

Effect of Light Activation on Resin-modified Glass Ionomer Shear Bond Strength

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Clinical Relevance

All light-cured resin modified glass ionomers should be light polymerized after placement. This will greatly increase the bond strength of the material and should improve the longevity of the restoration.

SUMMARY

Objective: Recent studies confirmed that resin-modified glass ionomers (RMGIs) set on the basis of two competing mechanisms, an acid-base reaction and a light-activated resin polymerization. This study evaluated the effect of the setting mechanism on bond strength by measuring the shear bond strength of three

RMGIs to dentin with and without light activation.

Methods: Sixty human molars were ground to midcoronal dentin and randomly divided into six even groups: 1) Ketac Nano (KN), 2) KN without light cure (woLC), 3) Fuji Filling LC (FF), 4) FF woLC, 5) Fuji II LC (FII), and 6) FII woLC. The dentin surfaces of the specimens were conditioned/primed according to the manufacturers' instructions. A 1.54-mm diameter plastic tube was filled with RMGI material and affixed to the dentin surface. Groups 1, 3, and 5 were light cured for 20 seconds, and groups 2, 4, and 6 were immediately placed in a damp dark box with no light curing at 37°C for 24 hours. Shear bond strength testing was performed in an Instron device at 1 mm/min. Data were analyzed with a one-way analysis of variance (ANOVA) and Tukey/Kramer test ($\alpha=0.05$).

Results: Mean \pm standard deviation shear bond strength values (MPa) are: 7.1 \pm 4.2 (KN), 11.7 \pm 3.9 (FF), 10.2 \pm 3.2 (FF woLC), 12.5 \pm 5.1 (FII), and 0.3 \pm 0.4 (FII woLC). Two

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KN, all KN woLC, and seven FII woLC specimens debonded before testing. Tukey/Kramer analysis revealed no significant differences in bond strength between the three light-cured RMGIs. KN and FII showed significantly lower bond strength without light cure, but no significant difference was observed between FF and FF woLC.

Conclusions: The results of this study strongly suggest that light activation is necessary to obtain optimal bond strength between RMGI and dentin. FF may contain components that chemically activate resin polymerization. Clinically, KN and FII need to be light cured after placement of these RMGIs.

INTRODUCTION

Resin-modified glass ionomers (RMGIs) were introduced as a hybrid between conventional resin composites and glass ionomers.¹ Because these materials do not have the strength or wear resistance of conventional composites,² their use as posterior occlusal load bearing restoratives is questionable.³⁻⁶ Resin-modified glass ionomers have poor retention as pit and fissure sealants⁷ but excellent longevity as cervical restorations⁸ and as liners and bases.⁹ In these applications, it is critical for the RMGI to develop an effective bond to tooth structure.

Traditional glass ionomers bond to dentin by an ionic bond with hydroxyapatite, and conventional composite materials bond to dentin through micro-mechanical interlocking with collagen fibrils and dentinal tubules. RMGIs contain components of glass ionomers (fluoro-aluminosilicate glasses and polyacrylic acid) as well as resin composites (photo or chemical initiators and methacrylate monomers).¹⁰ Due to their hybrid nature, RMGIs bond to dentin through both an ionic bond between polyacrylic acid and hydroxyapatite and mechanical interlocking with collagen and the resin monomer.¹¹ The initiation of this bond can be attributed to the various methods of polymerization of the RMGIs.

These materials polymerize by up to three mechanisms: 1) an acid-base reaction between the polyacrylic acid and the fluoro-aluminosilicate glass, 2) a photo-initiated free-radical reaction between methacrylate monomers, and 3) a chemically-initiated reaction between methacrylate monomers remaining after photo-initiation.¹² Recent studies have shown the acid-base and photo-initiated free-radical reactions inhibit each other, with photopolymerization of RMGIs reducing the acid-base reaction.^{10,13} Another

recent study concluded that the acid-base induced chemical interaction between RMGI and dentin is the main mechanism of bonding with RMGI.¹⁴ Therefore, it is hypothesized that RMGIs allowed to polymerize without light activation will develop bond strength through the ionic bonding between RMGIs and dentin and should therefore have an equivalent or higher bond strength than light-cured RMGIs.

To test the hypothesis, the bond strength of RMGI to dentin with and without light polymerization was compared. It was assumed that bond strength to dentin with uncured RMGI would be attributed to an acid-base reaction. The null hypothesis was that there would be no difference between the light-cured and uncured groups.

MATERIALS AND METHODS

Sixty freshly extracted human molars were collected from the University of Alabama at Birmingham School of Dentistry. The occlusal surfaces of the teeth were ground to expose midcoronal dentin with a grinding wheel (Model 108; Wehmer Co, Addison, IL, USA). Specimens were polished with 600-grit SiC paper under water. The teeth were evenly and randomly divided into six groups: 1) Ketac Nano (KN), 2) KN without light cure (woLC), 3) Fuji Filling LC (FF), 4) FF woLC, 5) Fuji II LC (FII), and 6) FII woLC.

Immediately following polishing, all groups (n=10) were conditioned and primed according to the manufacturers' instructions (Table 1). A transparent rubber tube (1.54 mm inner diameter and 4 mm length) was filled to a depth of 2 mm with the corresponding RMGI and pressed to the prepared surface of each sample. Groups 1, 3, and 5 were then light polymerized with an Elipar S10 LED curing light (813 mW/cm² 3M ESPE, Seefeld, Germany) from above and both sides for 20 seconds and placed in a damp, dark box. Groups 2, 4, and 6 were placed in the damp dark box within one minute after affixing the rubber tube without light curing. The box was then placed in a 37°C incubator (Queue; ThermoElectron, Waltham, MA, USA) for 24 hours.

The specimens were removed from storage and dried with absorbent wipes. The rubber tube was cut away to reveal a 1.54-mm diameter RMGI cylinder bonded to each specimen. The specimens were loaded into a custom fixture in a universal testing device (Instron, model 4411, NVLAP, Canton, MA, USA) that secured the teeth with the bonded RMGI from three sides. A blade attached to the upper member of the Instron was used to fracture the

Table 1: Products Used in This Study			
Material	LOT	Composition	Directions
Ketac Nano – A3	20090515	Silane-treated glass (40%), Silane-treated zirconia (20%), PEGDMA (5%), silane-treated silica (5%), HEMA (1%-15%), Glass powder (<5%), Bis-GMA (<5%), TEGDMA (<1%)	—
Ketac Nano GI Primer	7AC	Water (40%), HEMA (35%), copolymer of acrylic and itaconic acids (10%)	Rinse and dry tooth, apply primer for 15 s, dry for 10 s, and light cure for 10 s
GC Fuji Filling LC – A2	0905011	Paste A: alumino-silicate glass (75%), HEMA (10%), UDMA (2%); paste B: water (20%), polyacrylic acid (20%), UDMA (12%), silica (10%)	—
GC Self Conditioner	0906021	Ethanol (28%), water (30%), HEMA (20%), 4-methacryloxyethyl trimellitate anhydride (5%)	Rinse and dry tooth, apply conditioner for 10 s, dry for 5 s
GC Fuji II LC Capsules – A2	0804255	Powder: alumino-silicate glass (75%); liquid: polyacrylic acid (20%), HEMA (35%), propriety ingredient (5%), 2,2,4 trimethyl hexamethylene dicarbonate (5%), TEGDMA (4%)	—
GC Cavity Conditioner	0604191	Polyacrylic acid (20%), water (77%), aluminum chloride hydrate (3%), blue food additive (<0.1%)	Rinse and dry tooth, apply conditioner for 10 s, rinse and blot dry
Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, hydroxy ethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.			

specimens at a crosshead speed of 1 mm/min. The maximum load required to separate the RMGI cylinder from dentin was recorded. Shear bond strength (SBS) was determined: maximum load/surface area of composite post. The fracture surfaces of the specimens were examined under light microscopy at 100× magnification (VHX-600, Keyence Co, Osaka, Japan) to determine the mode of failure.

The normal quantile plot of shear bond strength by treatment was examined to determine acceptable normality for the data. Differences between treatment groups were analyzed statistically by analysis of variance (ANOVA) ($\alpha=0.05$). Groups were compared to the mean using a Tukey *post-hoc* test ($\alpha=0.05$).

RESULTS

Table 2 shows the mean SBS and standard deviations of all groups tested. Two KN, all KN woLC, and seven FII woLC specimens debonded before testing. For these groups, the specimens with premature failures were given a value of zero and included in the statistical analyses. A one-way ANOVA revealed significant differences among testing groups

($p<0.05$). A Tukey/Kramer analysis revealed no significant differences in bond strength between the three light-cured RMGIs (Ketac Nano, Fuji II LC, and Fuji Filling). KN was assumed to be greater than KN woLC because the bond with KN woLC was too weak to survive testing. FII was significantly

Table 2: Mean Shear Bond Strength (MPa) and Standard Deviation of the Resin-modified Glass Ionomers Tested (N=10)	
Group	Mean ± SD
1 Ketac Nano (KN)	7.07 ± 4.21
2 Ketac Nano without light cure (KN woLC),	0
3 Fuji Filling LC (FF)	11.66 ± 3.91
4 Fuji Filling LC without light cure (FF woLC)	10.18 ± 3.23
5 Fuji II LC (FII)	12.46 ± 5.06
6 Fuji II LC without light cure (FII woLC)	0.26 ± 0.45

Table 3: Mode of Failure of Each Group

Group	Adhesive	Mix	Cohesive
1 Ketac Nano (KN)	4	5	1
2 Ketac Nano without light cure (KN woLC),	8	2	—
3 Fuji Filling LC (FF)	7	3	—
4 Fuji Filling LC without light cure (FF woLC)	8	2	—
5 Fuji II LC (FII)	4	3	3
6 Fuji II LC without light cure (FII woLC)	8	2	—

greater than FII woLC ($p < 0.05$), but no significant difference was observed between FF and FF woLC. Fracture modes of failure are presented in Table 3. Uncured specimens most frequently fractured by adhesive failure, while some light-cured specimens failed cohesively. The ratios of adhesive:mixed:cohesive fractures for cured KN and FF, respectively, were 4:5:1 and 4:3:3, and uncured KN and FF were 8:2:0 for both. The same ratio for cured and uncured FII samples were 7:3:0 and 8:2:0, respectively.

DISCUSSION

Light-cured RMGIs develop bond strength through the fast-acting light-initiated reaction of resin monomers as well as an acid-base reaction and a chemically-initiated free-radical reaction. Non-light-cured RMGIs polymerize through only the acid-base and chemical-initiated free-radical reactions. Our study reports low bond strength of non-light-cured RMGIs (excluding FF) and relatively high bond strength of cured RMGIs. Therefore, our results suggest that most RMGIs do not develop adequate bond strength without light-activated resin polymerization, rejecting the null hypothesis.

The non-light-polymerized Fuji Filling LC was the only non-light-cured material to produce the same SBS as the light-cured specimens. The manufacturer of this material indicated that a chemical initiator is included in FF but not FII, and it is possible that the chemical initiator activated the free radical resin polymerization in FF. The manufacturer of KN indicated that a chemical initiator is not included

in their material. Therefore, the KN woLC and FII woLC groups polymerized only through an acid-base reaction.

The contribution of the acid-base and free radical reactions on the mechanism of bonding of RMGIs has not been completely elucidated, and both mechanisms of bonding have been reported in dental literature. Mitra and others¹⁵ and Coutinho and others¹⁶ analyzed the reaction between methacrylated copolyalkenoic acid (a key component of RMGIs) and pure hydroxyapatite (HAP) crystals. Fourier transform infra-red spectroscopy and X-ray photoelectron spectroscopy analyses indicated an ionic bond between the methacrylated polyalkenoic acid and HAP, suggesting an acid-base reaction. On the other hand, scanning electron microscopy analysis of interfacial microstructure between RMGIs and dentin, performed by Coutinho and others,¹⁶ showed a submicron hybrid layer of RMGI and an analysis by Mitra and others¹⁵ showed tag-like structures of RMGI penetrating dentin. Carvalho and others¹⁷ describe a similar phenomenon terming it a demineralized, resin-infiltrated zone—a characteristic of free-radical polymerization based bonding.

A study by Cardoso and others¹⁴ measured the bond strength of RMGIs bonded to fractured dentin with and without the use of a conditioner. Micrographs revealed the presence of a hybrid layer in the conditioned dentin specimens but not in the unconditioned specimens. However, no difference in bond strength was measured between the conditioned and unconditioned dentin groups. Based on these observations, the authors suggested that the mechanical interlocking provided by a hybrid layer is not the primary mechanism of bonding with RMGIs. The authors later acknowledged the possibility of a nanometer-scaled hybrid layer created by the mild self-etching properties of the polyalkenoic acid present in the RMGI. In summary, the effect of either bonding mechanism towards the adhesion of RMGIs to dentin is still unclear.

Traits of both ionic (acid-base) and resin infiltration (free-radical) bonding are reported in current literature; however, this study suggests resin infiltration creates the strength of the bond of RMGI to dentin. *In vitro* testing has shown that the SBS of glass ionomer materials is weaker than that of resin composites. Additionally, previous studies have reported that RMGIs have SBS values greater than glass ionomer materials but less than composites.^{18,19} Therefore, it would be expected that conditions that enable a RMGI to behave like a

resin composite material, such as light polymerization, would favor higher SBS values.

The failure analysis revealed that more of the light-cured specimens of FII and KN failed by cohesive and mixed failures. This result further indicates that a stronger bond was formed between light-cured RMGI and dentin than uncured RMGI and dentin. The failure modes of cured and uncured FF were nearly identical, most likely due to the chemical-initiated free radical polymerization of the uncured FF.

All RMGI groups in this study were applied preceding a coat of conditioner or primer as indicated by the manufacturer. FF and FII both use a conditioner. Conditioners can remove the smear layer and partially demineralize dentin,²⁰ and the use of a conditioner can significantly improve bond strength.^{21,22} KN uses a light-cured primer. The requirement to light polymerize this primer indicates that it contains a photo-initiated resin component—similar to an adhesive. Pereira and others²³ concluded that the SBS of RMGI to dentin improved with the use of an adhesive, a technique that facilitates resin bonding. The layer of adhesive, however, decreases the fluoride release from these materials.²⁴⁻²⁵ The results of Pereira and others reinforce the finding that light-activated resin polymerization provides the strength of the bond between RMGI and dentin.

A limitation of this study is that only three RMGIs were examined. Varying chemistries of other RMGIs may produce different results. Future studies could compare the SBS of additional RMGIs.

CONCLUSIONS

KN and FII have increased bond strength after light polymerization. The results suggest that light activation is needed to obtain optimal bond strength to dentin with some RMGIs. Clinically, KN and FII should be light cured after placement to achieve optimum bond strength.

Conflict of Interest Declaration

The authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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