

Original Article

Effect of acid pre-conditioning and/or delayed light irradiation on enamel bond strength of three resin-modified glass ionomers

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ABSTRACT

Background: Polymerization of resin-modified glass-ionomers (RMGIs) is mediated through two competing mechanisms: An acid-base reaction and a light-dependent resin polymerization. Furthermore, pre-conditioning with acid has resulted in an increase in enamel bond strength of some RMGIs. This *in vitro* study evaluated the effect of pre-conditioning and/or delayed irradiation on bond strength of three RMGIs to enamel.

Materials and Methods: In this *in vitro* study, 144 flat enamel surfaces of human molars were ground using consecutively finer abrasives up to 600-grit silicon carbide paper. Each surface was rinsed and gently air-dried (n = 12). The RMGIs (Fuji II LC Improved; Ionolux and Vitremer) were bonded to enamel surfaces using the following protocols: Groups 1: Based on manufacturers' instructions; Groups 2: Pre-conditioning with phosphoric acid for 30 s; groups 3: A 2-min delay in irradiation; groups 4: Pre-conditioning with acid for 30 s plus a 2-min delay in light activation. After 24-h storage at 37°C and 500 rounds of thermocycling, the samples underwent shear bond strength (SBS) test. Data was analyzed with 3-way ANOVA and Tukey HSD test ($\alpha = 0.05$).

Results: There were significant differences between the study groups (P < 0.001). Acid-pre-conditioning increased Fuji II LC SBS values; it significantly decreased SBS values of Vitremer but had no effect on SBS values of Ionolux. Ionolux and Vitremer exhibited decreased enamel bond strength after a delay in light activation (P < 0.05). A 2-min delay in light activation combined with acid pre-conditioning increased RMGI SBS values only for Fuji II LC.

Conclusion: Within the limitations of the present study, the effect of acid pre-conditioning, delaying irradiation and both on enamel bond strength of RMGIs was material-dependent. Further investigations are recommended.

Key Words: Acid pre-conditioning, bond strength, delayed irradiation, enamel, resin-modified glass-ionomer

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INTRODUCTION

Glass-ionomers (GI) were manufactured in the late 1960s and introduced to the dental field in 1972.^[1] Resin-modified glass-ionomers (RMGIs) are



hybrids of conventional composite resins and GI.^[2] RMGIs are composed of glass-ionomer ingredients (fluoro-aluminosilicate glasses and polyacrylic acid) and composite resins (photo or chemical initiators and methacrylate monomers).^[3] RMGIs have the advantage of bonding to dentin through both an ionic bond between polyacrylic acid and hydroxyapatite and mechanical interlocking with collagen and the resin monomer due to their hybrid nature. Bond initiation is mediated by various methods of RMGI polymerization.^[2,3]

These materials undergo polymerization by a maximum of three mechanisms: (1) An acid-base reaction taking place between the polyacrylic acid and

the fluoro-aluminosilicate glass; (2) a photo-initiated free-radical reaction occurring between methacrylate monomers; and (3) a chemically-initiated reaction involving methacrylate monomers remaining after photo-activation.^[4]

Based on the results of some recent studies the acid-base and photo-initiated free-radical reactions have a reciprocal inhibitory effect on each other, and photoactivation of RMGIs decreases the rate of the acid-base reaction.^[5-8] The results of a recent study showed that the chief mechanism of bonding with RMGI is mediated by the acid-base chemical interaction between RMGI and dentin.[7] A recent study showed that some RMGIs polymerized without photo-activation achieve bond strength via ionic bonding between RMGIs and dentin; as a result, they exhibit equivalent or higher bond strength values compared to photo-polymerized RMGIs.[4] In contrast, photo-polymerization is required to achieve sufficient bond strength with dentin with the use of some RMGI brands, including Ketac Nano and Fuji II LC.[4] They strongly believe that photo-polymerization is absolutely necessary for optimal bond strength between RMGI and dentin. Thomas, et al. evaluated the effect of delaying visible light-curing process on the efficacy of polymerization and orthodontic bond strength of a dual-curing RMGI (Fuji Ortho LC).[5] They reported that delaying the photo-activation of an orthodontic RMGI restricts the acid-base reaction rate, reducing conversion degree of photo-activated components, and effecting changes in the structure of the material, without compromising orthodontic shear bond strength (SBS). Recently, a study showed that enamel bond strength of an RMGI restoration might improve by delaying the light activation procedure when a cavity conditioner is used. In contrast, delaying the light activation of an RMGI restoration compromises RMGI bond strength to dentin. [6]

RMGIs still exhibit lower bond strength values compared to composite resins and various bonding protocols have been proposed to increase their bond strength. Since RMGIs do not exhibit the strength or wear resistance comparable to that of conventional composite resins, [9] their use in posterior load-bearing areas is not recommended. [10,11] RMGIs exhibit poor retentive properties when used as pit and fissure sealants; [12] however, they exhibit superb longevity in cervical areas [13] and when used as liners and bases. [14] In such applications, RMGIs should form an effective bond to tooth structure with the use of dental

adhesives and etching the enamel with phosphoric acid. [15] A number of factors potentially contribute to a proper bond strength between enamel and RMGI, which include type of the enamel conditioner, [15] duration of the etching procedure, [16] the oral cavity environment, and clinician's expertise and dexterity. [17]

Some RMGI manufacturers recommend the use of 10% polyacrylic acid enamel conditioner and a light-curing duration of 20-40 s. RMGIs have higher bond failure rates compared to composite resins; therefore, different bonding protocols have been evaluated to improve their initial bond strength. These protocols include use of 37% phosphoric acid in order to etch the enamel, [15] use of other conditioners [18] before application of RMGI and extension of light-curing times for an extra 5-10 s. [17]

For orthodontic applications, measures should be adopted to minimize decalcification problems and achieve optimal bond strength values; however, for the restorative applications the final goal is to achieve a high bond strength value with the least microleakage. A few studies have shown that application of self-etching adhesives, rather than dentin or cavity conditioners of RMGIs, improves the SBS^[19,20] and marginal integrity^[21,22] of RMGIs to tooth structures.

It has recently been hypothesized that delayed polymerization of RMGIs will improve bond strength via ionic bonding between RMGIs and enamel, resulting in bond strength values equivalent to or higher than those of light-cured RMGIs, especially for orthodontic applications.^[5] On the other hand, some researchers have assumed that etching with phosphoric acid might allow the resin component to better bond to the enamel surface for restorative purposes.[6,7,20] Therefore, the aim of the present study was to investigate the effect of delayed irradiation and/or pre-conditioning of three RMGIs on bond strength to enamel. The specific hypothesis tested in this study was that a delay in photopolymerization and/or acid pre-conditioning does not influence the bond strength of RMGIs to enamel.

MATERIALS AND METHODS

In this invitro study, 72 sound extracted human third molars were used. The teeth were stored in 0.2% thymol solution at 4°C for 3 months after extraction and used for the purpose of the present study after informed patient consent was obtained, based on the guidelines of the Medical Ethics Committee of Isfahan

University of Medical Sciences. The tooth roots were removed; then the crowns were sectioned mesiodistally and embedded in flat cylindrical molds made of acrylic resin, with the buccal and lingual surfaces placed horizontally. All the buccal and lingual enamel surfaces were ground using wet silicon carbide papers up to grit 600 to achieve flat and smooth enamel surfaces. Three brands of RMGI restorative materials were selected for this study: Fuji II LC I: Improved Version; Ionolux and Vitremer) [Table 1]. The test materials were bonded to enamel surfaces according to the following distinct protocols:

Groups 1 (control): The material was mixed, injected into the mold and then light-activated according to manufacturer's instructions.

Groups 2: The enamel surfaces were pre-conditioned with phosphoric acid (37.5% H3Po4, Kerr, Orange, CA, USA) for 30 s; after rinsing and drying, each material was bonded similar to group 1.

Groups 3: Light-curing was delayed; the material was mixed, injected into the mold, formed, and then allowed to set without the application of light for 2-min, and then light-activated according to manufacturer's instructions.

Groups 4: Enamel surfaces were pre-conditioned with acid and light activation was delayed; enamel surfaces

were pre-conditioned similar to the procedure in group 2 and then mixed, introduced into the molds, and light-activated 2-min after application similar to group 3.

Cylindrical plastic molds (Orthorings, Ortho organizers Inc., CA, USA), measuring 2 mm in internal diameter and 1 mm in height, were fixed on enamel surfaces at ambient temperature $(21^{\circ}\text{C} \pm 1^{\circ}\text{C})$. Each one of the RMGICs in the present study was used according to manufacturers' instructions [Table 1]. Two encapsulated materials, including Fuji II LC and Ionolux, were mixed according to manufacturer's instructions in a mechanical mixer (CapMix 1, 3M ESPE, St Paul, MN, USA). For Vitremer, the powder and liquid were hand-mixed according to manufacturer's instructions [Table 1]. For each one of RMGIs, sample subgroups (n = 12) were prepared as follows:

All the RMGI samples were polymerized using a halogen light-curing unit (Coltolux 2.5, Coltene AG, Feldwiesenstrasse Altstätten/Switzerland) with a light output power of 480 mW/cm² at a distance of 1 mm from the RMGI surface. After a 24-h period of storage at 37°C, the specimens were subjected to 500 rounds of thermocycling between 5°C and 55°C

Table 1: Materials used in the study, their compositions and mode of their applications according to the manufacturer instructions

Material name and manufacturer	Manufacturers' directions	Materials compositions
Cavity conditioner (GC, Tokyo, Japan)	Apply with a brush for 10 s, rinse thoroughly	Polyacrylic acid (20%), aluminum chloride (3%), distilled water
Fuji II LC	Shake the capsule, push the plunger until it is flush with main body, place the capsule into a metal GC capsule applier and click the lever once, set the capsule in a mixer and mix for 10 s, load it into the applier and inject in the prepared cavity and cure for 20 s	Powder: Fluoro alumino-silicate glass
(improved version) (GC, Tokyo, Japan)		Liquid: Poly acrylic acid (20-25%); 2-hydroxyl ethyl methacrylate (30-35%); proprietary ingredient (5-15%); 2, 2, 4, trimethyl hexa methylene dicarbonate (1-5%) Powder/liquid: 0/33 g/0/085 ml
Ionolux (Voco GmbH, Cuxhaven Germany)	Shake bottles thoroughly before use. Powder/ liquid-ratio: 3.2 g (1 measuring spoon)/1.0 g (2 drops). Work the powder in two steps in the liquid. Use spatulas of solid plastic or other abrasion proof instruments. Spread out paste several times for a homogenous consistency. Apply material in maximum 2 mm layers, lightcure 20 s per layer	Powder Polyacrylic acid Fluorosilicate glass Amine Liquid HEMA Polyacrylic acid solution Glycerindimethacrylate Urethandimethacrylate Butylhydroxytoluol
Vitremer (3M ESPE, St. Paul, MN, USA)	Apply Vitremer primer for 30 s. Air dries for 15 s. Light cure for 20 s. Mix the Vitremer powder and liquid in 2.5/1 ratio for 45 s. Apply the paste. Light cure for 40 s. Apply finishing gloss. Light cure for 20 s	Powder: Fluoroaluminosilicate glass; redox system Liquid: Aqueous solution of a modified polyalkenoic acid, HEMA

HEMA: Hydroxy ethyl methacrylate; LC: Light-curing

(Mp Based, KARA1000 Inc., Tehran, Iran) with dwell and transfer times of 30 and 12 s, respectively. Subsequent to fixation, the samples underwent SBS test at a crosshead speed of 0.5 mm/min using a universal testing machine (Dartec, HC10, Dartec Ltd, Stourbridge, UK). [23] SBS values were calculated by dividing the force at failure by the initial bonded surface area.

Three-way ANOVA was used to analyze the effect of curing protocol and pre-conditioning on SBS values using SPSS 13. In addition, one-way ANOVA and a post hoc Tukey test were used to assess differences in SBS values between the groups within the materials. Statistical significance was set at P < 0.05. The fracture patterns of RMGI cylinders on enamel surfaces were evaluated under a light microscope (MBC-10, St. Petersburg, Russia) at \times 16 and designated as follows:

- 1. Cohesive fracture: Fracture in the RMGI.
- 2. Adhesive fracture: Fracture in the adhesive interface.
- 3. Mixed fracture: A combination of adhesive/cohesive (RMGI) fracture [Table 2].

RESULTS

SBS values in megapascal (MPa) (mean \pm SD), minimum/maximum values and 95% confidence interval for the groups are presented in Table 2. ANOVA revealed significant differences in SBS values between the study groups (P < 0.001).

Three-way ANOVA revealed that SBS values in the study groups were influenced by "the type of RMGI" (F = 4.653, P = 0.011) and "pre-conditioning" (F = 38.452, P < 0.001), but not influenced by "delaying the irradiation" (F = 0.165, P = 0.685).

Among Fuji II LC groups, specimens in groups 3 and 4 exhibited higher SBS values compared to specimens in groups 1 and 2 (P < 0.05). There were no statistically significant differences between groups 1 and 2 (P > 0.05) [Table 3].

Among Ionolux groups, specimens in group 3 exhibited the highest mean SBS values between the four groups (P < 0.05). A significant difference was observed only between groups 2 and 3 (P > 0.05) [Table 3].

Among Vitremer groups, specimens in group 1 had the highest mean SBS values between the four groups (P < 0.05). A significant difference was observed only between group 1 and the other groups (P > 0.05) [Table 2].

One-way ANOVA revealed significant differences in the control group specimens (P < 0.05). Multiple comparisons by Tukey test for the three restorative materials demonstrated significantly higher SBS values for Vitremer compared to the other two RMGIs (P < 0.05).

Pre-conditioning with acid increased the SBS values of Fuji II LC bond to enamel; however, it did not improve bond strength of the two other RMGIs.

Delay in light-curing process decreased bond strength of all the studied RMGIs to enamel.

The combination of two variables, including pre-conditioning with acid and delaying the light-curing procedure, was material-dependent. In other words, this strategy was successful only for Fuji II LC.

The fracture modes are presented in Table 3. According to the results, the majority of adhesive

Table 3: Different failure modes in the study groups (n=12)

RMGIs Groups	Fuji II LC C-DC-AP-	Ionolux C-DC-AP-	Vitremer C-DC-AP-
010 p 5	DC+AP	DC+AP	DC+AP
Modes of fracture			
Adhesive	5-4-0-1	4-7-2-6	0-5-2-0
Cohesive	3-3-5-4	3-0-3-2	5-1-4-4
Mixed	4-5-7-7	5-5-7-4	7-6-6-8

RMGI: Resin-modified glass-ionomer; C: Control; DC: Delayed curing; AP: Acid pre-conditioning; DC+AP: Delayed curing+Acid pre-conditioning

Table 2: Shear bond strength values (MPa) for the three resin-modified glass-ionomers at four different treatments (mean±SD)

Group	Control	Delayed light curing	Acid pre-conditioning	Acid pre-conditioning+delayed light curing
RMGI				
Fuji II LC	9.34±3.56 ^{Aa}	11.81±5.00 ^{Aa}	17.77±3.66 ^{Ba}	20.25±5.46 ^{Ba}
Ionolux	13.61±3.67 ^{Ab}	10.19±3.34 ^{ABa}	15.52±4.77 ^{ACa}	11.17±2.84 ^{Ab}
Vitremer	19.34±3.59 ^{Ac}	7.13±3.82 ^{Bb}	14.80±2.70 ^{Ca}	15.34±2.78 ^{ca}

RMGI: Resin-modified glass-ionomer; LC: Light-curing; Means followed by different letters show statistical differences: (α=0.05); Capital letters: Comparison of each procedure for each RMGI (row); Lower case letters: Comparison of RMGIs at each procedure (column)

fractures were observed in groups 3 and 4. All the cohesive fractures were found in RMGI.

DISCUSSION

Bond strength of light-activated RMGIs is achieved via the rapid reaction of resin monomers under the influence of radiant energy, an acid-base reaction and a chemically-activated free-radical reaction. Polymerization of non-light-activated RMGIs is mediated via solely acid-base and chemically-activated free-radical reactions. [4,6] The results of the present study showed a low bond strength value for delayed light-activated RMGIs (except for Fuji II LC) and comparatively a high bond strength value for immediately light-activated RMGIs, especially for Vitremer. Therefore, the results indicate that generally RMGIs do not achieve sufficient bond strength without light-initiated resin polymerization, refuting the null hypothesis of this study.

Tests carried out in vitro have shown that GI have lower SBS values compared to composite resins. In addition, previous studies have shown that RMGIs have SBS values greater than those of GI but less than those of composite resins.^[24,25] Current literature is replete with the specifics of both ionic (acid-base) resin infiltration (free-radical) and procedures; however, the results of this study suggest that infiltration of resin is responsible for the bond strength of RMGI to enamel. Therefore, it is expected that conditions contributing to the behavior of an RMGI similar to that of a light-activated composite resin, such as light polymerization on an enamel substrate, will give rise to higher SBS values.

In fact, it appears that it is advisable for RMGI manufacturers to indicate on the product package that it has dual polymerization (both photo-initiated and chemical). In addition, it appears that the behavior of the material is under the influence of which part has an acid-base nature and which part has a resin nature. It is probable that the presence of chemical polymerization as a third polymerization mechanism in some cases influences the recently proposed "network competition" hypothesis. Vitremer material does not polymerize if its powder and fluid are mixed without light activation; however, the results of the present study showed that with light-activation according to manufacturer's instructions approximately 19 MPa of bond strength is achieved, which is comparable to that of composite resin. It is probable that the resin component comprises a sizeable part of Vitremer composition, which does not hold in the case of Fuji II LC and if light-activation is delayed or is withheld the flow of the material decreases significantly, producing a half-polymerized state. In addition, it is possible that the primer of Vitremer is more acidic than the conditioner of Fuji II LC, resulting in higher bond strength with enamel; however, further studies are necessary. Summarily, the various chemical components of complex materials such as RMGIs might give rise to diverse clinical behaviors. Therefore, the clinicians should be provided with more information by the manufacturers so that they will be able to render better restorative, preventive and orthodontic services.

The Fuji II LC material, which underwent delayed light-activation was the only material to achieve the same SBS values as the light-cured specimens and even a little higher SBS mean was noted. Some manufacturers have reported incorporation of a chemical initiator into their material. It appears that presence of a chemical initiator in the RMGI might activate the free-radical resin polymerization in RMGI. Therefore, these materials might polymerize without light-activation in addition to acid-base reaction and might have higher bond strength in the absence or deficiency of light.

In the present study, two of three materials (Fuji II LC and Vitremer) are including conditioner or primer, which be applied on substrate based on manufacturers' instructions. Fuji II LC has its special conditioner. Conditioners can eliminate or alter the smear layer and partially demineralize dentin, [3,26] which results in a significantly higher bond strength.[27] Vitremer has a light-cured primer. The requirement to light-cure this primer is an indication that it has a photo-activated resin component similar to an adhesive. That's why the bond strength of this RMGI is much higher than those of Ionolux and Fuji II LC. Some studies have shown^[22,28] that the SBS of RMGI to dentin increases when an adhesive is applied, which is a technique facilitating resin bonding. Nevertheless, the adhesive layer decreases the release of fluoride from these materials.[29,30] The results of the above-mentioned studies confirm the finding that light-curing of resin provides the bond strength between RMGI and dentin.

Bond strength of Ionolux was less than those of the two other materials under study, which might be attributed to a lack of conditioner in this system and surface preparation, especially considering the fact that a relative increase in bond strength of this material to enamel was observed after conditioning of the enamel in group 3; of course, it is probable that phosphoric acid might be aggressive for the bonding of this material. Further studies are required in this respect.

In the present study, no significant differences were observed in bond strength after light-activation between the immediate (control) and 2-min delay groups for Fuji II LC and Ionolux, but the delayed group of Vitremer exhibited significantly lower bond strength values. The catastrophic decrease in bond strength values for Vitremer in the delayed group is an indication that the light-activation reaction is more critical for the setting reaction of this material. Nicholson, et al.[31] hypothesized earlier that the acid-base and light-activation reactions in RMGIs compete with and inhibit each other. Young[32] showed in an in vitro study that subsequent to the formation of photopolymer cross-linked network after photoactivation, there is a decrease in the diffusion of acid-base reactants. Eliades and Palaghias^[33] reported that the acid-base reaction rate is lower in light-vs. dark-cured RMGIs. Berzins et al.[7] evaluated the theoretical competing reactions in RMGIs and concluded that the light-activated exothermic reaction significantly decreases over time as the acid-base reaction proceeds. This phenomenon was partially confirmed by the results of the present study because it appears that such conclusions cannot be generalized to all the available types of RMGIs and it is very important for the clinician to know at which part of the spectrum the material they use is located; the spectrum extends from composite resins on one end to GI on the other.

In the present study a 2-min delay in irradiation was evaluated. In routine daily practice, there are reasons for delaying light-activation, for example, in bracket bonding, fissure sealant therapy of several teeth and small restorations in one quadrant in one appointment. According to the results of a recent study, mean bond strength increases with Fuji II LC subsequent to a 2-min delay in light activation, which is to some extent consistent with the results of the present study. Therefore, in general, it appears delaying light activation of RMGIs results in more acid-base reactions, limiting diffusion of polymerizable components, with a final effect on bond strength.

It seems paradoxical that the specimens in the 2-min delay group showed significantly lower bond strength values for Vitremer, but exhibited almost higher bond strength values for Fuji II LC. However, this phenomenon can be explained in several ways. First, since RMGI materials form bonds with tooth structures by means of both micromechanical interlocking and chemical reaction, it is conceivable that a greater GI character in the RMGI, such as Fuji II LC, might result in an increase in the chemical nature of the bond, improving bond strength.[3] Nevertheless, if this happens at the expense of a higher percentage of light activation, the final properties of the material might be more like those of GI, resulting in low bond strength values,[18] in a manner similar to Vitremer. These differences in chemical compositions might result in differences in bond strength.

In this study, specimens in groups 3 and 4 were conditioned with phosphoric acid. The authors hypothesized that enamel preparation might not have been sufficient to make a distinction between the RMGI groups with presumably different physical properties. The results of the present study showed that phosphoric acid increases RMGI SBS values in comparison to 20% polyacrylic acid with Fuji II LC, consistent with the results of some previous studies[15,17] and in contrast with those of another one.[16] An increase in bond strength with the use of 37% phosphoric acid for Fuji II LC and Ionolux can be explained by the creation of rougher enamel surfaces compared to those created with the use of polyacrylic acid.[34,35] However, in this study, application of phosphoric acid did not result in an increase in bond strength for Vitremer. In one study, 10% polyacrylic acid conditioner was used for 20 s, based on manufacturer's instructions, for bracket bonding.^[5] Another study^[36] showed that conditioning of enamel surfaces with 10% polyacrylic acid gives rise to a smooth surface, on which no signs of etching of enamel prisms can be found, but conditioning with 37% phosphoric acid does not. The authors proposed that conditioning with 10% polyacrylic acid is not sufficient to achieve a mechanical bond of the adhesive. Bishara et al.[35] reported that conditioning with 20% polyacrylic acid results in a significant (P < 0.05) increase in the SBS value of RMGI compared to conditioning with 10% polyacrylic acid, which is consistent with the results of this study with Fuji II LC. Cacciafesta, et al.[37] reported higher RMGI bond strength values

subsequent to conditioning with 37% phosphoric acid compared to conditioning with 10% polyacrylic acid. In addition, Godoy-Bezerra et al.[38] reported that conditioning the enamel with 10% polyacrylic acid did not lead to an increase in SBS values of an RMGI. Therefore, based on their studies, it can be hypothesized that conditioning with 10% polyacrylic acid results in insufficient preparation of the enamel surface, making it difficult to make a distinction between the bonding groups based on enamel adhesive bond. Maybe that's why the GC uses 20% polyacrylic acid as a cavity conditioner, which is recommended for enamel. Valente, et al.[16] reiterated that RMGIs require etching to increase their bond strength values and reported that application of phosphoric acid for 15 s is sufficient to achieve optimal bond strength. In the present research 37% phosphoric acid was used for 20 s for conditioning the enamel surfaces so that it would be possible to directly compare it with 20% polyacrylic acid, which should be applied for 20 s based on manufacturer's instructions. Further investigations are necessary to evaluate the bonding properties of RMGIs with increased preparation of the enamel surface to determine whether it can be considered a factor or not.

Finally. during bonding procedures using resin-modified glass-ionomer, clinicians prepare the RMGI and apply it in several seats, including brackets, cavities, fissures etc., to the teeth prior to light activation. [39,40] During the process a delay between mixing and light-activation, which might be variable based on the number of seats bonded, provides the glass-ionomer components with an opportunity to react, affecting the material and its bonding capacity depending on the chemical composition of the material. Therefore, based on available knowledge, generally light-cured RMGIs (especially those which do not have chemical polymerization reaction) should be light-activated after placement. This will give rise to a significant increase in the bond strength and might increase restoration durability. In addition, pre-conditioning of enamel with acid does not always improve bond strength and it might depend on the material composition, in addition to its pH and composition of its primer/conditioner.

Analysis of failure modes showed that the majority of the light-cured specimens of the RMGIs under study had cohesive and mixed failure patterns, which is another indication that a stronger bond had formed between light-activated RMGI and enamel compared to delay-cured RMGI and enamel. The failure patterns of cured and delay-cured Fuji II LC were almost identical, which might be attributed to the chemically-activated free radical polymerization of the material.

A limitation of this study was that only three RMGI materials were included. Different chemical compositions of other RMGIs might give rise to different results. Future studies are necessary to compare the bond strength of other RMGI materials.

CONCLUSIONS

Within the limitations of this *in vitro* study, it is possible to draw the following conclusions:

- Ionolux and Vitremer exhibited decreased enamel bond strength after a delay in light activation. The results suggest that immediate light activation is necessary to obtain optimal bond strength to enamel with these two RMGI materials.
- Although conditioning with 37% phosphoric acid increased Fuji II LC SBS values, it significantly decreased SBS values of Vitremer but had no effect on SBS values of Ionolux.
- 3. A 2-min delay in light activation combined with acid pre-conditioning increased RMGI SBS values only for Fuji II LC and had a pernicious influence on the possible positive effects of conditioning with phosphoric acid for Ionolux.
- 4. More laboratory research and clinical studies are necessary to substantiate the findings of this *in vitro* study.

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