

INTRODUCTION

Dentin bonding requires a conditioner, a primer, and a bonding agent. Traditional (etch/rinse) dentin bonding agents consist of separate steps to condition, prime, and bond. The newest self-etching/self-adhesive systems combine all of the agents in one step. The traditional system requires rinsing after conditioning. This exposes the collagen of the demineralized dentin. The primer and the bonding agents then penetrate the surface and create a resin-dentin hybrid layer. This zone of resin diffusion provides predominantly micro-mechanical bonding of the resin to the dentin. Problems with bonding may arise if there is inadequate penetration of the primer and bonding agent due to deep demineralization, collagen collapse from desiccation, or an over wet substrate leading to a compromised bond. The combination of components of an adhesive system into one step minimizes the potential for these problems. The primer and bonding agent penetrates at the same time and is limited by the depth of the acidic primer demineralization.¹ The smear layer is dissolved and becomes incorporated into the hybrid layer. This incorporation of the smear layer into the hybrid layer may weaken the bond. The etch and rinse systems have a longer history of use and also yield higher enamel bond strengths compared to the newer self-etching/self-adhesive systems.¹⁻³ However, recently, there have been some indications that the bond strengths and other mechanical properties of some self-etching/self-adhesive systems approach those of the etch and rinse systems in dentin.^{1,3-8} Laboratory testing is used to determine the differences in some mechanical properties that may influence clinical performance, and thus, provide knowledge to help aid in material selection.^{1,5}

The luting of indirect dental restorations is a critical step in determining their success. There are many options to choose from when deciding what type of luting agent

to use. The newest options include the so-called “universal” self-etching/self-adhesive resin luting agents, which claim to be suitable for all types of indirect restorations (metal, composite, and porcelain inlays, onlays, crowns, bridges and endodontic posts), except for veneers, without the need for additional enamel/dentin adhesives. This subgroup was introduced in 2002 with RelyX Unicem (3M ESPE). The goal in the development of these newer self-etching/self-adhesive resin luting agents is to combine the easy handling of glass ionomer luting cements, requiring no pre-treatment of tooth structure, with the increased mechanical properties of traditional resin luting agents⁹. The ease-of-use is appealing to the practitioner because of a decreased number of steps and a wide range of applications of use. With the simplified application procedure, there is also a decrease in the technique-sensitivity of the procedure.¹ Compared to other traditional resin luting agents which require an additional etch and rinse step, these self-etching/self-adhesive luting agents contain the necessary chemical components in one product. They do not remove the smear layer, but incorporate it into the cement, as compared to having the smear layer completely removed and rinsed away. This mechanism decreases patient discomfort because the smear layer is incorporated in the dentin/resin hybrid layer, thus not exposing dentinal tubules.^{1, 10}

The mechanical properties of resin luting agents are generally superior to those of the newer self-etching/self-adhesive luting agents.^{1, 9, 11-13} This leads to the choice of possibly having to sacrifice mechanical properties for the benefit of patient sensitivity issues. Ideally, if the mechanical properties of the self-etching/self-adhesive luting agents are comparable to etch and rinse resin luting agents, then it may be worth making a change in material selection for certain clinical situations.

The objective of this study was to test some mechanical properties of four of these self-etching/self-adhesive resin luting agents and compare them to a traditional resin luting agent and a resin-modified glass ionomer luting cement, both of which have longer histories of clinical success. All four of the self-etching/self-adhesive resin luting agents are expected to have similar mechanisms for bonding. By comparing the mechanical properties of the newer luting agents to the controls, the objective was to determine the potential for clinical success of the newer luting agents. If the newer, easier to use luting agents, that reportedly decrease post-operative sensitivity, can show similar properties to the traditional resin luting agents, then their use may not involve a compromise between strength, patient comfort, and ease of application.

The mechanical properties tested were flexural strength and shear bond strength. The flexural strength of a specimen is an indication of the mechanical strength of a material and is useful for assessing the property of brittle materials. The failure potential of restorations under functional stress is related to the mechanical properties of individual components, and flexural strength indicates the ability of a cement to resist stress without fracture.⁵ The flexural strength test included making rectangular beams of each material, storing them in water for periods of time (24 hours and 90 days)^{2, 14} and then performing a three-point bending test on a universal testing machine. The 90 day groups were thermocycled to simulate accelerated aging of the specimens through thermal fatigue.¹⁴

The shear bond strength of a material is the force per unit area required to break the bond of the material. The resulting test value indicates how strong the bond was.¹⁵ The shear bond strength test involved preparing human molar specimens, making flat dentin surfaces. Composite cylinders were fabricated and were cemented to the dentin

surfaces with each of the materials to be tested. The specimens were then stored in water for two different periods of time, 24 hours or 90 days, and then a knife edge shear test was performed on a universal testing machine. The 90 day groups were thermocycled.

A Weibull-distribution survival analysis was performed to compare the different cement products at the different time periods. Results are reported with and without adjustments for multiple comparisons at a 5% significance level.

The purpose of this study was to test the mechanical properties of flexural strength and shear bond strength of four self-etching/self-adhesive resin luting agents. These were compared to control luting agents with a longer history of use, including a traditional resin luting agent and a resin-modified glass ionomer luting cement.

The first null hypothesis was that the self-etching/self-adhesive luting agents and the controls would have no difference in flexural strength at 24 hours or 90 days.

The second null hypothesis was that the self-etching/self-adhesive luting agents and the controls would have no difference in shear bond strength at 24 hours or 90 days.

The first alternative hypothesis was that the self-etching/self-adhesive luting agents would have different flexural strengths compared to the controls.

The second alternative hypothesis was that the self-etching/self-adhesive luting agents would have different shear bond strengths compared to the controls.

REVIEW OF LITERATURE

FLEXURAL STRENGTH

In 2003 Piwowarczyk and Lauer⁹ published the results of a study comparing the flexural strength, after water storage, of 12 different luting agents from different material classes. Included in the material classes tested were two zinc phosphate cements, two glass ionomer luting cements, three resin-modified glass ionomer luting cements, four resin luting agents, and one self-adhesive universal resin luting agent. Groups of materials were stored in distilled water at 37°C, and tested after either 24 hours or 150 days. The resin luting agents had the highest flexural strengths. The self-adhesive universal resin luting agent was statistically stronger than the resin-modified glass ionomer, glass ionomer, and zinc phosphate cements, but not as strong as the other resin luting agents. The self-adhesive universal resin luting agent showed a significant decrease in strength between the 24 hour and the 150 day testing groups.

A 2008 report published by Saskalauskaite, et al.⁵ tested the flexural strength of three self-etching resin, two conventional resin, and two resin-modified glass ionomer luting agents and compared the results. They found a tendency of the self-etching resin luting agents to show similar flexural strengths to the conventional resin luting agents after 24 hours storage at 37°C. The resin-modified glass ionomer luting cements had significantly lower flexural strengths.

SHEAR BOND STRENGTH

In 2004 Piwowarczyk, et al.¹⁶ published a study that evaluated the shear bond strength of several luting agents to gold alloy or different ceramic materials. Luting agents tested were a zinc phosphate cement, two glass ionomer cements, three resin-

modified glass ionomer cements, four resin luting agents, and a self-adhesive resin luting agent. Pre-polymerized resin composite cylinders were bonded to the prosthodontic materials using each of the luting agents. Materials that had dual cure options were tested separately as self-cured and light-cured groups. Groups of specimens were tested after 30 minutes and others were tested after 14 days and thermocycling. The highest shear bond strength values to gold alloys, aluminum oxide ceramics, and pressable ceramics, were exhibited by the self-adhesive luting agent. Low shear bond strengths were noted after only 30 minutes. The bond strengths of the resin luting agents were significantly higher than the zinc phosphate, glass ionomer, and resin-modified glass ionomer luting agents after 14 days and thermocycling. Light polymerization resulted in improvement of bond strengths compared to autopolymerization alone.

Abo-Hamar, et al.⁴ published a study in 2005 which assessed the shear bond strength of a universal self-adhesive resin luting agent, and compared it to three conventional resin luting agents and a glass ionomer luting cement. The groups were stored in distilled water at 37°C and tested after either 24 hours or after being thermocycled. The bond strength of the universal self-adhesive resin luting agent to dentin was not significantly different from the resin luting agents, but was statistically greater than the glass ionomer luting cement. After thermocycling, the universal self-adhesive resin luting agent showed significantly higher bond strength than two of the resin luting agents and the glass ionomer luting cement, but was significantly lower than the other one resin luting agent. The bond strength to enamel, before and after thermocycling, was significantly lower than the resin luting agents, but still higher than the glass ionomer luting cement. After thermocycling, the bond strength to enamel was

significantly lower than to dentin for the universal self-adhesive resin luting agent and the glass ionomer luting cement. It was suggested that it could be used for luting ceramic crowns when there was little or no enamel, but may not be an ideal material for luting inlays or partial coverage crowns that have a significant amount of enamel remaining.

In 2007 Piwowarczyk, et al.⁷ published a study to examine the long-term adhesion of dual-polymerizing resin-based luting agents to human dentin. Five resin luting agents, one compomer, and one self-adhesive universal luting agent were used to bond pre-cured composite to dentin surfaces and stored in water at 37°C for 150 days. Half of the specimens were thermocycled. Another subgroup of half of the specimens was dual-polymerized with light activation. Shear bond strength testing was performed on the specimens. The results led to the conclusion that light polymerization had a significant effect by increasing bond strengths compared to autopolymerization alone. Also, thermocycling had a significant effect by decreasing the bond strengths compared to the groups that did not experience thermocycling. A failure mode analysis revealed that most fractures occurred adhesively at the dentin-cement interface. The self-adhesive resin luting agent exhibited comparable bond strengths to the etch and rinse systems tested, on both the thermocycled and non-thermocycled groups. It also showed significantly greater bond strength compared to the compomer luting agent.

A shear bond strength to dentin evaluation was reported in 2008 by Holderegger, et al.¹² A universal, self-etching/self-adhesive resin luting agent was compared to three conventional resin luting agents. This study used two different testing centers and pooled data. Prepared specimens were stored in water for 24 hours. Half were thermocycled for 1500 cycles between 5°C and 55°C. The shear test results showed that the self-

etching/self-adhesive resin luting agent had the lowest bond strength. After thermocycling, all of the resin luting agents showed decreases in shear bond strengths. However, the self-etching/self-adhesive resin luting agent showed the least influence by the thermocycling, yet was still lower overall.

In 2009, a shear bond strength evaluation was reported by Luhrs, et al.¹³ The study compared bond strengths of four self-adhesive resin luting agents and two conventional resin luting agents to lithium disilicate ceramic and enamel or dentin. The ceramics were pre-treated by sandblasting with 50 μm Al_2O_3 , hydrofluoric acid etching, and silanation prior to the application of the tested resin luting agents. The specimens were light cured, and then stored in distilled water for 24 hours at 37°C. Failure modes were also evaluated. Overall, it was concluded that the self-adhesive resin luting agents were inferior to the conventional resin luting agents.

MICROTENSILE BOND STRENGTH

Microtensile bond strength of a self-adhesive luting agent was tested and compared to an etch and rinse luting system. DeMunck, et al.³ compared the two types of luting agents to both enamel and dentin, and tested the self-adhesive agent with and without prior acid etching. The bonded specimens were stored in water for 24 hours prior to testing. The bond strength to enamel was significantly greater for the etch and rinse luting agent, but the bond strength to dentin revealed no significant difference, compared to the self-adhesive luting agent. When acid etched prior to using the self-adhesive luting agent, the enamel bond strength was raised to the level of the etch and rinse luting agent, but a detrimental effect was noted for the dentin bond strength.

Goracci, et al.⁶ did a microtensile bond strength study in 2006 that looked at bond strengths to both enamel and dentin. RelyX Unicem (3M ESPE) and Maxcem (Kerr) were compared to Panavia F (Kuraray). For adhesion to enamel, the Panavia group had significantly greater bond strength than RelyX Unicem, both of which had significantly greater bond strength than Maxcem. For adhesion to dentin, no significant differences in bond strength were found between the Panavia and the RelyX Unicem groups, and they both significantly outperformed the Maxcem group. Scanning Electron Microscope (SEM) observations were made and it was concluded that the application of RelyX Unicem or Maxcem did not result in the formation of a hybrid layer in dentin.

A 2007 study, published by Hikita et al.,⁸ tested the bonding effectiveness of adhesive luting agents to enamel and dentin. Five different resin luting agents, including a self-adhesive, were used to lute composite resin blocks to human molars. They were then stored for 24 hours in water at 37°C. Microtensile bond strength testing was performed. The authors also created experimental groups that pretreated the tooth surface, with the use of etchants, as an additional step for use with the self-adhesive resin luting agent. Pre-etching, in combination with the use of the self-adhesive resin luting agent, produced a greater bond strength to enamel, but a weaker bond strength to dentin, compared to just applying the self-adhesive resin luting agent as directed by the manufacturer. Fracture analysis, for both enamel and dentin specimens, determined that most of these weakly bonded specimens failed adhesively at the cement-tooth interface. Overall, the authors concluded that etch and rinse, self-etching, and self adhesive luting agents were equally effective in bonding to enamel and dentin, but only on the condition that a correct adhesive procedure was carried out.

In 2009 D'Arcangelo, et al.¹⁷ published a study that evaluated the microtensile bond strength of dentin to either indirect resin-based composites or ceramic restorations. It compared two etch and rinse luting agents, a self-etching luting system, and a self-adhesive luting agent. A SEM evaluation was done to determine the mode of fracture. It was concluded that etch and rinse luting agents have more reliable bonding than self-etching/self-adhesive luting agents, when evaluating the interface between dentin and indirect resin. Alternatively, the self-adhesive luting agent used in the study, RelyX Unicem (3M ESPE), showed higher microtensile bond strength to glass ceramic disks, suggesting that maybe self-etching/self-adhesive luting agents may be preferred when cementing glass ceramics, but not for resin-based composites.

RETENTIVE STRENGTH

Zidan and Ferguson¹⁸ reported in 2003 that conventional resin luting agents provide the greatest bonding ability of indirect restorations, particularly when required on over-tapered preparations, compared to zinc phosphate and glass ionomer luting cements. High noble metal crowns were luted on preparations with tapers of 6-degrees, 12-degrees, or 24-degrees. The 24-degree tapered preparations were significantly different in retentive strength compared to the 6-degree and 12 degree preparations. The resin luting agents yielded retentive values that were double that of zinc phosphate or conventional glass ionomer luting cements.

MARGINAL INTEGRITY

The luting of porcelain inlays by using either etch and rinse luting agents or self-etching luting agents was evaluated by Frankenberger, et al.¹¹ in 2008. Four etch and

rinse systems were used and compared to five self-etching systems. MOD preparations were made on extracted human molars, with one of the proximal boxes below the cemento-enamel junction and the other terminating in enamel. Porcelain inlays were fabricated and luted with each of the different luting systems. The specimens were thermocycled and marginal integrity was evaluated by SEM. Significantly higher percentages of gap-free margins in enamel were noted for all of the etch and rinse systems. For the margins on dentin, no significant difference was noted between many of the self-etching and etch and rinse systems.

PHYSICAL PROPERTIES

Han, et al.¹⁹ published a 2007 study in which four self-adhesive resin luting agents were tested for pH value, film thickness, filler particle percentage, and morphological changes. Materials that had higher percentages of filler particles had greater film thickness. The pH values were measured 20 seconds after light curing, as well as at 90 seconds and 48 hours after mixing. Results showed that there were significant differences in the materials tested and that might lead to a difference in clinical performance.

Radovic, et al.²⁰ published a literature review of self-adhesive resin luting agents in 2008. There were only *in vitro* studies published at the time of the review and a great majority of the studies used RelyX Unicem (3M ESPE), while a few used Maxcem (Kerr). Categories of the studies summarized included: adhesion to tooth structure (enamel, dentin, and root dentin), adhesion to various restorative materials, mechanical properties, as well as others. The review concluded that the self-adhesive luting agents

bond to dentin and restorative materials satisfactorily and comparably to other multistep resin luting agents. However, the adhesion of the self-adhesive luting agents to enamel is weaker than other resin luting agents.

CLINICAL STUDY

To date, a great majority of all of the research involving these newest self-etching/self-adhesive resin luting agents has been done *in vitro*. An exception is a prospective clinical trial by Behr, et al.²¹ comparing a self-adhesive resin luting agent, RelyX Unicem (3M ESPE), to zinc phosphate cement. Forty nine patients received 49 metal-based restorations, randomly luted with one of the previously mentioned luting agents. Restorations were examined annually for plaque, bleeding, and attachment. During the observation time (mean: 38 months; min: 2 years; max: 4.5 years), no restorations were lost or needed recementation, and the self-adhesive resin luting agent clinically performed as well as the zinc phosphate cement.

MATERIALS AND METHODS

The materials used are listed in Table I. Four self-etching/self-adhesive luting agents [Multilink Automix (Ivoclar Vivadent) 'MA', Maxcem Elite (Kerr) 'ME', RelyX Unicem (3M ESPE) 'RU', SmartCem 2 (Dentsply) 'SC'], one conventional, etch and rinse, resin luting agent [RelyX ARC (3M ESPE) 'RA'], and one resin-reinforced glass ionomer luting cement [Fuji Plus (GC) 'FP'] were compared. The materials were mixed and dispensed according to manufacturer instructions. RelyX ARC was packaged in a pre-measured "clicker" that dispensed the proper paste-paste ratio automatically and it was hand-mixed using a spatula and mixing pad. Fuji Plus and RelyX Unicem were packaged in capsules with pre-measured amounts of the proper ratio of powder and liquid that was mixed in an amalgam triturator. Multilink Automix, Maxcem Elite and SmartCem 2 were packaged in dual paste syringes and were dispensed through attached mixing tips that ensured consistent and proper mixes of the paste-paste ratio. Multilink Automix required the use of a self-etching primer, adding an additional step to the luting procedure. RelyX ARC required separate steps for etching, rinsing, and priming/bonding. The packaging for both the Multilink Automix and the RelyX ARC included its own additional items, including a silanating agent.

FLEXURAL STRENGTH

The specimens for the flexural strength three-point bending test were fabricated according to ISO 4049, by placing the materials into a stainless steel split mold (Figure 1). The internal dimensions of the split mold were 2 ± 0.1 mm deep X 2 ± 0.1 mm wide X 25 ± 2 mm long. Polytetrafluoroethylene (PTFE) Release Agent Dry Lubricant (Miller-Stephenson Chemical Company, Inc., Danbury, CT) was sprayed onto the stainless steel mold. The release agent was thinned by a short, quick blast of Dust-Off Compressed Gas

(Falcon Safety Products, Inc., Branchburg, NJ). A glass slab and a Mylar strip were placed beneath the mold. The tested luting agents were mixed according to manufacturer instructions and placed into the mold slightly overfilled. Another Mylar strip and glass slide were placed on top and pressed firmly down with finger pressure to prevent porosities from forming in the specimen. The specimen was then light-cured through the glass slide by three overlapping, 20 second cycles on the top and then turned over to cure the opposite side of the specimen, using the Optilux VCL 401 (Demetron Research Corp., Danbury, CT) halogen curing unit with an output range of 460-515 mW/cm², as tested with a Cure Rite visible curing light meter (Dentsply Caulk, Milford, DE). Excess material was carefully trimmed with a scalpel blade (Figure 1), the specimens were carefully removed from the mold, and lightly wet polished on the edges with 400 and 600 grit Silicon Carbide (SiC) paper to ensure true edges. The specimens were stored in deionized water at 37°C. After 24 hours, 15 specimens of each group were tested on a MTS Sintech ReNew 1123 universal testing machine (MTS Systems Corporation, St. Paul, MN) using a three-point bending test. Also, 15 of the specimens of each group remained stored in deionized water at 37°C before and after thermocycling. The thermocycling was begun 14 days after specimen preparation and consisted of 2500 cycles between 6 and 48°C with a 30 second dwell time and a 10 second transit time.^{22, 23} After the designated time periods, and prior to testing, the height and width of each specimen was measured using a digital micrometer. The mean of three measurements, for both width and thickness, was used in the calculations of the flexural strength. Those measurements were entered into the program and the specimens were placed on the testing apparatus. A three-point bending jig was attached to the testing machine and

connected to a computer with a software program (Test-Works 4.0, MTS Systems Corporation, St. Paul, MN) that controlled the testing machine and recorded the data for each specimen. The set up included two lower rods that were 20 mm apart and an upper rod that applied the load. That upper rod was centered between the two lower rods (Figure 2). The test was carried out using a cross head speed of 1 mm/min. Flexural strength values were recorded for statistical analysis. The following equation was used to determine flexural strength, the maximum stress before fracture, in Megapascals (MPa):

$$\text{Stress} = 3 \times \text{Load} \times \text{Length} / (2 \times \text{Width} \times \text{Thickness}^2)$$

where the length was the distance between the lower support rods (20mm), width was the mean specimen width, and thickness was the mean specimen height.²⁴

Fifteen specimens of each group of luting agent were prepared for the 24 hour testing group and 15 specimens per group were also prepared for the 90 day testing group. There were a total of 12 groups in the flexural strength test. (24hr MA, 24hr SC, 24hr RU, 24hr ME, 24hr FP, 24hr RA, 90day MA, 90day SC, 90day RU, 90day ME, 90day FP, 90day RA)

SHEAR BOND STRENGTH

Human molar teeth were prepared for bonding. Composite resin cylinders of a standard diameter were prepared and the tested luting agents were cemented to the dentin surfaces.⁸ The dentin shear bond strength test was based on ISO 11405. The resin cylinders were luted to the teeth, and then stored in deionized water at 37°C.

Extracted, non-carious, and restoration-free human molar teeth stored in 0.1% thymol solution were used. A flat dentin bonding surface was prepared on the occlusal surface by removing the cusps and grooves on the Lapcraft L'il Trimmer cutting machine (Lapcraft, Inc., Powell, OH) (Figure 3). The flat portions were wet sanded using 240 and 320 grit SiC paper. The dentin surfaces were placed flat down on a Mylar sheet. Plastic cylinders (approx 15-16 mm internal diameter and 20-25 mm tall) were placed over and around each tooth. The teeth were mounted in the cylinders by using Fastray (Bosworth Company, Skokie, IL) self-curing acrylic resin (Figure 3). The acrylic resin was mixed and poured into the cylinders until it completely covered the tooth and filled the cylinder. After the acrylic resin had set, any excess material was removed, and the teeth were polished by using 400 and 600 grit SiC paper. The specimens were placed in deionized water and refrigerated at 5°C until the resin cylinders were luted to the dentin prior to the shear bond strength testing.

The indirect resin cylinders were fabricated by directly placing the material (Premise Indirect, Kerr Corporation, Orange, CA) into a metal split mold that formed 4mm diameter resin cylinders. PTFE Release Agent Dry Lubricant (Miller-Stephenson Chemical Company, Inc.) was sprayed onto the stainless steel mold. After directly packing the material into the mold, initial polymerization was initiated by using the Optilux VCL 401 (Demetron Research Corp.) halogen curing unit on the visible end of the material for 20 seconds, as recommended by the manufacturer. The mold was opened and then additional curing was done from the newly exposed side. Final polymerization occurred in the Premise Indirect HP curing unit (Kerr Corporation, Orange, CA) for 20 minutes under a nitrogen atmosphere at 60 psi and a temperature of 138°C (Figure 4).

The bonding surfaces of the composite cylinders were roughened by sandblasting, followed by silanation to enhance the retention between the resin luting agents and the indirect composite.²⁵ The Premise Indirect specimens were mounted in two layers of red boxing wax to hold them in place and expose the bonding surfaces. The surface was sandblasted with 50 μ m Al₂O₃.²⁵ The resin cylinders were removed from the wax and remaining wax was removed from the sides with a scalpel. The cylinders were then rinsed off with deionized water. Finally, they were placed in an ultrasonic bath for two minutes, dried, and stored until they were luted to the previously prepared dentin surfaces, using the tested luting agents.

The resin cylinders were luted with each of the testing groups to the previously prepared dentin surfaces as described below:

FP: Just prior to luting, the resin cylinders were silanated using Ultradent Silane (Ultradent Products, Inc., South Jordan, UT) and allowed to dry. The tooth specimens were rinsed with deionized water for 5 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. The FP capsule was activated and mixed for 10 seconds on high in a ProMix (Dentsply Caulk) model 400 triturator. The luting agent was then dispensed through the tip of the capsule.

ME: Just prior to luting, the resin cylinders were silanated using Ultradent Silane (Ultradent Products, Inc.) and allowed to dry. The tooth specimens were rinsed with deionized water for 5 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. The luting agent was dispensed through the mixing tip.

SC: Just prior to luting, the resin cylinders were silanated using Ultradent Silane (Ultradent Products, Inc.) and allowed to dry. The tooth specimens were rinsed with deionized water for 5 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. The luting agent was dispensed through the mixing tip.

RU: Just prior to luting, the resin cylinders were silanated using Ultradent Silane (Ultradent Products, Inc.) and allowed to dry. The tooth specimens were rinsed with deionized water for 5 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. The RU Maxicap capsule was activated and mixed for 15 seconds on high in a ProMix (Dentsply Caulk) model 400 triturator. The luting agent was then dispensed through the tip of the capsule.

MA: Just prior to luting, the resin cylinders were silanated using Monobond S (Ivoclar Vivadent) and allowed to dry. The tooth specimens were rinsed with deionized water for 5 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. A 1:1 mix of Multilink Primer A and B (Ivoclar Vivadent) was applied to the tooth with a microbrush under slight pressure for 15 seconds. It was lightly air dried for 5 seconds. The luting agent was dispensed through its mixing tip.

RA: Just prior to luting, the resin cylinders were silanated using RelyX Ceramic Primer (3M ESPE) and allowed to dry. The tooth specimens were etched with phosphoric acid, Scotchbond Etchant (3M ESPE), for 15 seconds, rinsed with deionized water for 10 seconds, and then the excess water was blotted with a gauze square, leaving a moist surface, not wet or dry. Two coats of adhesive, Adper Single Bond Plus (3M

ESPE), were placed for 15 seconds each with gentle agitation. The adhesive was light cured for 10 seconds. The luting agent was dispensed from its “clicker” dispenser, onto a mixing pad and hand mixed with a plastic spatula for 10 seconds.

Each material was mixed, and applied to the composite’s bonding surface, then seated on the dentin surface of the tooth specimen. A standard 1 kg load was placed to secure the resin cylinders while the excess cement was removed from around the luted interface (Figure 5). After the excess was removed, the margins were light cured for 30 seconds. The teeth and bonded restoration complex was then removed from under the load and additional curing was done on the lab bench to be sure to get access to the parts that might not have been adequately photoinitiated due to limited access with the load in place. A final removal of cured excess cement was carefully accomplished using a scalpel blade.

Upon completion of luting the resin cylinders to the dentin surfaces, half of each group of test material samples were placed in deionized water at 37°C for 24 hours and the other half for 90 days and thermocycling. The specimens were stored in deionized water before and after thermocycling. The thermocycling was begun 14 days after specimen preparation and consisted of 2500 cycles between 6 and 48°C with a 30 second dwell time and a 10 second transit time.

After the designated periods of time, and prior to testing, the diameter of each luted resin cylinder specimen was measured using a digital micrometer. The mean of three measurements was used to enter into the computer for the calculations of flexural strength. The specimens were tested on the MTS testing machine. The cylinder molds

were placed in a special holder, attached to the stationary base of the MTS. The long axis of each resin cylinder was perpendicular to the force that was applied. A stainless steel ring with a knife-edged inner circumference was adapted to the flat surface of the specimen at the interface of the composite cylinder and the dentin surface. The stainless steel ring was attached via a chain to the 125N load cell on the MTS (Figure 6). The specimen was loaded by a shear force with a cross-head speed of 1 mm/min until the fracture occurred. The shear bond strength was obtained on the computer with the software program (Test-Works 4.0, MTS Systems Corporation, St. Paul, MN). The values were recorded for statistical analysis. The following equation was used to determine shear bond strength, the maximum stress before debonding, in Megapascals (MPa):

$$\text{Stress} = \text{Load} / (\pi \times \text{Diameter})$$

where the diameter was the mean of the specimen diameter.²⁴

Twenty four specimens of each group of luting agent were prepared for the 24 hour testing group and also for the 90 day testing group. There were a total of 12 groups in the shear bond strength test. (24hr MA, 24hr SC, 24hr RU, 24hr ME, 24hr FP, 24hr RA, 90day MA, 90day SC, 90day RU, 90day ME, 90day FP, 90day RA).

STATISTICAL METHODS

Because some shear bond strength specimens debonded prematurely, comparisons between the treatment combinations for differences in flexural strength and shear bond strength were performed using a Weibull distribution survival analysis, using the force required for bond failure in place of the usual 'time to event' seen in typical survival

analyses. Shear bond strength specimens that debonded before placement on the testing machine were accommodated in the survival analysis model as left-censored observations, and specimens that did not debond prior to the end of testing were accommodated as right-censored observations. Statistical analyses were performed using S-PLUS version 8.1 (TIBCO Software Inc., Palo Alto, CA) and R version 2.8.0 (The R Project for Statistical Computing, <http://www.r-project.org/>).

Shear bond strength results can show considerable variation, and may not be normally distributed. When that is the case, a Weibull distribution can relate the probability of failure at a certain stress level. It allows for predicting the likelihood of failure of a material at low stress values.²⁶ The probability of failure (P_f) of a specimen from a group of N specimens is given by:

$$P_f = n / (N + 1)$$

where n is the ranking number of the specimen. The specimens are ranked from weakest to strongest (1 to N).²⁶⁻²⁸

For consistency, a Weibull distribution was also used in the analysis of flexural strength data. It has been shown that the distribution function can also be accurately fitted to data from three-point bend tests.²⁸

The Weibull Characteristic Strength is the strength at which 63.2% of the specimens in a group failed (36.8% had still survived, meaning that they remained unbroken/not debonded).²⁶⁻²⁸ For the bond strength summary statistics, specimens that debonded prematurely were given a value of 0.05. To account for the debonded specimens, a value needed to be assigned that was greater than 0.00 and lower than the lowest tested value, 0.1.

Based on the data collected in previous dental materials lab studies using the same testing methods, the flexural strength measurements were expected to range between 80-100 MPa with a standard deviation in the 10-15 MPa range,²⁹ and the shear bond strength measurements were expected to range between 15-20 MPa with a standard deviation in the range of 6-7 MPa.³⁰ With a sample size of 15 specimens per treatment combination (180 total) for the flexural strength comparisons, the study would have 80% power to detect a difference of 15.9 MPa between any two treatment combinations, assuming a two-sided test at a 5% significance level for each test. With a sample size of 20 specimens per treatment combination (240 total) for the shear bond strength comparisons, the study would have 80% power to detect a difference of 6.4 MPa between any two treatment combinations, assuming a two-sided test at a 5% significance level for each test.

RESULTS

FLEXURAL STRENGTH

The mean values and standard deviations are shown in Table II. The characteristic flexural strength of Weibull distribution is shown in Table III. Tables IV and V show the inter-group differences for the flexural strength comparisons at 24 hours and 90 days, respectively. Figure 7 shows the estimated survival function of the groups at various flexural strengths using the individual observations, while Figure 8 shows the estimated survival function curves based on the Weibull model.

The groups listed in decreasing order of flexural strength at 24 hours were: MA > RA > SC > ME > RU > FP. All groups were significantly different ($p < 0.05$). The groups listed in decreasing order of flexural strength at 90 days were: MA > RA > SC > RU > ME > FP. Only one pair was not significantly different, SC 90d & RA 90d ($p = 0.2010$).

MA had the highest flexural strength at 24 hours and 90 days with thermocycling, followed by RA and SC. FP had the lowest flexural strength at both 24 hours and 90 days with thermocycling.

All of the luting agents tested, with the exception of FP, showed significant effects of thermocycling and extended water storage time, with a decrease in flexural strength. FP ($p < 0.0001$) showed a significant increase in flexural strength between 24 hours and 90 days.

SHEAR BOND STRENGTH

Mean values and standard deviations are shown in Tables VI and VII for the 24 hour and 90 day groups, respectively. Tables VI and VII also show the number of specimens per group that debonded prior to testing, as well as those that failed to debond

near the upper limit of the 125 N load cell used. The characteristic shear bond strength of Weibull distribution is shown in Table VII. Tables IX and X show the inter-group differences for the shear bond strength comparisons at 24 hours and 90 days, respectively. Figure 9 shows the estimated survival function of the groups at various bond strengths using the individual observations, while Figure 10 shows the estimated survival function curves based on the Weibull model.

The groups listed in decreasing order of shear bond strength after 24 hours were: $RU > RA > MA > FP > ME > SC$. There was no statistically significant difference between RU, RA, and MA, which had significantly higher shear bond strengths than FP, ME, and SC; FP had significantly higher bond strength than SC. At 24 hours, SC and ME showed no difference ($p=0.3090$) in shear bond strength; ME also showed no difference to FP ($p=0.0985$); RU showed no difference to MA ($p=0.0984$) or RA ($p=0.2100$); and MA showed no difference to RA ($p=0.4380$). In summary, compared to the controls, at 24 hours, RU and MA performed equal to RA, and superior to FP. ME performed equal to FP and FP performed better than SC.

After 90 days and thermocycling, the groups listed in decreasing order of shear bond strength were: $RA > RU > MA > SC > FP > ME$. At 90 days, RA had significantly higher shear bond strength than all other groups ($p<0.05$). RU had significantly higher shear bond strength than SC, FP, and ME; MA had significantly higher shear bond strength than ME. At 90 days, FP showed no difference to ME ($p=0.7060$), SC ($p=0.5910$), or MA ($p=0.0557$); SC showed no difference to ME ($p=0.3400$) or MA ($p=0.1360$); and MA showed no difference to RU ($p=0.0918$). In summary, compared to the controls, after 90 days and thermocycling, none of the other materials had shear bond

strengths equal to RA. RU had higher bond strength than FP, while MA, SC and ME performed equal to FP.

For RA, ME, and FP, there were significant differences between the 24 hour and 90 day groups ($p < 0.05$), indicating that there was a statistically significant effect of water storage time and thermocycling on the shear bond strength of those materials. The ME ($p = 0.0015$) and FP ($p = 0.0014$) groups decreased significantly, but the RA ($p = 0.0054$) group increased significantly. The RU ($p = 0.2760$), MA ($p = 0.3210$), and SC ($p = 0.7010$) groups showed no statistically significant effect from the extended water storage time and thermocycling on shear bond strength.

TABLES AND FIGURES

TABLE I

Materials Used – Types, Dispensing Systems, and Lot Numbers

Group	Product	Manufacturer	Material Type*	Dispensing system	Lot Numbers#
FP	Fuji Plus	GC America Alsip, IL	Resin Reinforced GIC	Capsule	0809021 (SBS) 0808056 (FS)
RA	RelyX ARC	3M ESPE St. Paul, MN	Adhesive Resin Luting Agent	Paste-Paste "Clicker"	GN8JA (SBS) GP8HX (FS)
	Adper Single Bond Plus	3M ESPE St. Paul, MN	Bonding Agent		8UK
MA	Multilink Automix	Ivoclar Vivadent, Inc. Amherst, NY	SE Resin Luting Agent	Automix syringe w/A-B Primers	L37636
	Multilink Primer A and B	Ivoclar Vivadent, Inc. Amherst, NY	Self-Etching Primer/Adhesive		L37328 L40477
ME	Maxcem Elite	Kerr Corporation Orange, CA	SA Resin Luting Agent	Automix Syringe	3151076 3095231 (24SBS)
RU	RelyX Unicem	3M ESPE St. Paul, MN	SA Resin Luting Agent	Capsule	346262
SC	SmartCem 2	Dentsply International York, PA	SA Resin Luting Agent	Automix Syringe	0808051
	Premise Indirect	Kerr Corporation Orange, CA	Indirect Composite Resin		2987209

*GIC= Glass Ionomer Cement, SE= Includes Separate Application of Self-Etching Dental Adhesive, SA=Self-Adhesive Resin

#SBS= Shear Bond Strength, FS=Flexural Strength, 24SBS= 24 Hour Shear Bond Strength

TABLE II

Flexural Strength [Mean (MPa) and Standard Deviation]

Material	N	24 Hours	90 Days
FP	15	5.35 ± 1.16	10.94 ± 2.12
RA	15	103.33 ± 13.93	78.97 ± 12.34
MA	15	115.22 ± 14.53	93.68 ± 12.61
ME	15	55.25 ± 13.86	29.69 ± 7.48
RU	15	44.34 ± 6.06	39.60 ± 5.14
SC	15	86.83 ± 11.41	73.53 ± 12.58

TABLE III

Characteristic Flexural Strength of Weibull Distribution (MPa)

Material	24 Hours	90 Days	p-value
FP	6	12	<0.0001
RA	110	85 ^a	<0.0001
MA	122	99	<0.0001
ME	61	33	<0.0001
RU	47	42	0.0063
SC	92	79 ^a	0.0012
<p>The same superscripted letters indicate no significant difference in 90 day data ($p>0.05$). The given p-values are for 24 hour and 90 day differences of a material.</p>			

TABLE IV

Inter-Group Differences for 24 hour Flexural Strength Comparisons

	FP	RA	MA	ME	RU	SC
FP	-	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
RA	<0.0001	-	0.0208	<0.0001	<0.0001	0.0001
MA	<0.0001	0.0208	-	<0.0001	<0.0001	<0.0001
ME	<0.0001	<0.0001	<0.0001	-	0.0003	<0.0001
RU	<0.0001	<0.0001	<0.0001	0.0003	-	<0.0001
SC	<0.0001	0.0001	<0.0001	<0.0001	<0.0001	-

TABLE V

Inter-Group Differences for 90 day Flexural Strength Comparisons

	FP	RA	MA	ME	RU	SC
FP	-	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
RA	<0.0001	-	0.0011	<0.0001	<0.0001	0.2010
MA	<0.0001	0.0011	-	<0.0001	<0.0001	<0.0001
ME	<0.0001	<0.0001	<0.0001	-	0.0003	<0.0001
RU	<0.0001	<0.0001	<0.0001	0.0003	-	<0.0001
SC	<0.0001	0.2010	<0.0001	<0.0001	<0.0001	-

TABLE VI

Shear Bond Strength, 24 hour [Mean (MPa) and Standard Deviation]

Material	N	Early Debond	Failure to Debond	24 Hours
FP	24	1	0	2.17 ± 1.63
RA	24	0	4	4.76 ± 2.84
MA	25	1	4	3.92 ± 2.90
ME	24	1	0	1.45 ± 0.82
RU	25	0	5	6.26 ± 1.80
SC	22	2	0	1.28 ± 1.30

TABLE VII

Shear Bond Strength, 90 day [Mean (MPa) and Standard Deviation]

Material	N	Early Debond	Failure to Debond	90 Days
FP	22	2	0	0.86 ± 1.08
RA	24	0	13	7.39 ± 2.29
MA	21	2	4	2.66 ± 3.50
ME	23	2	0	0.63 ± 0.73
RU	24	0	3	5.59 ± 2.02
SC	21	2	0	1.16 ± 1.48

TABLE VIII

Characteristic Shear Bond Strength of Weibull Distribution (MPa)

Material	24 Hours	90 Days	p-value
FP	2.3 ^b	0.7 ^{c,d}	0.0014
RA	5.9 ^a	>9.5 ^a	0.0054
MA	4.6 ^a	2.5 ^{b,c}	0.3210 *
ME	1.6 ^{b,c}	0.6 ^d	0.0015
RU	7.3 ^a	6.5 ^b	0.2760 *
SC	1.1 ^c	0.9 ^{c,d}	0.7010 *
The same superscripted letters indicate no significant difference in 24 hour or 90 day data ($p > 0.05$). Superscripted * indicates no significant difference between 24 hour and 90 day of a material ($p > 0.05$)			

TABLE IX

Inter-Group Differences for 24 hour Shear Bond Strength Comparisons

	FP	RA	MA	ME	RU	SC
FP	-	0.0001	0.0327	0.0985	<0.0001	0.0410
RA	0.0001	-	0.4380	<0.0001	0.2100	<0.0001
MA	0.0327	0.4380	-	0.0005	0.0984	0.0006
ME	0.0985	<0.0001	0.0005	-	<0.0001	0.3090
RU	<0.0001	0.2100	0.0984	<0.0001	-	<0.0001
SC	0.0410	<0.0001	0.0006	0.3090	<0.0001	-

TABLE X

Inter-Group Differences for 90 day Shear Bond Strength Comparisons

	FP	RA	MA	ME	RU	SC
FP	-	<0.0001	0.0557	0.7060	<0.0001	0.5910
RA	<0.0001	-	0.0094	<0.0001	0.0046	<0.0001
MA	0.0557	0.0094	-	0.0235	0.0918	0.1360
ME	0.7060	<0.0001	0.0235	-	<0.0001	0.3400
RU	<0.0001	0.0046	0.0918	<0.0001	-	<0.0001
SC	0.5910	<0.0001	0.1360	0.3400	<0.0001	-

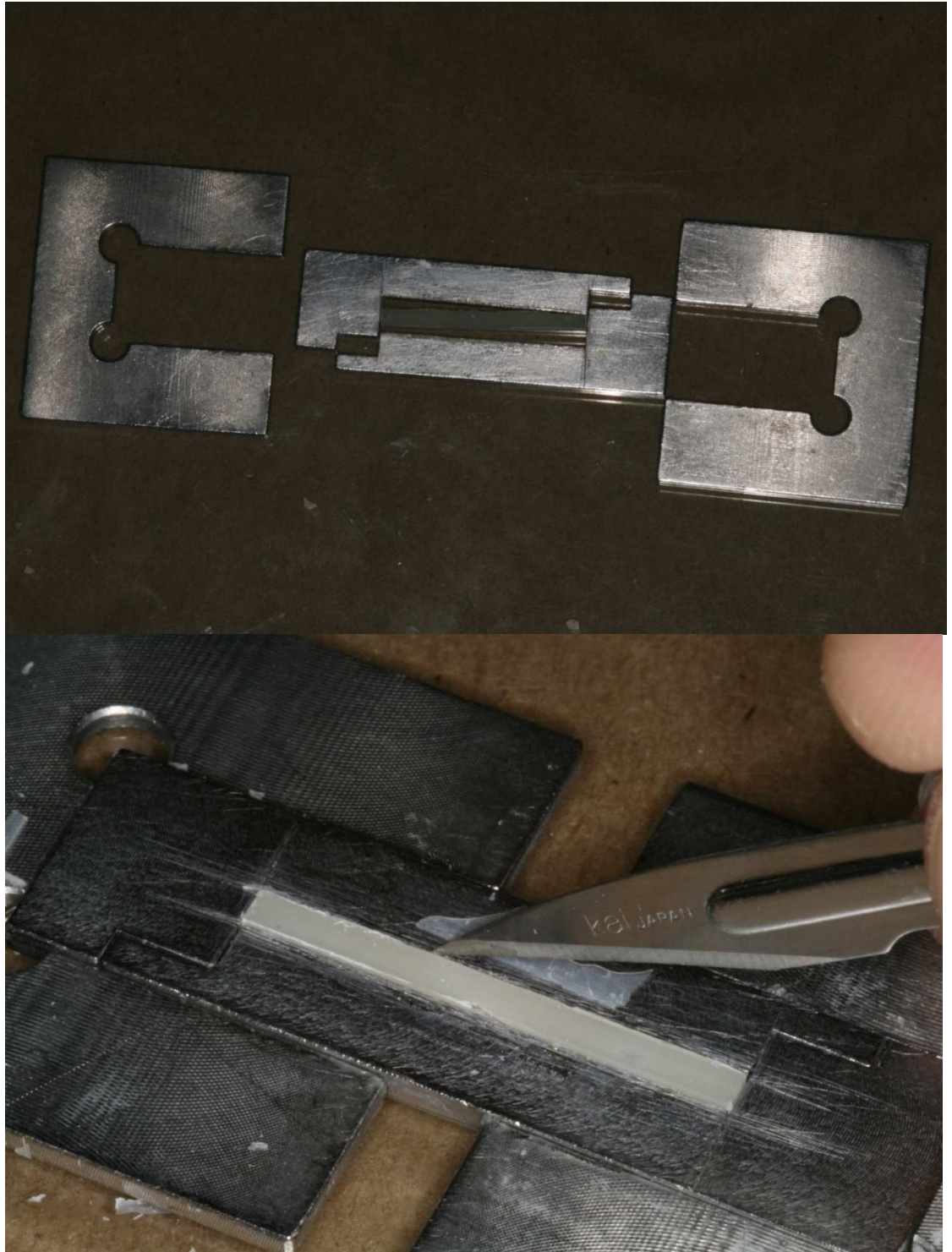


FIGURE 1: Top - stainless steel split mold for fabricating flexural strength testing specimens; Bottom – excess material carefully trimmed with scalpel blade prior to removal of specimen from the split mold.



FIGURE 2: Top – flexural strength/three-point-bending testing apparatus on MTS machine; Bottom – close view of apparatus.



FIGURE 3: Molar preparation: Top – removal of cusps and occlusal enamel; Bottom – sequence of steps for mounting molars in acrylic resin.



FIGURE 4: Indirect resin cylinder fabrication: Top Left – stainless steel split mold; Top Right – resin placement into mold; Middle Left – initial photopolymerization; Middle Right – additional polymerization; Bottom – Premise Indirect HP curing unit for final polymerization.

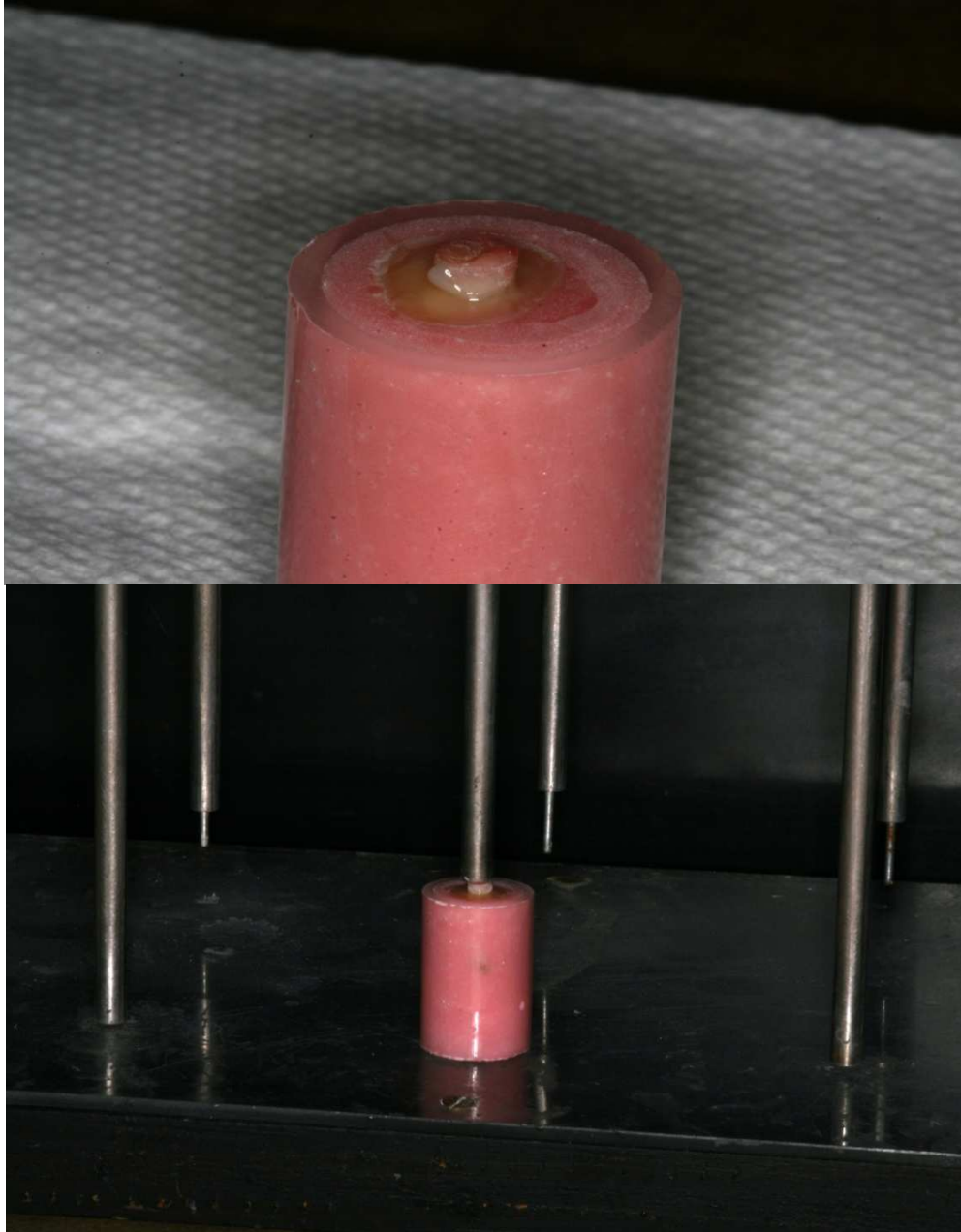


FIGURE 5: Top – indirect resin cylinder luted to dentin; Bottom – 1 kg load placed on resin prior to photo initiation of polymerization.



FIGURE 6: Top – shear bond strength/knife-edge shear testing apparatus on MTS machine; Bottom – close view of apparatus.

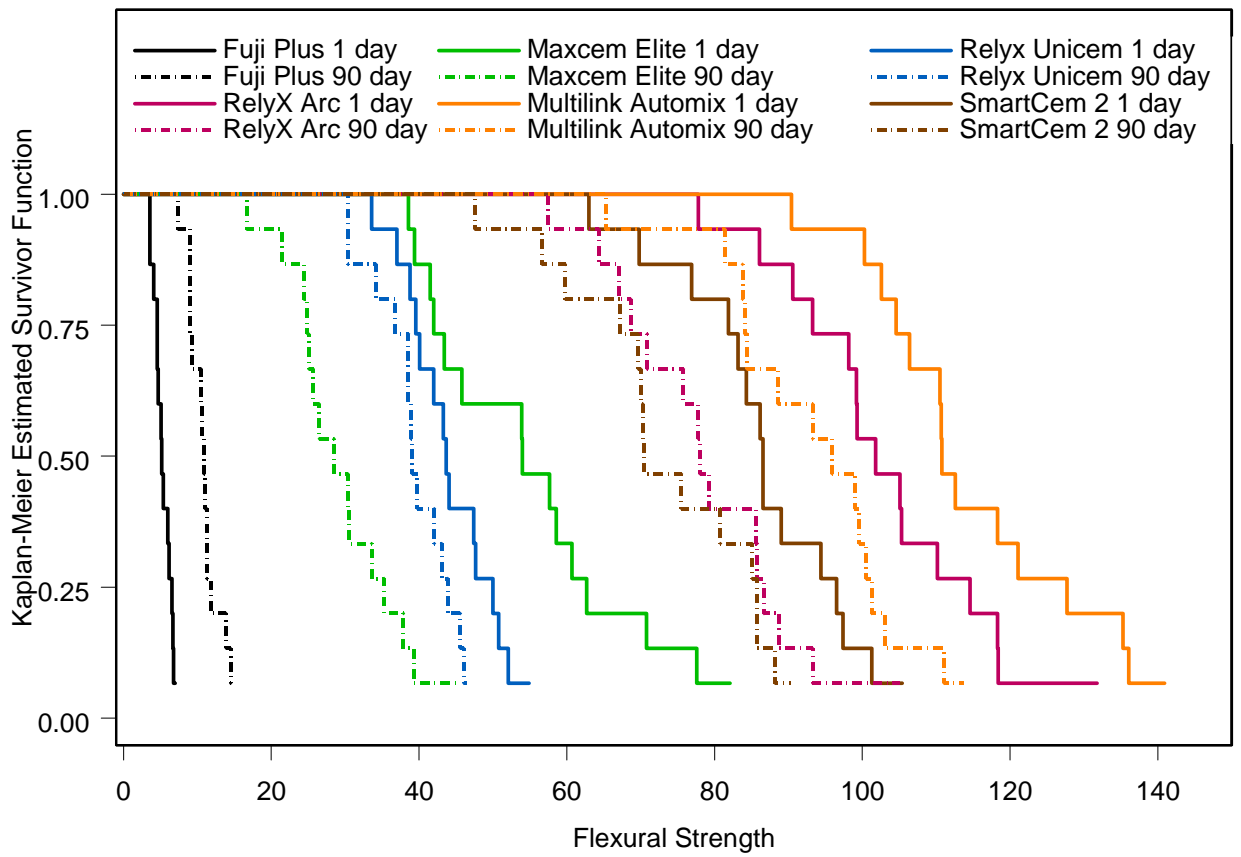


FIGURE 7: Flexural strength plot: The 'jagged' curves plot the survival functions using the individual observations.

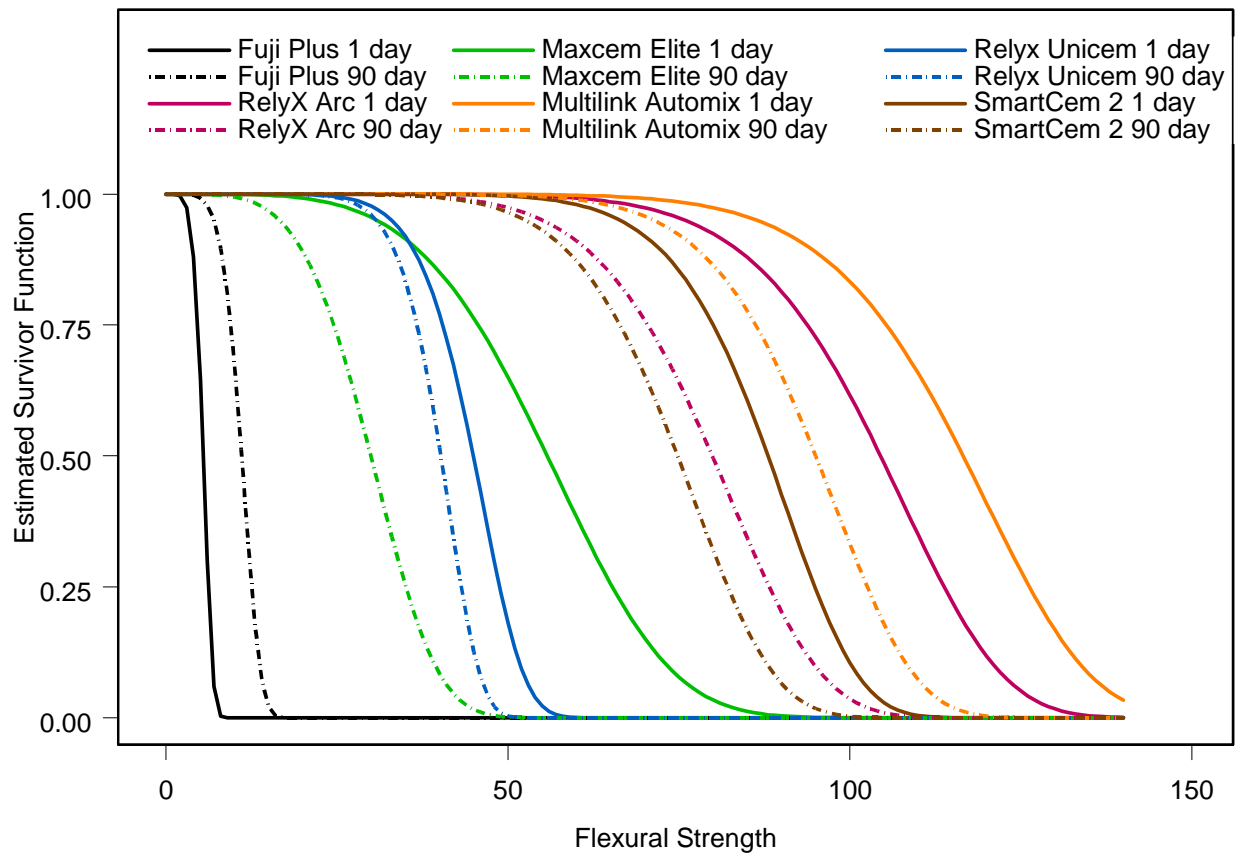


FIGURE 8: Flexural strength plot: The smoothed lines are the survival curves fitted by the Weibull models

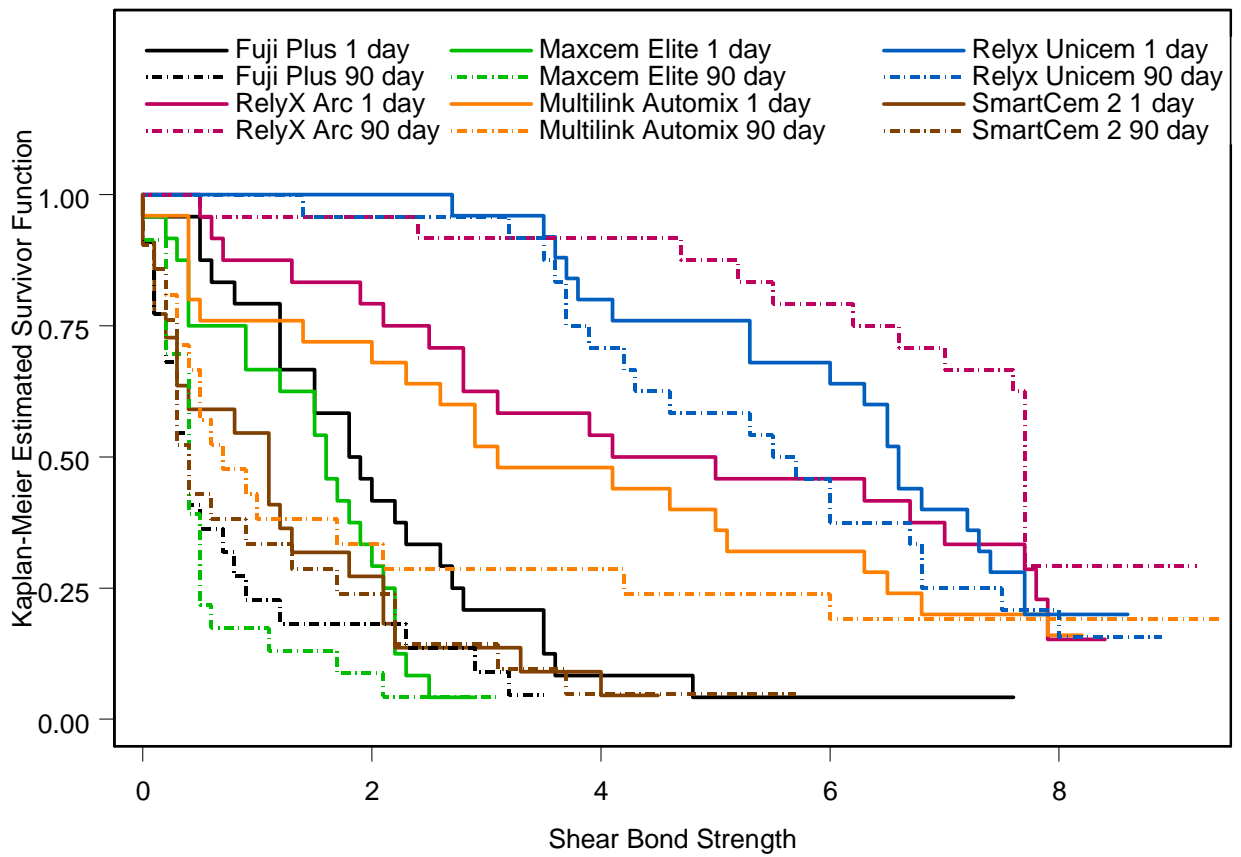


FIGURE 9: Shear bond strength plot: The 'jagged' curves plot the survival functions using the individual observations.

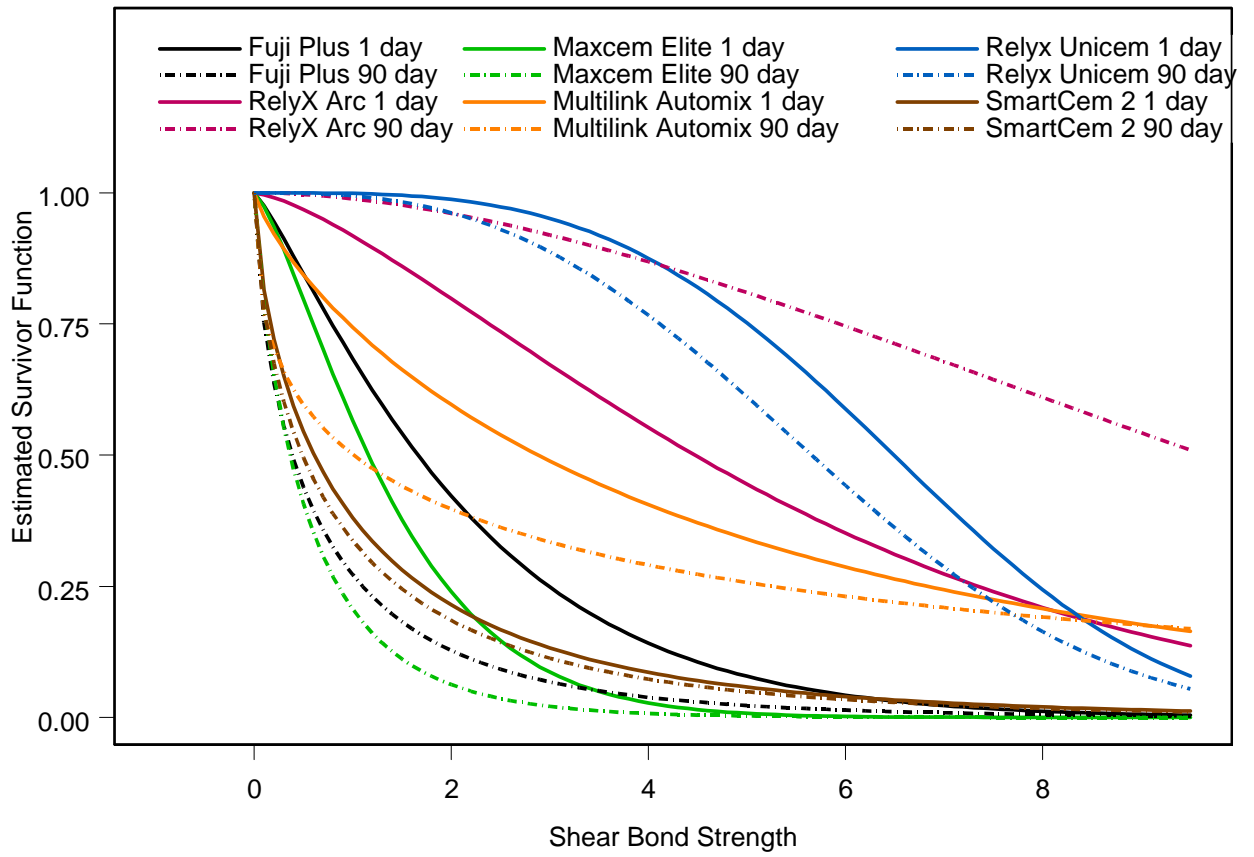


FIGURE 10: Shear bond strength plot: The smoothed lines are the survival curves fitted by the Weibull models.

DISCUSSION

Self-etching/self-adhesive luting agents are a relatively new category of luting agent. Described as being almost “universal” in their application, they are still resin luting agents. It must be determined whether they are capable of performing like conventional etch and rinse resin luting agents. Conventional resin luting agents have provided the greatest bonding ability for indirect restorations, particularly when required on over-tapered preparations when compared to zinc phosphate and glass ionomer cements.¹⁸ The self-etching/self-adhesive luting agents simplify the cementation technique and have the potential to decrease post operative sensitivity and technique sensitivity.^{1,10}

It is desirable to test and know the comparative nature of the newer self-etching/self-adhesive luting agents, with respect to the physical and mechanical properties that they possess, to ultimately estimate clinical performance. The current study was designed to test and compare the flexural strength and shear bond strength of some self-etching/self-adhesive luting agents to both a conventional resin luting agent and a resin-modified glass ionomer luting cement.

Considerable differences in flexural and shear bond strengths were noted among the tested luting agents. The results of the tests apply to the specific conditions of this study and all specimens were treated the same throughout the study.

FLEXURAL STRENGTH

There was a significant variation in the results of the three-point-bending tests of the six materials. The resin-modified glass ionomer luting cement, FP, had the lowest flexural strength, consistent with other studies.^{5,9}

The tested self-etching/self-adhesive resin luting agents had greater flexural strength than the resin-reinforced glass ionomer luting cement tested. For comparison between the etch and rinse resin luting agent and the tested self-etching/self-adhesive luting agents, the flexural strengths varied in significance. At 90 days, SC ($p=0.2010$) was not statistically different from RA. At both 24 hours and 90 days, MA had greater flexural strength than RA. ME and RU had lower flexural strengths than RA for both time periods. MA uses a separate self-etching primer during its bonding procedure. The primer was not incorporated into the fabrication of the flexural strength specimens. Thus, a reasonable expectation was that the flexural strength beams of MA would have had similar chemical composition to a traditional resin luting agent. The results did not support that expectation.

The aging of the flexural strength specimens, by water storage and thermocycling, had a significant effect on all of the materials tested ($p<0.05$). All of the materials showed a significant decrease in flexural strength, except for FP. FP showed an increase in flexural strength from 24 hours to 90 days. This was consistent with a study that showed an increase in flexural strength over a period of 150 days.⁹ The increase in strength is believed to be due to the maturation and reconstruction of the silicate network during the period of time after the gelation that occurs as the cross-linking is being completed.³¹

SHEAR BOND STRENGTH

Stresses placed on tooth-restoration complexes present in a variety of types, mainly tensile and shear.¹⁵ The shear bond strength test was used in this study.

Indirect resin composite cylinders were luted to dentin surfaces with the intention of reproducing the clinical situation of delivering an indirect restoration. Adhesion of the luting cements to the processed composites has been difficult to achieve, so it has been recommended to roughen the surface by sandblasting, followed by silanation, to enhance the retention between resin luting agents and the indirect composites.²⁵ The resulting data were difficult to directly compare to other studies due to differences in materials used and testing conditions. For example, some studies applied cylinders of luting materials directly to the dentin in order to try to get pure bond strength data without any possible bonding effects from restorative materials.^{4, 12}

It is important to consider the difference in failure type. The difference between an adhesive failure (luting agent-dentin or luting agent-restorative material) and a cohesive failure (in luting agent) may also give an indication of material properties of the luting agents. Studies support that the self-etching/self-adhesive luting agents tend to favor adhesive failures,¹³ whereas the etch and rinse luting agents favor cohesive failures¹⁷ or mixed adhesive/cohesive failures.^{4, 13} No evaluation was done in the present study to determine the type of failure in the shear bond strength test. If there have been cohesive failures within the material itself, then it can be assumed that the adhesive bonding effectiveness would be at least superior to the cohesive strength. This may be particularly important with the FP, reinforced glass ionomer luting cement, since glass ionomer cements have been shown to fail more frequently in a cohesive manner.¹ Adhesive failures might have occurred at the dentin-luting agent interface, or at the indirect resin-luting agent interface. The intention was to have the dentin-luting agent interface evaluated in this study. A future look at the debonded specimens may indicate a

specific deficiency in some of the materials tested with regard to their bonding effectiveness to either particular substrate.

Thermocycling has been shown to possibly have a significant effect on bond strength to dentin.^{7, 12} However, it has also been shown that it might not have any effect on dentin bond strength, while significantly decreasing bond strength to enamel for the self-etching/self-adhesive resin luting agents.⁴

This study found that water storage and thermocycling significantly decreased ($p < 0.05$) the bond strengths of FP and ME. There were no significant differences between the time groups of MA ($p = 0.3210$), RU ($p = 0.2760$) and SC ($p = 0.7010$). The present study found that the shear bond strength of the etch and rinse resin luting agent, RA, significantly increased ($p = 0.0054$) after water storage and thermocycling. This type of increase had been previously reported with resin luting agents and self-adhesive resin luting agents to various ceramics over 14 days,¹⁶ but no increases have been found in dentin bond strengths.

It is not known why the RA group had such an increase in shear bond strength after 90 days and thermocycling in this study. A possible explanation may result from the fact that, as a dual-curing resin, the RA resin luting agent may continue to undergo polymerization after 24 hours. There may be a difference in the degree of conversion between 24 hours and 90 days. The flexural strength showed a decrease in that time, as the specimens were completely exposed to the water and thermocycling during that storage period. The shear bond strength specimens may have had some protection from the effects of water, by the presence of dentin and the indirect resin material, not allowing as much water sorption and subsequent degradation of the luting agent. Further testing of

the material and possible re-testing of these groups with a larger load cell might reveal different results.

No difference was found between MA and RU after thermocycling, similar to results found in another study.¹² Taking into consideration the effect of aging by thermocycling, it is difficult to compare between studies, as shown by different results having been found between sites in a multi-centered trial.¹²

Originally, a sample size of 20 for the shear bond strength testing was planned. Some additional samples (4 per group) were made in the event of unplanned loss. Due to some premature debonding and also some failure to debond, there is a variation of sample sizes for the shear bond strength groups. The failures to debond happened because of using a small (125N) load cell, and approaching the maximum load sometimes before debonding could occur. The differences in group sample sizes did not induce any difference in the calculated results. Pre-testing failures have been noted in other studies.^{7,}
⁸ RA and RU were the only groups that did not have any pre-testing failures. RA, RU and MA were the only groups that had specimens that failed to debond.

Differences in the performance of the variety of the self-etching/self-adhesive luting agents, in this *in vitro* study, may be explained by the difference in physical properties of the materials. A 2007 study in which three of the tested agents (ME, SC and RU) were tested for pH value, film thickness, filler particle percentage, and morphological changes, showed that there were significant differences in the materials tested and that might lead to a difference in clinical performance.¹⁹ Of these materials, there were not any significant differences in filler particle percentage or film thickness, but there were significant differences in the pH values.¹⁹

The newer resin luting agents with self-adhesive capability are formulated to dispense in an acidic state, with a pH well below the neutral level of 7 (approximately pH 2). This allows them to demineralize and penetrate into the tooth. Reactions in the oral environment cause the pH levels to increase as these materials polymerize. In most cases, however, these cements do not reach neutral. Neutralization allows the luting agent to become more hydrophobic, a prerequisite to remaining intact in a moist environment. (RelyX Unicem Technical Product Profile, 3M ESPE). The Han et al.¹⁹ study supported this for some of the materials used in the current study. Even after 48 hours, the SC (pH 4) and ME (pH 2.4) luting agents remained very acidic, whereas RU (pH 7) achieved neutral acidity.¹⁹ It is speculated that at a prolonged low pH condition, there might be an adverse effect on luting agent-tooth interface.¹⁹ This may explain why the current study showed SC and ME performing equal to each other at both 24 hours ($p=0.3090$) and 90 days ($p=0.3400$), and how RU performed significantly better than both SC or ME at both 24 hours and 90 days ($p<0.05$). Similar to the data in this study, other studies have shown that RU had outperformed ME in testing.^{6, 11}

The present study did not evaluate the bond strength to enamel, but based on many other studies, the self-etching/self-adhesive luting agents bonding to enamel is inferior to etch and rinse systems.^{3, 4, 6, 8, 11} The bond strength to enamel has been shown to improve and become comparable to etch and rinse systems when preceded by acid etching, but that acid etching prior to applying a self-adhesive luting agent showed a detrimental effect when bonding to dentin.^{3, 8} These findings may make it reasonable that self-etching/self-adhesive resin luting agents might be used in any clinical situation, but to selectively etch only enamel and not dentin would require great precision. This would

contradict the idea that self-etching/self-adhesive resin luting agents are less technique-sensitive and less time-consuming to use.

Shear bond strength was chosen for this study. Microtensile bond strength is another frequently performed test and is referenced many times. The study design allowed for the larger testing surfaces required for the shear bond strength test. The shear test is easier to perform, as well.¹³ The absolute values of the different tests cannot be directly compared between studies, but conclusions can be made regarding the rankings of the tested materials.¹

Despite a short term clinical study that showed similar performance of RU compared to zinc phosphate cement,²¹ clinical data is insufficient, as the self-etching/self-adhesive luting agents have only been available since 2002.

The testing procedures of this study differed in many ways from a clinical situation, which must be remembered when trying to extrapolate any direct clinical implications.

When it is time to choose a material for a given luting opportunity, almost every situation is unique. There are many different variables to consider: restoration type (metal, composite, silicate based ceramic, alumina or zirconia ceramics); tooth substrate (enamel margins, dentin, metal or composite restorative build-up materials); preparation taper; tooth location and functional load expected. A different material may be preferred for each combination. As a clinician, and for convenience, it would be desirable to be able to use the same luting agent for every situation. Supply ordering decisions would also be simplified.

There are many other self-etching/self-adhesive resin luting agents on the market. There is potential for some to provide mechanical performance similar to conventional resin luting agents. They also offer a simpler, less technique-sensitive approach to luting indirect restorations. However, due to the variability between the different products tested, it may not be assumed that all of the luting agents in this classification are capable of the same performance. Further investigation of more properties and performance of the new materials is necessary as other materials become available. Along with clinical studies based on performance, it will be many more years until a clear picture is available to determine whether these self-etching/self-adhesive resin luting agents hold up to their potential when compared to traditional resin luting agents.

The null hypothesis that all of the luting agents would not have differences in flexural strength at 24 hours or 90 days was rejected. The only luting agents that did not have significantly different flexural strengths at either time period were the 90 day RA and SC groups.

The null hypothesis that all of the luting agents would not have differences in shear bond strength at 24 hours or 90 days was also rejected. At 24 hours, RU and MA performed as well as RA. At 90 days, however, RA significantly outperformed all others.

Based upon the results of this *in vitro* study, it can be concluded that the self-etching/self-adhesive luting agents, in the flexural strength and shear bond strength tests, performed at least as good as the resin-modified glass ionomer luting cement. In some cases they were as good as the traditional etch and rinse resin luting agent.

SUMMARY AND CONCLUSIONS

Traditional resin luting agents generally have had mechanical properties that are superior to the newer so-called “universal” self-etching/self-adhesive resin luting agents. However, recent reports indicate that some properties of these new luting agents have been improved, approaching those of the traditional etch and rinse resin luting agents. Dentin bond strengths are greater than enamel bond strengths of the self-etching/self-adhesive luting agents.

The objective of this study was to test some mechanical properties of four of these self-etching/self-adhesive resin luting agents (Maxcem Elite, Multilink Automix, RelyX Unicem, SmartCem 2) and compare them to a traditional etch and rinse resin luting agent (RelyX ARC) and a resin-modified glass ionomer luting cement (Fuji Plus), both of which have longer histories of clinical success. By comparing the properties of the newer luting agents to the older materials, it may be possible to determine how clinically successful the newer luting agents may be.

The flexural strength and shear bond strengths of the materials were tested after storage in distilled water for 24 hours or 90 days. The 90 day groups were thermocycled to simulate accelerated aging. A Weibull-distribution survival analysis was performed.

The results revealed significant differences in the flexural strength of all materials tested at 24 hours. Extended water storage and thermocycling had significant effects on the flexural strength of all groups. After 90 days and thermocycling, only SC and RA were not significantly different ($p=0.2010$). At both time periods, FP had the lowest and MA the highest flexural strength.

The shear bond strength test results showed RU, RA, and MA to have the highest bond strengths, followed by FP, while ME and SC had the lowest at 24 hours. After 90 days and thermocycling, RA had significantly higher bond strength than all other groups, followed by RU and MA, while SC, FP, and ME had the lowest. The effect of time in water and thermocycling showed mixed results. The MA, RU, and SC groups showed no significant effect of the extended time in water and thermocycling.

The self-etching/self-adhesive resin luting agents all performed at least as well as FP, with the exception of SC (24 hour shear bond strength). They did not perform as well as RA, with the exception of SC (90 day flexural strength), MA (24 hour shear bond strength, 24 hour flexural strength, and 90 day flexural strength), and RU (24 hour shear bond strength). Based on a review of the literature and the results of this study, the newer luting agents should expect to have clinical success at least as good as resin-modified glass ionomer luting cements. They should approach the clinical success of traditional etch and rinse resin luting agents for the routine luting of indirect restorations to preparations consisting predominantly of dentin. However, for clinical applications that have sufficient enamel present and/or high-strength and high-retentive requirements, the traditional etch and rinse resin luting agents would still be the material of choice.

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ABSTRACT

FLEXURAL STRENGTH AND SHEAR BOND STRENGTH
OF SELF-ETCHING/SELF-ADHESIVE
RESIN LUTING AGENTS

by

Richard S. Adcock

Indiana University School of Dentistry

Indianapolis, Indiana

Traditional resin luting agents generally have mechanical properties that are superior to the newer so-called “universal” self-etching/self-adhesive resin luting agents. However, recent reports indicate that some properties of these new luting agents have been improved, approaching those of the traditional etch and rinse resin luting agents.

The objective of this study was to test some mechanical properties of four of these self-etching/self-adhesive resin luting agents [Maxcem Elite (ME), Multilink Automix (MA), RelyX Unicem (RU), SmartCem 2 (SC)] and compare them to a traditional etch and rinse resin luting agent [RelyX ARC (RA)] and a resin-modified glass ionomer luting cement [Fuji Plus (FP)], both of which have much longer histories of clinical success. By comparing the properties of the newer cements to the standards, it may be possible to determine how clinically successful the newer cements may be.

The mechanical properties tested were flexural strength (FS) and shear bond strength (SBS). The FS test included making beams of each material, storing them in water for periods of time (24 hours and 90 days) and then performing a three-point bending test on a universal testing machine. The 90 day groups were thermocycled. The SBS test involved preparing human molar specimens, making flat dentin surfaces. Composite cylinders were fabricated, luted to the dentin surfaces with each of the materials tested, stored in water for periods of time (24 hours or 90 days), and then a knife edge shear test was performed on a universal testing machine. The 90 day groups were thermocycled. A Weibull-distribution survival analysis was performed.

The results revealed significant differences in the FS of all materials tested at 24 hours. After 90 days and thermocycling, only SC and RA were not significantly different. At both time periods, FP had the lowest and MA the highest FS. The SBS results showed MA, RA, and RU to have the highest bond strengths; SC and ME the lowest at 24 hours. After 90 days and thermocycling, RA had significantly higher bond strength than all other groups; ME, FP and SC had the lowest.

The self-etching/self-adhesive resin luting agents all performed at least as well as FP, with the exception of SC (SBS 24 hour). They did not all perform as well as RA, with the exception of SC (FS 90 day), MA (SBS 24 hour, FS 24 hour and 90 day), and RU (SBS 24 hour). The newer luting agents should expect to have clinical success, regarding flexural strength and shear bond strength, at least as good as resin-modified glass ionomer luting cements and approach the level of traditional etch and rinse resin luting agents.