 0.5-min delay group (10%) as compared to the 4.0-min delay group (60%). Collectively, these values indicate that with increasing time the ARI scores tend to decrease. The data supported the hypothesis that the adhesive fracture pattern as determined by the ARI would vary as a function of delayed polymerization time.

TABLE 2

ADHESIVE REMNANT INDEX MEASUREMENTS

Delay Time Group*	Number of specimens (%) with each ARI Score per group		
	ARI Score 0	ARI Score 1	ARI Score 2
0.5 minute	1/10 (10%)	6/10 (60%)	3/10 (30%)
2 minute	3/10 (30%)	6/10 (60%)	1/10 (10%)
4 minute	6/10 (60%)	4/10 (40%)	0/10 (0%)

*There was a significant difference ($p < 0.05$) in ARI scores between 0.5 and 4.0 min delay.

Correlation Between Shear Bond Strength and Adhesive Remnant Index

We were not able to complete a correlation between SBS and DC. However, the Spearman correlation between SBS and ARI indicated no positive association between SBS and ARI measures across delay times.

CHAPTER 4

DISCUSSION

The fluoride release and recharge capabilities of RMGI adhesives make them a potentially attractive material for bonding orthodontic appliances. Multiple studies have investigated the SBS of RMGI adhesives and have concluded that the strength is sufficient for successful bracket bonding (Komori and Ishikawa 1997; Rix et al. 2001; Chitnis et al. 2006). For this material to be clinically useful, it is also important to study the effect of time delay that occurs from bracket placement to manipulation and subsequent light curing. Therefore, this study examined the effect of bracket manipulation and light curing after varying delay times on the SBS, DC, and adhesive fracture pattern of a RMGI adhesive.

Shear Bond Strength

The results of this study showed that there was no significant difference in the SBS between the 0.5, 2, or 4-min delay groups. The data did not support the hypothesis that the SBS of the RMGI adhesive would vary as a function of delayed polymerization time. Currently, there is the perception among many orthodontists that the SBS decreases as the delay from bracket placement to manipulation and light curing increases due to partial curing of the adhesive before the bracket has been manipulated into the final position. It was speculated that manipulating the bracket after a time delay might disrupt any bonds that developed due to ambient light polymerization; thus, negatively impacting the bond strength. However, this study does not support that theory.

Previous investigations have reported that the SBS of RMGI ranges from 3-21 MPa (Bishara et al. 2000; Sfondrini et al. 2001; Valente et al. 2002; Cacciafesta et al. 2004; Cheng

et al. 2011). The SBS values in the current study were 10.4-13.8 MPa, and fall into the range of previous reports. However, it is important to compare our results with studies of similar design. For example, studies that did not include delayed polymerization but used a similar bracket bonding and debonding protocol can be compared to the current 0.5-min delay group. The 0.5-min delay group of the current study had a SBS of 10.9 ± 4.2 MPa. Previous studies of similar design reported SBS of 7.9-12.4 MPa, and values of the current study fall within this range (Cacciafesta et al. 2004; Summers et al. 2004; Al Shamsi et al. 2006).

A 2012 study investigated the effect of various delay times (0-min, 2.5-min, 5-min, and 10-min) on the SBS of a RMGI adhesive, and found that there was no significant differences between the groups. The SBS of the groups ranged from 12.8-14.8 MPa, which is similar to the values reported in the current study. However, the 2012 study differs from the current study in two ways: the specimens were shielded from ambient light during the delay and bracket manipulation was not performed (Thomas et al. 2012). The current study was the first to investigate both the effect of clinically relevant delays from bracket placement to light curing, while the specimens were exposed to ambient light, and bracket manipulation on the SBS of a RMGI adhesive. Therefore, the current study provides SBS data that may be more applicable to the clinical use of RMGI adhesives than previous studies.

Degree of Conversion

Studies evaluating conventional resin adhesives frequently analyze the DC using Raman microspectroscopy; however, this is not routinely done for RMGI adhesives. As previously discussed, RMGI adhesives undergo two competing reactions during the curing

process, a light-initiated polymerization reaction and an acid-base reaction (Silverman et al. 1995; Sfondrini et al. 2001; Berzins et al. 2010; Cheng et al. 2011). Therefore, quantifying the DC for RMGI adhesives is more complicated than analyzing conventional resin adhesives (Young et al. 2000). Only a few studies have quantified DC of RMGI, but all of those investigations included FTIR spectroscopy (Kakaboura et al. 1996; Young 2002) rather than Raman microspectroscopy.

Researchers using FTIR spectroscopy reported approximately 90% of the monomer is polymerized but less than 50% of the polyacid is neutralized; and that both values vary based on distance of the adhesive from the light-cure unit and exposure of the adhesive to water during setting (Young 2002). In contrast, Kakaboura et al reported that 35-55% of carbon-carbon double bonds remain intact after light curing (Kakaboura et al. 1996), indicating a DC of 45-65%. It was also reported that more carbon-carbon double bonds remain intact (35-70%) when the specimen was light-cured after 20-min of dark storage (Kakaboura et al. 1996). This suggests that DC decreased to approximately 30-65% when a delay occurred from mixing of the RMGI to light curing. The results of studies that utilize FTIR provide some information regarding the DC of RMGI adhesives, but are not directly applicable to the current study.

While one of the goals of this study was to be the first study to quantify DC of RMGI using Raman microspectroscopy, several challenges, including weak peak signal with and without fluorescence, were encountered and as a result, DC could not be determined. The majority of spectra demonstrated a high level of background noise, likely from interfering fluorescence which overpowered the Raman microspectroscopy signal. In fact, only 34 of 123 spectra collected were useable. According to the author of *Spectroscopy of Polymers*,

approximately 95% of polymers exhibit fluorescence when Raman microspectroscopy is used in the visible excitation region (Koenig 1999). One method of reducing fluorescence is to use the Raman microspectroscopy laser in the near infra-red range, which was done in the current study, but did not seem to reduce the fluorescence adequately. Furthermore, a study by Young et al. demonstrated that Raman spectra peaks may exhibit altered appearance due to the complex polymer environment influencing the conformation of compounds. Young et al. also found that when combining components of a glass ionomer adhesive (glass, tartaric acid, and water) the Raman spectra showed broad weak peaks and a high level of background noise (Young et al. 2000). Based on those previous results, perhaps it is not surprising that the majority of spectra in this study included background noise. Moreover, based on these outcomes, Raman microspectroscopy may not be a useful tool for measuring RMGI adhesive DC.

Adhesive Remnant Index

The current study found a significant difference in the ARI score between delay groups of 0.5 and 4-min. The 0.5-min delay group was more likely to have an ARI score of 2 (30%), when compared to the 4-min delay group (0%). An ARI score of 2 indicates that the majority of the adhesive remained on the tooth after debonding. This suggests that a short delay time (0.5-min) may increase the likelihood that a large amount of adhesive will remain on the tooth after debonding. The brackets bonded with a 4-min delay were more likely to have an ARI score of 0 (60%), when compared to the 0.5-min delay group (10%). An ARI score of 0 indicates that there was almost no adhesive remaining on the tooth after debonding. Additionally, all specimens in the 4-min delay group had at least 50% of the

adhesive remain on the bracket base, rather than on the tooth, after debonding. With increasing time, the ARI scores tend to decrease. The results suggest that less adhesive remained on the tooth surface after debonding when the delay time was increased to 4.0-min. This could be beneficial in that less adhesive needs to be removed from enamel by grinding at the time of bracket removal when orthodontic treatment is completed. Additionally, with less adhesive requiring manual removal from the enamel surface, the risk of damaging the enamel may be reduced. There was no significant difference in the ARI scores for the 2-min delay group when compared to the 0.5 and 4-min delay groups.

There are several possible explanations for the difference in adhesive fracture pattern between the 0.5 and 4-min delay groups. It is likely that the specimens in the 4-min group had undergone a greater amount of adhesive setting than the 0.5-min group before the bracket was manipulated. The specimens in the 4-min group were exposed to ambient light for a greater amount of time before bracket manipulation. The ambient light may have started the light-initiated polymerization reaction that occurs in the adhesive. Additionally, the acid-base reaction of the adhesive begins once the adhesive is mixed. Therefore, it is probable that the acid-base reaction had proceeded to a greater degree in the 4-min group at the time of bracket manipulation, when compared to the 0.5-min group. Furthermore, moisture from the humidity in the environmental chamber may have accumulated on the enamel surface as the time delay increased, potentially weakening the enamel-adhesive interface in the 4-min group.

While no study has used the identical protocol of the current study, three studies evaluating RMGI adhesive without a delay (Komori and Ishikawa 1997; Rix et al. 2001; Chitnis et al. 2006) from bracket placement to light curing could be compared to the 0.5 min

group. Two of those studies reported that most specimens were assigned an ARI score of 0 or 1 (Rix et al. 2001; Chitnis et al. 2006), which is in agreement with the current study in which 70% of specimens were assigned an ARI score of 0 or 1, while another study (Komori and Ishikawa 1997) found that most specimens were assigned an ARI score of 2 (Komori and Ishikawa 1997). Only one previous study included various polymerization delays (immediate, 2.5-min, 5-min, and 10-min) and reported no difference in ARI scores between delay groups (Thomas et al. 2012). However, specimens were shielded from ambient light, so a direct comparison to the current study is not possible.

Correlation Between Shear Bond Strength and Adhesive Remnant Index

There was no positive association between SBS and ARI measures across delay times. Therefore, the adhesive fracture pattern did not impact the shear bond strengths that were observed.

Study Limitations

This study utilized maxillary third molars and maxillary premolar brackets. Because the bracket base was specifically designed to optimally bond to premolars, the curvature of the bracket may not have fit ideally on the maxillary third molars buccal surface. Reduced adaptation of the bracket base to the enamel surface may have impacted the SBS and ARI results. Although the teeth were carefully examined for defects, enamel irregularities may have been present, specifically since the teeth had undergone extraction.

To more closely simulate oral conditions, an environmental chamber with controlled humidity and temperature was used during bracket bonding. After bonding, the teeth were stored in PBS solution at 37°C for 24-hrs before SBS testing. While those conditions were

valuable to the study, the potential impact of actual saliva on the RMGI adhesive was not examined.

The brackets were debonded using a cross-head speed of 1mm/min. This was done for the purpose of comparison to similar studies. The cross-head speed does not imitate the forces that brackets experience intra-orally. Additionally, brackets bonded intraorally experience forces almost immediately after bonding, without a 24-hr delay.

Future Investigations

The current study demonstrated that a delay from bracket placement to manipulation and light curing does not significantly reduce the SBS when using metal brackets under simulated oral conditions. However, many practitioners use ceramic brackets due to their more esthetic appearance. Ceramic brackets are translucent and allow light to penetrate the bracket to the adhesive. Future studies could investigate whether a delay from bracket placement to manipulation and light curing has detrimental effects on the SBS for ceramic brackets when using a RMGI adhesive.

Enamel protective sealants consisting of unfilled resins have been marketed as another approach to preventing enamel decalcification adjacent to orthodontic brackets. Orthodontic brackets may be bonded onto the surface of the sealant, or the sealant may be applied after brackets are bonded to the teeth. Orthodontists interested in reducing the frequency and severity of enamel decalcification would likely be interested in using a RMGI adhesive along with a protective sealant applied to the facial surface of bonded teeth. Utilizing both methods of decalcification prevention could be beneficial for patients who continue to exhibit poor oral hygiene despite patient education and encouragement. Future

studies could investigate the effect that an enamel sealant has on the SBS of brackets bonded with RMGI adhesive. Future studies could also explore the effectiveness of combining the use of RMGI adhesive and enamel protective sealants on the prevention of enamel decalcification.

Clinical Implications

The current study showed that SBS values were not significantly altered when a delay of up to 4-min occurs between bracket placement and final manipulation and light curing when using a RMGI adhesive. This is relevant to many clinicians whose bracket bonding protocol includes bracket placement by dental auxiliary staff, and then final bracket manipulation and light curing by the orthodontist. In this scenario, a delay from bracket placement to manipulation and light curing is inevitable.

The current study demonstrated that specimens in the 4-min delay group tended to have less adhesive adhere to the enamel surface after bracket debonding, compared to the 0.5-min delay group. Therefore, clinicians may wish to implement a delay time of approximately 4-min before manipulation and light curing so that less adhesive remains on the enamel surface when brackets are debonded. Clinicians may find this desirable because there will be less time required to manually remove the adhesive from the enamel surface when brackets are removed after orthodontic treatment is complete. Additionally, with less adhesive requiring manual removal, the risk of damaging the enamel may be reduced.

CHAPTER 5

CONCLUSIONS

1. There was no significant difference in shear bond strength between delay groups of 0.5, 2, and 4-min.
2. The degree of conversion of the resin modified glass ionomer adhesive was analyzed; however, the data was not sufficient to determine if the degree of conversion varied as a function of delayed polymerization time.
3. There was a significant difference in the Adhesive Remnant Index scores between delay groups of 0.5 and 4-min. The 0.5-min delay group was more likely to have an ARI score of 2 (30%), when compared to the 4-min delay group (0%). The 4-min delay group was more likely to have an ARI score of 0 (60%), when compared to the 0.5-min delay group (10%). There was no significant difference in the ARI score for the 2-min delay group when compared to the 0.5 and 4-min delay groups.

LITERATURE CITED

- Al Shamsi A, Cunningham JL, Lamey PJ, Lynch E. Shear bond strength and residual adhesive after orthodontic bracket debonding. *Angle Orthod* 2006;76:694-9.
- Anjum A, Otsuki M, Matin K, Tagami J. Preservation in the liquid media produces alterations in enamel surface properties. *J Dent* 2009;37:884-90.
- Arici S, Arici N. Effects of thermocycling on the bond strength of a resin-modified glass ionomer cement: An in vitro comparative study. *Angle Orthod* 2003;73:692-6.
- Arici S, Caniklioglu CM, Arici N, Ozer M, Oguz B. Adhesive thickness effects on the bond strength of a light-cured resin-modified glass ionomer cement. *Angle Orthod* 2005;75:254-9.
- Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. *Am J Orthod* 1984;85:333-40.
- Berzins DW, Abey S, Costache MC, Wilkie CA, Roberts HW. Resin-modified glass-ionomer setting reaction competition. *J Dent Res* 2010;89:82-6.
- Bishara SE, VonWald L, Laffoon JF, Jakobsen JR. Effect of changing enamel conditioner concentration on the shear bond strength of a resin-modified glass ionomer adhesive. *Am J Orthod Dentofacial Orthop* 2000;118:311-6.
- Bishara SE, Ostby AW, Laffoon JF, Warren J. Shear bond strength comparison of two adhesive systems following thermocycling. A new self-etch primer and a resin-modified glass ionomer. *Angle Orthod* 2007;77:337-41.
- Braga RR, Mitra SB. Materials for adhesion and luting. In: Craig's restorative dental materials. JM Powers and RL Sakaguchi editors Philadelphia, PA: Mosby, Inc.; 2012, pp. 327-47.
- Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955;34:849-53.
- Cacciafesta V, Sfondrini MF, Scribante A, De Angelis M, Klersy C. Effect of blood contamination on shear bond strength of brackets bonded with a self-etching primer combined with a resin-modified glass ionomer. *Am J Orthod Dentofacial Orthop* 2004;126:703-8.
- Cerveira GP, Berthold TB, Souto AA, Spohr AM, Marchioro EM. Degree of conversion and hardness of an orthodontic resin cured with a light-emitting diode and a quartz-tungsten-halogen light. *Eur J Orthod* 2010;32:83-6.

- Cheng HY, Chen CH, Li CL, Tsai HH, Chou TH, Wang WN. Bond strength of orthodontic light-cured resin-modified glass ionomer cement. *Eur J Orthod* 2011;33:180-4.
- Chitnis D, Dunn WJ, Gonzales DA. Comparison of in-vitro bond strengths between resin-modified glass ionomer, polyacid-modified composite resin, and giomer adhesive systems. *Am J Orthod Dentofacial Orthop* 2006;129:330 e11-6.
- Chung CK, Millett DT, Creanor SL, Gilmour WH, Foye RH. Fluoride release and cariostatic ability of a compomer and a resin-modified glass ionomer cement used for orthodontic bonding. *J Dent* 1998;26:533-8.
- Corekci B, Malkoc S, Ozturk B, Gunduz B, Toy E. Polymerization capacity of orthodontic composites analyzed by fourier transform infrared spectroscopy. *Am J Orthod Dentofacial Orthop* 2011;139:e299-304.
- Croll TP, Nicholson JW. Glass ionomer cements in pediatric dentistry: Review of the literature. *Pediatr Dent* 2002;24:423-9.
- Eliades T, Brantley WA. The inappropriateness of conventional orthodontic bond strength assessment protocols. *Eur J Orthod* 2000;22:13-23.
- Eliades T, Eliades G, Bradley TG, Watts DC. Degree of cure of orthodontic adhesives with various polymerization initiation modes. *Eur J Orthod* 2000;22:395-9.
- Eliades T. Orthodontic materials research and applications: Part 1. Current status and projected future developments in bonding and adhesives. *Am J Orthod Dentofacial Orthop* 2006;130:445-51.
- Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU. In-vitro orthodontic bond strength testing: A systematic review and meta-analysis. *Am J Orthod Dentofacial Orthop* 2010;137:615-22 e3.
- Forsten L. Resin-modified glass ionomer cements: Fluoride release and uptake. *Acta Odontol Scand* 1995;53:222-5.
- GC America. GC Fuji Ortho LC paste pak automix instructions. Tokyo, Japan; 2010.
- Godoy-Bezerra J, Vieira S, Oliveira JH, Lara F. Shear bond strength of resin-modified glass ionomer cement with saliva present and different enamel pretreatments. *Angle Orthod* 2006;76:470-4.
- Hegarty DJ, Macfarlane TV. In vivo bracket retention comparison of a resin-modified glass ionomer cement and a resin-based bracket adhesive system after a year. *Am J Orthod Dentofacial Orthop* 2002;121:496-501.

- Kakaboura A, Eliades G, Palaghias G. An FTIR study on the setting mechanism of resin-modified glass ionomer restoratives. *Dent Mater* 1996;12:173-8.
- Keim RG, Gottlieb EL, Nelson AH, Vogels DS, 3rd. 2008 jco study of orthodontic diagnosis and treatment procedures, part 1: Results and trends. *J Clin Orthod* 2008;42:625-40.
- Koenig JL. Raman spectroscopy of polymers. In: *Spectroscopy of polymers* 2nd ed. New York, NY: Elsevier Science Inc.; 1999, pp. 215.
- Komori A, Ishikawa H. Evaluation of a resin-reinforced glass ionomer cement for use as an orthodontic bonding agent. *Angle Orthod* 1997;67:189-95.
- McLean JW, Nicholson JW, Wilson AD. Proposed nomenclature for glass-ionomer dental cements and related materials. *Quintessence Int* 1994;25:587-9.
- Miguel JA, Almeida MA, Chevitarrese O. Clinical comparison between a glass ionomer cement and a composite for direct bonding of orthodontic brackets. *Am J Orthod Dentofacial Orthop* 1995;107:484-7.
- Millett DT, Glenny AM, Mattick CR, Hickman J, Mandall NA. Adhesives for fixed orthodontic bands. *Cochrane Database Syst Rev* 2007:CD004485.
- Mitra SB, Sakaguchi RL. Restorative materials- composites and polymers. In: *Craig's restorative dental materials*. JM Powers and RL Sakaguchi editors. 13th ed. Philadelphia, PA: Mosby, Inc.; 2012, pp. 161-98.
- Montasser MA, Drummond JL. Reliability of the adhesive remnant index score system with different magnifications. *Angle Orthod* 2009;79:773-6.
- Murfitt PG, Quick AN, Swain MV, Herbison GP. A randomised clinical trial to investigate bond failure rates using a self-etching primer. *Eur J Orthod* 2006;28:444-9.
- Newman RA, Newman GV, Sengupta A. In vitro bond strengths of resin modified glass ionomer cements and composite resin self-cure adhesives: Introduction of an adhesive system with increased bond strength and inhibition of decalcification. *Angle Orthod* 2001;71:312-7.
- Nicholson JW. Chemistry of glass-ionomer cements: A review. *Biomaterials* 1998;19:485-94.
- Norevall LI, Marcusson A, Persson M. A clinical evaluation of a glass ionomer cement as an orthodontic bonding adhesive compared with an acrylic resin. *Eur J Orthod* 1996;18:373-84.

- Pearson GJ, Atkinson AS. Long-term flexural strength of glass ionomer cements. *Biomaterials* 1991;12:658-60.
- Pereira TB, Jansen WC, Pithon MM, Souki BQ, Tanaka OM, Oliveira DD. Effects of enamel deproteinization on bracket bonding with conventional and resin-modified glass ionomer cements. *Eur J Orthod* 2013;35:442-6.
- Pianelli C, Devaux J, Bebelman S, Leloup G. The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins. *J Biomed Mater Res* 1999;48:675-81.
- Plasmans PJ, Creugers NH, Hermsen RJ, Vrijhoef MM. Intraoral humidity during operative procedures. *J Dent* 1994;22:89-91.
- Powers JM, Sakaguchi RL. Bonding to dental substrates. In: *Craig's restorative dental materials*. JM Powers and RL Sakaguchi editors. 12th ed. St. Louis, MO: Mosby, Inc.; 2006, pp. 213-34.
- Rajagopal R, Padmanabhan S, Gnanamani J. A comparison of shear bond strength and debonding characteristics of conventional, moisture-insensitive, and self-etching primers in vitro. *Angle Orthod* 2004;74:264-8.
- Read MJ. The bonding of orthodontic attachments using a visible light cured adhesive. *Br J Orthod* 1984;11:16-20.
- Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod* 1975;2:171-8.
- Rix D, Foley TF, Mamandras A. Comparison of bond strength of three adhesives: Composite resin, hybrid gic, and glass-filled gic. *Am J Orthod Dentofacial Orthop* 2001;119:36-42.
- Sfondrini MF, Cacciafesta V, Pistorio A, Sfondrini G. Effects of conventional and high-intensity light-curing on enamel shear bond strength of composite resin and resin-modified glass-ionomer. *Am J Orthod Dentofacial Orthop* 2001;119:30-5.
- Silverman E, Cohen M, Demke RS, Silverman M. A new light-cured glass ionomer cement that bonds brackets to teeth without etching in the presence of saliva. *Am J Orthod Dentofacial Orthop* 1995;108:231-6.
- Summers A, Kao E, Gilmore J, Gunel E, Ngan P. Comparison of bond strength between a conventional resin adhesive and a resin-modified glass ionomer adhesive: An in vitro and in vivo study. *Am J Orthod Dentofacial Orthop* 2004;126:200-6; quiz 54-5.
- Tavas MA, Watts DC. Bonding of orthodontic brackets by transillumination of a light activated composite: An in vitro study. *Br J Orthod* 1979;6:207-8.

- Thomas JT, Roberts HW, Diaz L, Bradley TG, Berzins DW. Effect of light-cure initiation time on polymerization efficiency and orthodontic bond strength with a resin-modified glass-ionomer. *Orthod Craniofac Res* 2012;15:124-34.
- Unitek M. Bonding technique guide. Monrovia, CA: 3M Unitek; 2012.
- Valente RM, De Rijk WG, Drummond JL, Evans CA. Etching conditions for resin-modified glass ionomer cement for orthodontic brackets. *Am J Orthod Dentofacial Orthop* 2002;121:516-20.
- Wilson AD, Kent BE. A new translucent cement for dentistry. The glass ionomer cement. *Br Dent J* 1972;132:133-5.
- Wright AB, Lee RT, Lynch E, Young KA. Clinical and microbiologic evaluation of a resin modified glass ionomer cement for orthodontic bonding. *Am J Orthod Dentofacial Orthop* 1996;110:469-75.
- Young AM, Sherpa A, Pearson G, Schottlander B, Waters DN. Use of raman spectroscopy in the characterisation of the acid-base reaction in glass-ionomer cements. *Biomaterials* 2000;21:1971-9.
- Young AM. Ftir investigation of polymerisation and polyacid neutralisation kinetics in resin-modified glass-ionomer dental cements. *Biomaterials* 2002;23:3289-95.
- Zachrisson BU, Buyukyilmaz T. Bonding in orthodontics. In: *Orthodontics: Current principles and techniques*. LW Graber, RL Vanarsdall and KWL Vig editors. 5th ed. Philadelphia, PA: Mosby; 2012, pp. 727-84.

APPENDIX

PRELIMINARY DATA COLLECTION

Fuji Ortho LC adhesive is reported to have a working time of 5 minutes (GC America 2010). The working time was tested using etched glass slides. The adhesive was applied to a metal bracket base, placed onto the etched glass slide, and excess adhesive removed. The bracket was not light cured. The bracket was then manipulated at various time points to determine the consistency of the adhesive and whether bracket manipulation was possible. Table 1 shows the adhesive's physical properties and bracket movability over time. Time was measured from when the bracket was placed onto the glass slide.

TABLE 1

WORKING TIME OF RESIN MODIFIED GLASS IONOMER ADHESIVE

Time (minutes)	0.5	1	2	3	4	5
Bracket manipulation	Movable	Movable	Movable	Movable	Slightly movable	Not moveable without breaking the bond
Physical state of adhesive	Liquid	Liquid	Liquid	Semi-solid	Firm semi-solid	Solid

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